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In current modern society there is a prevailing effort of active application or at least of getting closer to the conditions of sustainable development. One of the main ideas of this strategy could be characterized as an attempt of creating a waste-free society with defined criteria of the waste end, then as an effort to the multiple use of natural resources while reducing the energy consumption, CO_2 production etc. Current waste production offers a wide range of options of using those materials as secondary raw materials into the road constructions. With an overall increase in the industrial production, much higher amount of waste is produced every day. The waste disposal is therefore a serious environmental, economic and social problem. The primer purpose of the use of secondary materials in the road construction is a reduction of environmental burdens, as well as the economic and energy savings. The use of secondary materials will also play an increasing role in the optimization of material resources in the near future. The construction industry has therefore invested in recent years into the development and use of technologies for recycling and valorization of waste originating in particular from the energy industry.

Conference Topics:

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ii. **Secondary Materials - Current Trends of Research, Development and Innovation**

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Contents

Steelwork Waste – Non-Standard Metallurgical By-Product

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Abstract: More than 20 years ago a material named "*studený odval*" (steelwork waste) was decided to use as an alternative fill in embankments of the D47 motorway. This material was certified and its price was very interesting therefore it became the object of interest of all investors and construction companies in the Ostrava region. In all constructions, where the steelwork waste was used, its volume changes were found as soon as the structure was handed over to the Client resulting in pavement deformations in case of motorway or deformations of floors in case of business or shopping centres. Volume stability was not cast in doubt in time of its utilization and therefore it was not analysed. Existing results show, that although the volume changes have lasted for many years, their stabilization has not been proven so far. Therefore it is not possible to predict how long volume changes will persist.

Keywords: Steelwork Waste; Volume Changes; Deformations

1 Introduction

Metallurgical by-products belong to traditional alternative source of material used as fill not only during construction of transport infrastructure but also in construction of shopping and business centres, simply in constructions with high demands on the fill volume.

Metallurgical by-products tend to be termed "*slags*" not just by laymen but even by the professional public. An accurate description and the use of correct terminology in designating the material are indispensable for determining the marginal conditions of its application as well as in retrospect, for analysing any defects or failures of structures caused by volume changes of the metallurgical by-products. The properties of aggregates based on blast furnace or steel slag are determined to a decisive degree by the process employed by the specific ironmaker or steelmaker in question, or as the case may be, the specific producer of a nonferrous metal.

More than 20 years ago a material named "*studený odval*" (steelwork waste) was decided to use as an alternative fill in embankments of the D47 motorway.

This decision, when investor began to think about this material as fill of the D47 motorway, started the execution of lot of laboratory and field tests, however also the certification of this material as the product. This material was certified and its price was very interesting therefore it became the object of interest of all investors and construction companies in the Ostrava region. All these inputs were resulted in usage of this material not only in embankments of the D47 motorway, but also in the subgrade of shopping and business centres in the Ostrava region.

In all constructions, where the steelwork waste was used, its volume changes were found as soon as the structure was handed over to the Client resulting in pavement deformations in case of motorway or deformations of floors in case of business or shopping centres.

A contribution tries to describe known properties of the steelwork waste and it does not plan to comment the dispute between the investor and the contractor of the D47 motorway. Unfortunately, now more than 10 years from its first utilisation as fill we do not know all aspects of behaviour of this very non-standard material.

2 Studený Odval (steelwork Waste) – What Is It?

Material named as steelwork waste or alternatively, mineral blends from the smelter, was not known in time of its utilisation in constructions even by professionals. The mark "*studený odval*" is a slang expression (it was a tipped material which was not hot and got cold) or a brand name. From the name itself it is not possible to estimate its origin, composition or properties.

Nevertheless, a definition of the steelwork waste occurred in 1995 in one report that was ordered by the Directorate of Highways and Motorways of the Czech Republic. *"It is a heterogeneous mixture of metallurgical by-products (mixed metallurgical slags, foundry sands, and refractories - lining e.g. of blast furnaces; such materials are produced in ironmaking and steelmaking. Steelworks waste or similar mineral blends may contain minor admixtures of other materials as well - such as wood, PVC etc.)."* [3].

The owner of the steelwork waste decided to make use from the interest of it and steelwork waste was certified as the product: "*Aggregate class A, B, C for road construction type /variant: Aggregate for subbase. Artificial aggregate homogenised material, aggregate B-0-125*". From the definition itself it does not follow that it is a mixture of metallurgical by-products.

The use of other metallurgical by-products than blast furnace or steel slags (materials analogous to steelworks waste) in foreign countries is rather limited. U.S. federal regulations even require steel slags to be free from any residues of furnace lining (FHWA RD 97-148) [14].

The German regulation TL BuB E-StB 07 also describes, in addition to blast furnace and steel slags, mixtures of mineral substances deriving from ferrous metallurgical (*Hüttenmineralstoffgemischen)* composed entirely of slags produced in ironmaking and steelmaking, plus unsorted refractories.

Chemical composition of this material is similar to the composition of steelwork waste (Fig. 1). In practice, however, these materials which we may regard as similar to steelworks waste are not used in roadbuilding, owing to the substantial risk of volume instability [1, 15].

The steelwork waste contain higher share of Al_2O_3 and especially of CaO than steel slags. Very good conformity between the steelwork waste from pits [4] and the *Hüttenmineralstoffgemischen* composition is apparent from the phase diagram. Anomalous is the sample from km 155.350 (of the D47 motorway) with lower share of SiO_2 and higher share of CaO. The reason of this different composition has not been clarified, yet.

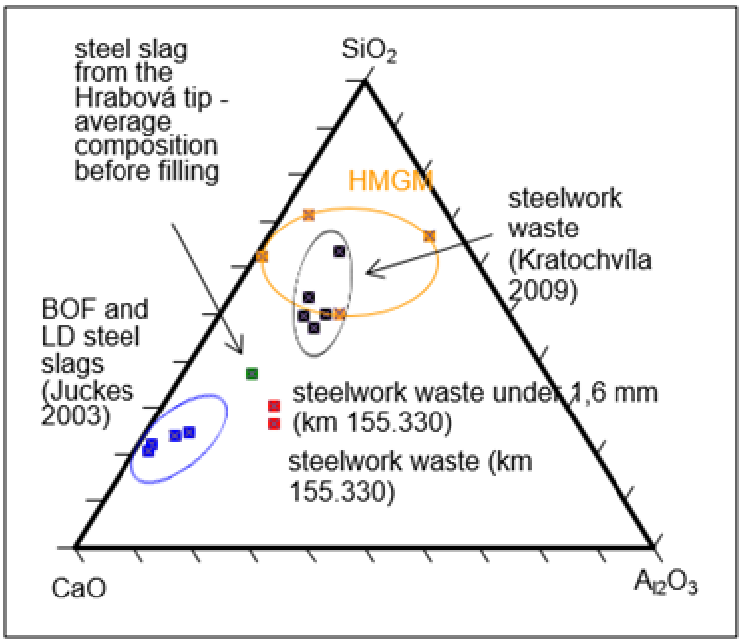

Fig. 1: Composition of the steelwork waste, HMGM (*Hüttenmineralstoffgemischen*) and steel slags in the phase diagram $CaO\text{-}SiO_2\text{-}Al_2O_3$.

3 The Steelwork Waste Properties

During preparatory phase of the D47 motorway construction there were carried out pilot plant compaction trials of the blast furnace slag, steel slag and steelwork waste from the Hrabová tip. Works were ordered by the Directorate of Highways and Motorways of the Czech Republic. Measured and recommended parameters from 1996 are summarized in the Tab. 1.

Fig. 2: The steelwork waste (heap on the right) with debris of metallurgical ceramics excavated form the pit under the floor of the Demos hall in Ostrava (11/2014).

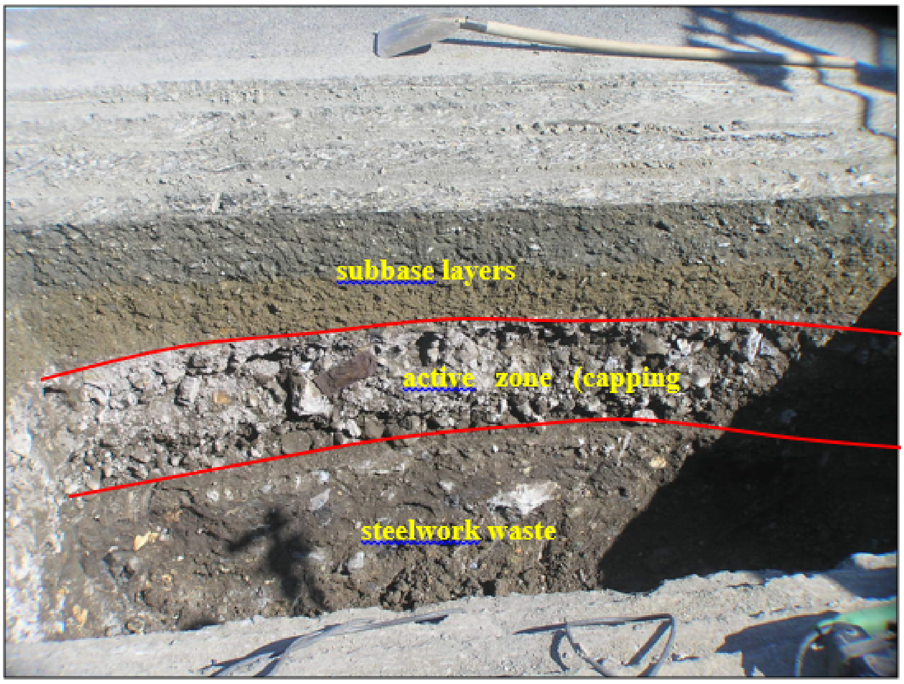

Fig. 3: Pit KSJ22 (km 155.876 of the D47 motorway, right lane) with visible deformed layers of the active zone (capping layer) resulting by the volume changes in underlying steelwork waste in embankment.

Tab. 1: Recommended mechanical properties of metallurgical by-products (adapted after [9]).

parameter	blast furnace slag (the Hrabová tip)	steel slag (the Hrabová tip)	steelwork waste (the Hrabová tip)
fraction	0-300 mm	0-200 mm	0-300 mm
CSN 736133 classification	G2 GP	G1 GW	G3 GF
moisture w_n (%)	4	2	6,9-12,9
maximum dry density (ρ_{dmax}) (kg.m^{-3}) according to the Proctor Standard test	2330	2681	1930
internal friction angle φ (°)	37	35	30
cohesion c (kPa)	3	5	5
coefficient of permeability k_f (m.s^{-1})	1,00.10^{-2}	4,20. 10^{-2}	1,00.10^{-8}
deformation modulus E_{def} (MPa)	100-131	90-130	80-100

The steelwork waste represents by its grain size distribution very suitable and well compacted fill. Grain size distribution curves of the steelwork waste samples from pits on the D47 motorway from 2012 and samples from the Hrabová tip from 1996 [9] are shown in the Fig. 4. Samples from 2012 contain smaller share of sandy and gravel fractions (over 0.25 mm) and higher share of coarse fraction (over 63 mm) comparing with samples from 1996 either from tip, or from the trial test.

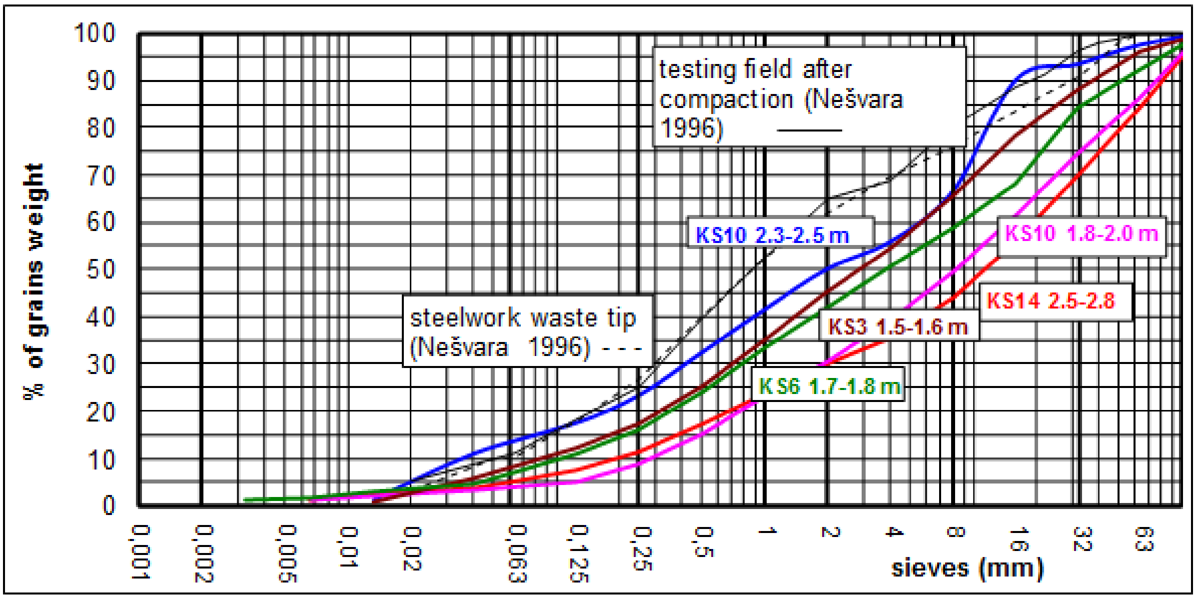

Fig. 4: Grain size distribution of the steelwork waste sampled from pits from 2012 and from preparatory phase of the D47 motorway (1996).

At a general level, the shearing properties of coarse-grained materials - which is what the metallurgical by-products represent - are relatively favourable. In case that the steelwork waste is formed of a mixture of coarse-grained and fine-grained fractions, with the share of undersize up to 4 mm constituting at least 10 %, the shear strength of the material is determined by the shear strength of the 0-4 mm fraction. In 1996, this fraction was subjected to tests in the shear box apparatus using samples compacted by Standard Proctor energy.

It is necessary to stress considerably lower maximum dry density of steelwork waste in comparison with blast furnace and steel slags. It is caused by higher share of silica and magnesium materials and by higher porosity, too. Steelwork waste is less permeable after compaction than blast furnace and steel slags.

The internal friction angle values are nearly the same for both blast furnace and steel slags. The value was

Tab. 2: Statistical evaluation of static loading tests conducted on steelworks waste layers used as fill of the D47 motorway embankment at km 150.330-152.660.

Steelworks waste layer	E_{def2} (MPa) - median)	E_{def2}/E_{def1} (median)	Number of tests
1	117,8	2,01	11
2	111,0	2,12	6
3	119,3	2,30	9
4	119,7	2,16	10
5	128,7	1,93	14
6	104,0	2,25	13
7	110,3	2,44	11
8	107,3	2,06	7
9	101,2	2,17	7
10-14	114,1	1,90	12
15-19	111,6	1,79	10
20-25	142,6	1,63	6
final layer under active zone	188,5	1,63	30
whole set	118,3	1,99	146

lower for steelworks waste (φ = 27.11°), due to a higher content of the fine-grained fraction. High values of effective cohesion are due to the coarser grains being securely braced against one another $i.e.$, wedged. In the long term, however, the high cohesion values cannot be counted on to persist [6].

The deformation properties of metallurgical by-products were determined $i.a.$, by on-site plate loading tests. Generally, the deformation modulus values attained are high (above 80 MPa). Pilot scale tests conducted in 1996 confirmed that the value of the deformation modulus grows higher with the number of passes of the compactor and with decreasing E_{def2}/E_{def1} ratio.

Out of the 146 static loading tests run for the purpose of inspection on the steelworks waste layers along the section of km 150.330-152.660 on the D47 motorway, only three tests gave deformation modulus values below 80 MPa. The median value of the entire set of these data was E_{def2}=118.3 MPa. For no more than six tests the E_{def2}/E_{def1} ratio was higher than 3.00. Results of statistic evaluation of plate loading tests for each embankment layer in km 150.330-152.660 are presented in Tab. 2 and Fig. 5.

Based on the above results it can be stated that according to these criteria, the part of the embankment filled with steelworks waste material and the active zone where steel slag of the 0-90 mm fraction was used were sufficiently compacted and that the degree of compaction and deformation modulus parameters attained, as verified by the inspection tests, were fully compliant with the stipulations of relevant standards, regulations, and project documentation. Similar results as in case of the D47 motorway there were recorded also in constructions of business and shopping centres. This fact caused that steelwork waste became an interesting fill without any possible "*problems*" during its utilization.

4 Volume Changes of Steelwork Waste and Their Development

As it was above-mentioned steelwork waste fulfilled common criteria used by investors and contractors during earthworks. All constructions were passed by investors without defects and backlogs. During construction and finishing of all critical constructions in the Ostrava region (including the D47 motorway) nobody from responsible representatives both on the investor and contractors' sides suppose that a few time after constructions finishing the worst property of steelwork waste – volume instability – will start to manifest.

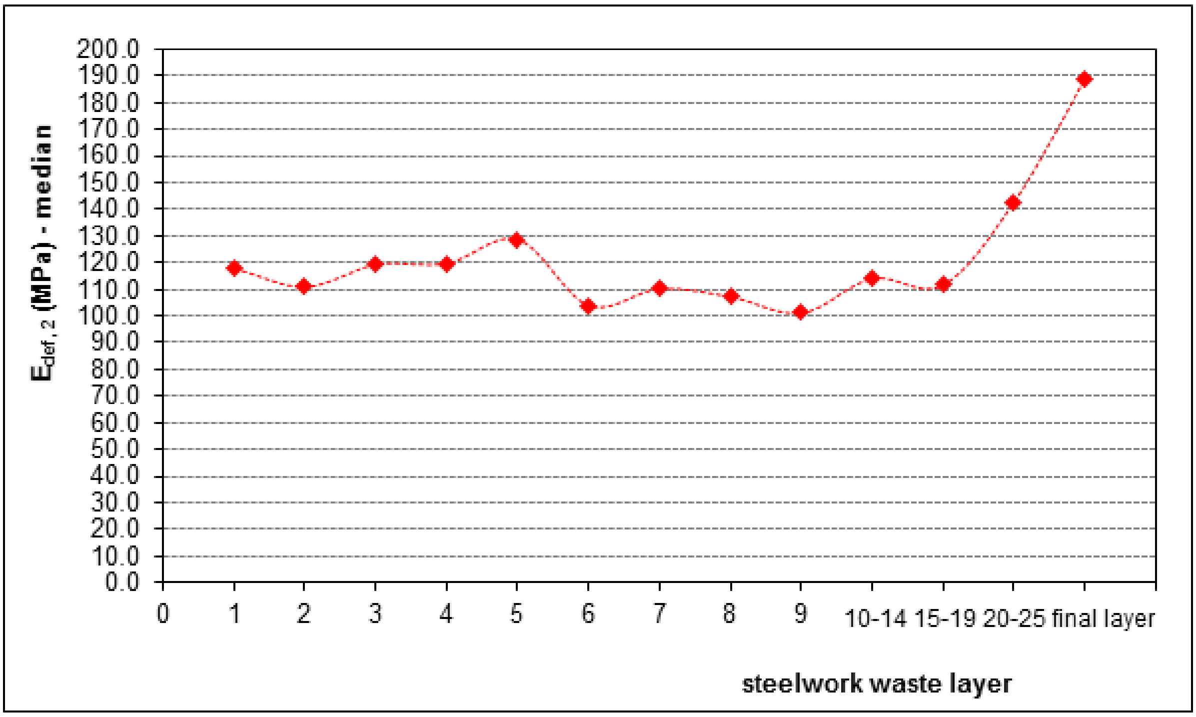

Fig. 5: Trend followed by the deformation modulus values deriving from static loading tests conducted in course-by-course fashion on steelworks waste fill of D47 motorway embankment at km 150.330-152.660.

The chief cause underlying the volume changes (swelling) of steelwork waste is the presence of free lime and the mineralogical composition of the materials concerned and surface of grains under 1 mm. Free lime converts to portlandite $Ca(OH)_2$ in the presence of water. The density of portlandite is less than that of calcium oxide; hence the reaction is manifested by an increase of volume. Free lime originates from undissolved (residual) rock particles in the furnace charge and from lime precipitated during the cooling process and during the transformation of C_3S to C_2S.

Based on the experimental studies it is possible to say that principal influence on the volume changes is done by the fraction less than 1 mm. It forms minimally 20 % of total volume of steelwork waste: in some samples its share was even 50 % (e.g. sample from dump from 1996). Lower grains have in total greater surface that increases a volume changes potential. Higher the share of coarse fraction, the lower the volume changes potential [7]. In case of coarser grains only the surface crust has been originated.

The best information regarding values that volume changes steelwork waste can achieve are resulted from measured deformations. Laboratory tests have not lasted sufficiently long time and they are biased by the scale error, because small samples in laboratories cannot simulate behaviour of material in the embankment. In case of tests accelerating behaviour of volume instable materials by the combination of higher pressure and temperature (autoclave tests, steam chamber tests) there is a lack of correlations to estimate based on these results behaviour of material on the normal temperature and pressure.

Time development of vertical deformations in the D47 motorway was monitored in the period of 2007-2012 and it is similar for measured points. It is evident quick increase in the first year from construction finish (2007-2008) to the first grind off pavement. Then it is evident deceleration of vertical deformations in winter 2008/2009 and slower increase of deformations in the next period. Remedial works did not stop the process of volume deformations and vertical changes continued also in 2012. In the Fig. 6 there is shown a dependence of pavement uplift in km 150.334 in the right lane of the D47 motorway on time without influence of remedial works.

The volume changes within the embankment act in all directions, and this is why the various cases of deformation were not limited to vertical lifts alone of the motorway but were also manifested in displacements of bridge abutments due to increased pressures within the soil in situations where the changes of volume were restricted by the rigid structure of the bridge abutments.

Slow and not finishing development of vertical deformations was not observed not only in case of the D47 motorway embankment but also in case of other constructions. The Demos company hall in Ostrava should be an example. Under the floor there is 1.5 mm of steelwork waste. Since 2009 deformations have been measured and in profile 16 they achieved in October 2014 approx. 40 mm (Fig. 7).

Based on the measured data an approximately linear character of progressive lifts of individual points is evident. Any indication towards to stabilization of deformations or their slowing down has not been observed.

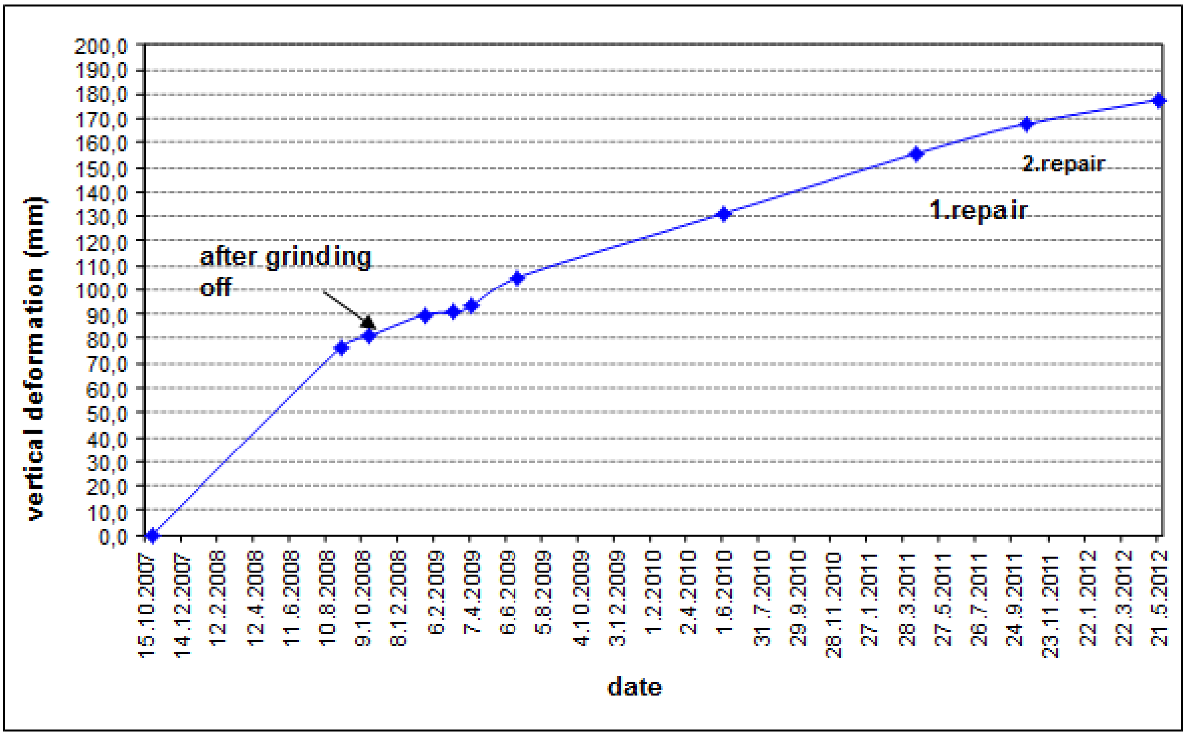

Fig. 6: Development during 2007-2012 of vertical deformation bumps in the right lane of the D47 motorway at km 150.344, disregarding the effect of remediation works (obtained by processing the results of geodetic surveying).

The measurement of volume changes in laboratory conditions is limited. Short time tests under higher temperature and pressure (e.g. autoclave test) provide results very quickly; however, it is complicated to correlate them with behaviour of material under normal conditions. Methodological limits represent difficulties, too. Autoclave test is carried out on the fraction 8/16 and according to the present specifications it is valid for blast furnace slag aggregate only (see appendix A of TP138) [13]. Steam test according to the EN 1744-1 is used only in case of steel slag aggregate and test is carried out for fraction 0/22.5. General short term test for any material has not been installed, yet.

Long term tests than remains to analyse volume changes. They are applicable for any material (both natural, or artificial origin). It is the swell test in the CBR mould according to the EN 13286-47 [12]. A disadvantage of this test is fact that volume changes are slow and results are obtained during several months, even years. Comparison of test results of steelwork waste swelling under standard and non-standard conditions is shown in figures 8 and 9.

The volume changes of water saturated samples tested at the temperature of 75 °C amounted to 27.4 % after 122 days for the samples compacted by 100 % Proctor Standard energy, and 43.1 % after 188 days for the sample compacted by Proctor Modified energy (Fig. 8).

The volume changes of steelwork waste sample under normal temperature and pressure after 3.5 years achieve 4.9 % and still the trend to stabilization has not been observed (Fig. 9).

Swelling pressure values of steelwork waste were measured in case of experimental samples only. The swelling pressure value of 1.548 MPa obtained for the sample KS1 from 1.7-2.0 mm after 48 days at 70 °C was higher than the values obtained from the autoclave test conducted at the pressure of 357 kPa and the temperature of 137 °C for 1 hour (max. 1.28 MPa) presented by Wang [8] for samples of BOF steel slag [10].

For two samples of steelworks waste, the time dependence of swelling pressure tested at the temperature of 70 °C was approximately the same. A more substantial series of tests will have to be run for the data learned to be valid. Increase of deformation is originated in mineralogical composition and changes of mineral phases of individual components of steelwork waste.

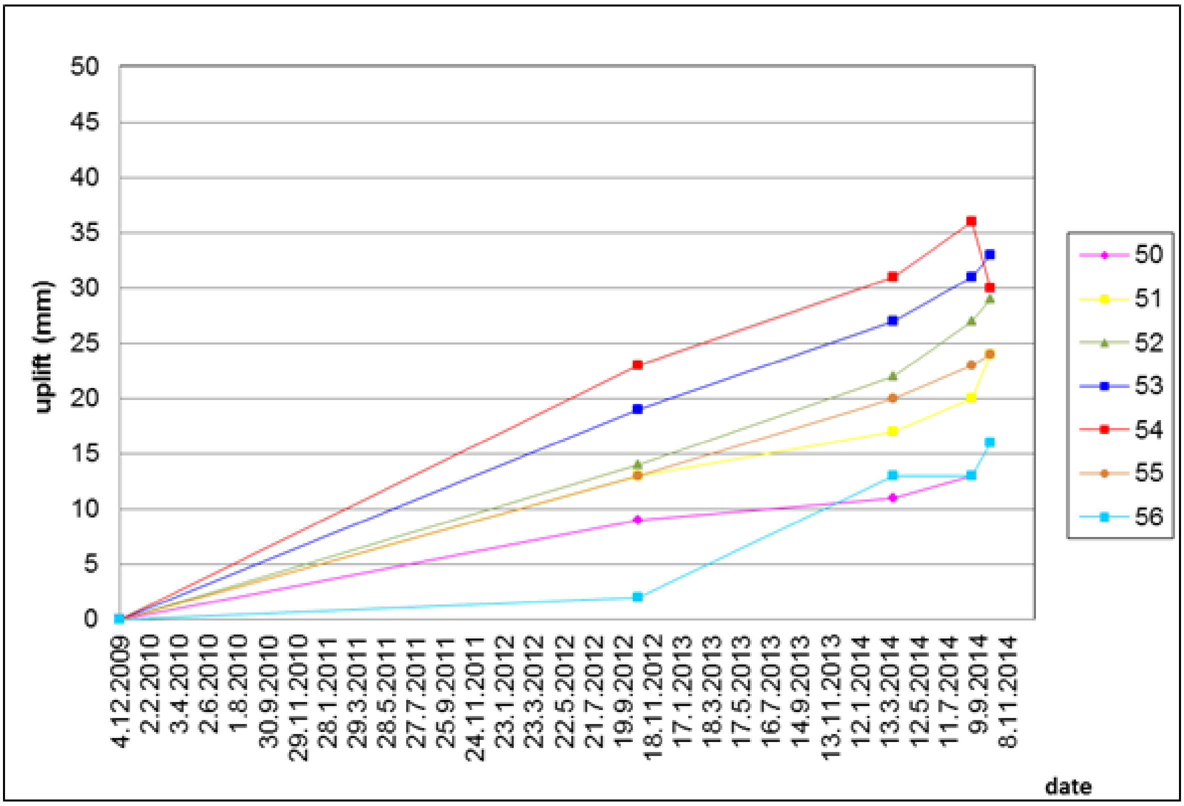

Fig. 7: Uplifts of floor, profile 16, the Demos hall in Ostrava in period of 12/2009-10/2014 (50-56 are numbers of measured points on the floor surface).

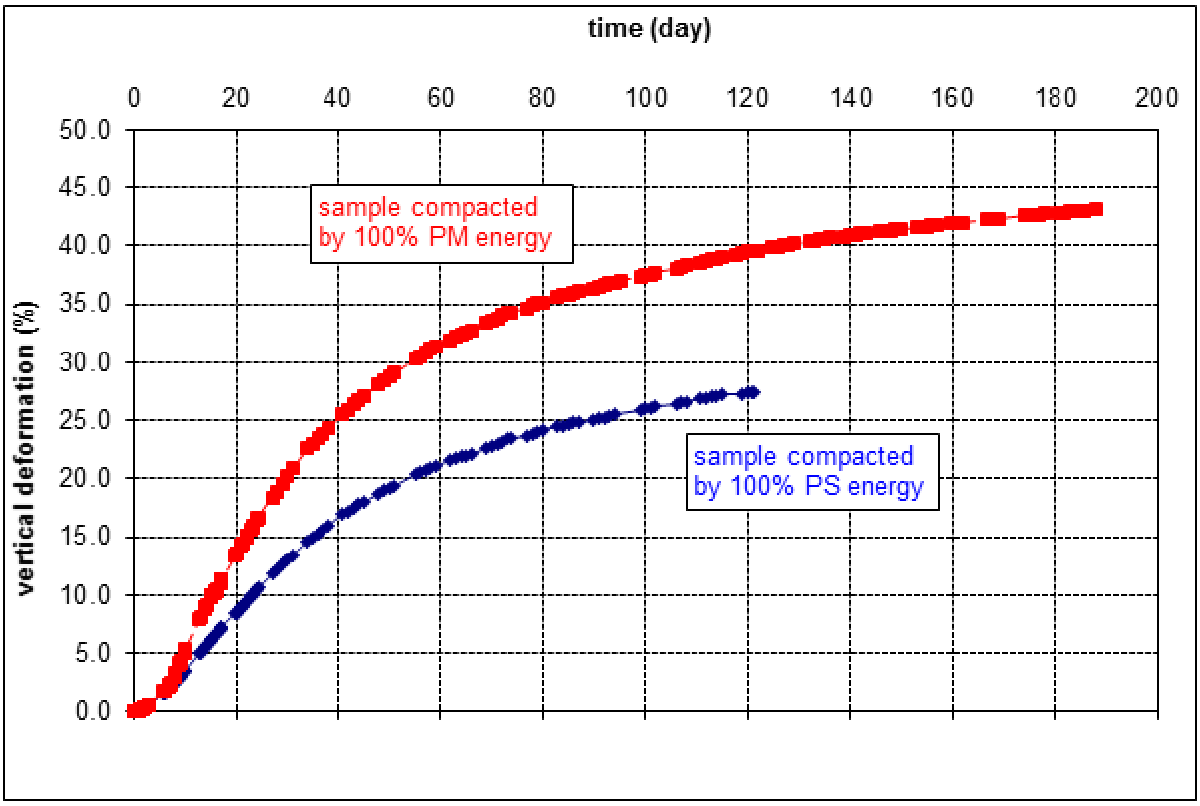

Fig. 8: Progression with time of the increment of vertical deformation of steelworks waste samples soaked with water at the temperature of 75 °C.

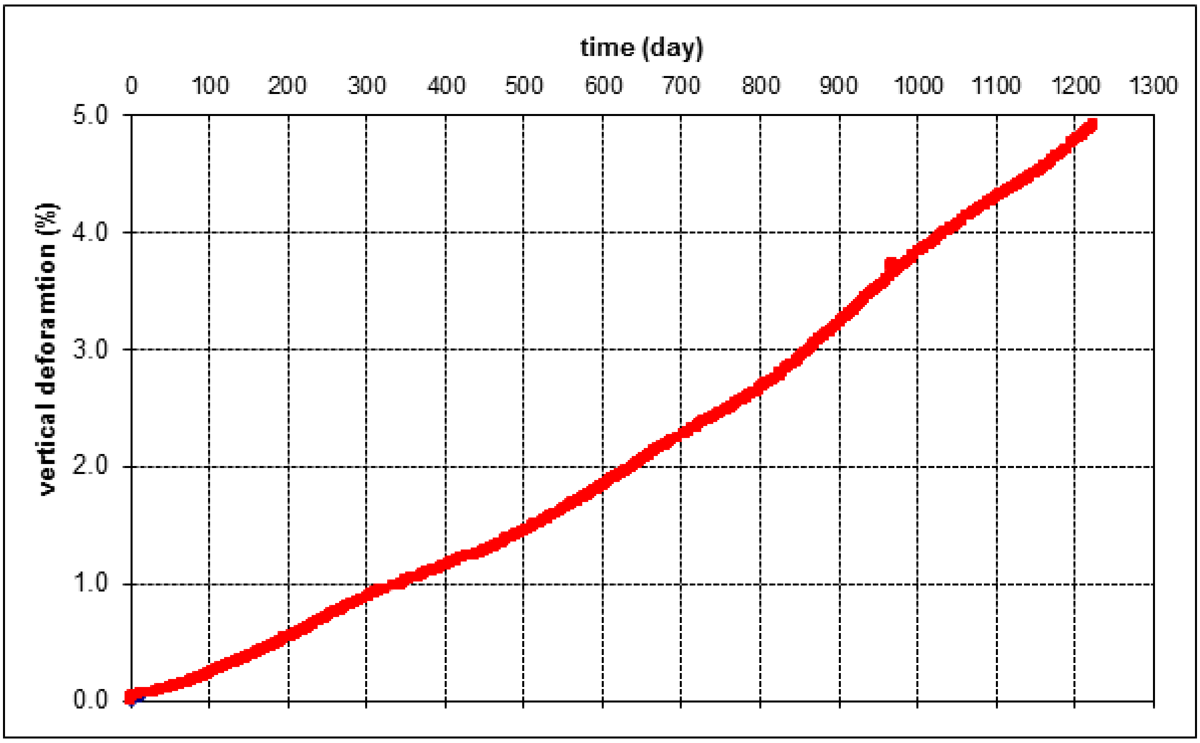

Fig. 9: Progression with time of the increment of vertical deformation of steelworks waste samples soaked with water under normal pressure and temperature (period of 30.1.2012-10.6.2015).

Fig. 10: View of a sample compacted by 100 % Standard Proctor energy at the temperature of 75 °C after 65 days.

It has to be pointed out, however that we still lack the values of correlation between swelling pressure values obtained in environments that accelerate the changes of volume (at higher temperatures) and those that would be obtained in tests performed at standard temperature and pressure.

Another problem is to estimate in which stage of volume changes a steelwork waste material is. The velocity of mineralogical and phase changes is well described in case of pure minerals. In case of such heterogeneous mixture which is represented by the steelwork waste, it is not possible definitely to set any prognosis.

Mineralogical analyses before and after autoclave tests were carried out in case of the Ikea shopping centre samples to find out changes in mineralogical association. It was observed that after autoclave tests a share of calcite increase comparing with share of portlandite. However, it was not possible to quantify mineralogical changes after autoclave tests to predict behaviour of this material in the future [7].

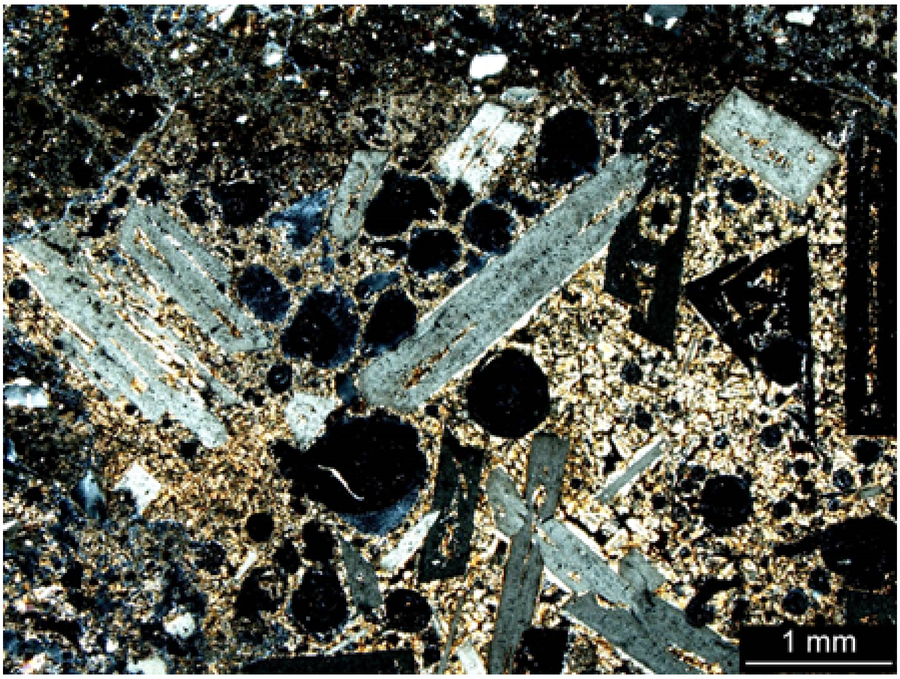

Fig. 11: Cemented mass from slag grains. Typical product of steelworks waste transformation. Sample after autoclave test. Photo: J. Ščučka.

5 Conclusion

Steelwork waste is non-standard material. A majority of its properties (almost all with exception of one – volume stability) shows sufficient results. Based on the existing analyses it is resulted that the reason of the pavement deformations on the D47 motorway and deformations of floors of many business, shopping and industrial centres in the Ostrava region was the usage of steelwork waste.

From the analyses performed so far it follows that the cardinal error committed in the case of the D47 motorway was the use of a material (steelworks waste) in respect of which no experience was available from other applications within the construction limits of a scope comparable to those of highways.

Results of tests show that although deformations lasted for many years their stabilization has not been definitely proven. Therefore we are not able to predict how many years deformations will be lasted. Values of swelling pressure are so high which show results of tests under non-standard conditions (temperature of 75 °C), when there were measured values so far unpublished, but also consequences of volume changes in constructions where steelwork waste was used.

This very expensive experience, because the remedial works of steelwork waste usage will be very expensive, maybe increase the cautiousness in case of utilization of unknown and untested materials. On the other side probably the opposite extreme will not occur when good secondary material will be excluded from constructions only therefore that they have its origin in metallurgical production.

There are many questions, however, we do not know if they will anytime answered because research financing to understand properties of this material is not in comparison of disputes actual.

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Use of Tailings Ashes in Road Embankment Structures

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Abstract: Road embankments requires processing of a large amounts of materials, therefore there is currently an effort to find a suitable replacement for traditionally used materials. One option is using some solid combustion products from coal (ashes), which production and subsequent disposal in the impoundments continues to grow [1].

Keywords: Ash; Embankment; Settlement.

1 Introduction

The largest amounts of waste material are in our region produced by energy production (thermal plants and heating plants). It is a waste of mineral origin – cinder, ash from coal and coke, fly ash and dust. In our region are mostly incinerated less valuable solid fuels (lignit and brown coal dust with heat value of about 2500kcal/kg). Compared to more valuable black coal dust (with heat value of 5800kcal/kg) is the amount of fuel required for produce a unit of electricity in our country about double. This even results in greater waste production. With continuous growing production of waste products increase problems with their landfilling [2]. In connection with growing production of waste deposited on tailings impoundments would be beneficial to use these materials secondarily.

Fig. 1: Deposition meander of ash impoundment in Zemianske Kostolany.

The resulting mixture of ash deposited on tailings impoundment consists of [2]:
- cinder, which is after the falling down from the hoppers of boiler installations milled - milled particles are mostly of larger dimensions,
- fly ash, which falls down from the separators.

Ashes thus can be divided into coarse-grained with dominant sand particles and fine-grained ashes with dominant silty particles. Size distribution of ashes is documented on Fig. 2.

2 Properties of Ashes Deposited on the Impoundment in Connection with Their Use in Embankments of Traffic Engineering

Alternative materials must show the same or better geomechanical properties than traditional, in order to road structure pursuance of the all the prescribed requirements. Assessment of suitability of using ashes in embankments of road constructions was carried out according to the criteria in Slovak (or Czech) technical standards and technical and qualitative terms (further just TKP) of the Ministry of transport, construction and regional development of Slovak republic (further just MDVRR).

(a) dry samples (b) wet samples

Fig. 2: Coarse-grained and fine-grained samples of ashes [3].

2.1 Requirements (criteria) of Suitability Using Ashes in Road Embankments Constructions

A) TKP MDVRR part 2 Earthworks for embankments from ashes states following [4]:
Ash used in embankments cannot contain more than 3 % particles like wood, organic waste atc., or more than 5 % of organic components. For the construction of the embankment body can be used dry fly and ash taken from power plants and impoundments, but must be mined over the water level. The proposal is effective especially in case of embankment foundation on weak subsoil (less weight of embankment).

B) According to Slovak technical standard STN 73 6133 are soils suitable for the soil embankments if [5]:

1. have a continuous grading curve and are well grained,
2. have missing or stable clay and silty particles (recommended fine-grained content of f_{max}=50%),
3. are with small amount of compaction work well compactable to high unit weights (ρ_{dmax}=1500kg.m^{-3}),
4. are not frost susceptible,
5. have very good permeability.

2.2 Assessment of Suitability Using Ashes in Road Embankments Constructions

To assess the suitability, the representative grading curves of coarse-grained (blue grading curve) and fine-grained (green grading curve) ash indicated on Fig. 3 were used. The red line shows the boundary between fine-grained and coarse-grained ash. Classification of fine-grained ash to class F5-F8 is only based on grain size distribution. Determination of consistency limits for these materials are not feasible.

1) Criterion of continuous grading curve and good grain is satisfied in both types of representative samples of ashes: **fine-grained ash is ok, coarse-grained ash is not**.

2) Ratio of fine particles is in case of coarse-grained ash f = 10 %, in case of fine-grained ash f = 90 %: **fine-grained ash is not satisfactory, coarse-grained ash is ok.**

3) Compactability parameters expressed by dry unit weight and optimal moisture can be found in Fig. 4 and Fig. 5.

Criterion of maximum dry unit weight at compaction (ρ_{dmax} 1500 kgm^{-3}) is established for soils where density is much higher than the density of ashes (soils - 2,5-2,7 gcm^{-3}, ashes - 1,8-2,1 gcm^{-3}). Assessment of suitability ashes into embankments according this criterion would therefore be pointless. The low density of these materials can be use for embankment foundation on weak subsoil.

4) Assessment of frost susceptibility was carried out according to the modified Scheibl criterion [5] – Fig. 6. The grading curve of fine-grained ash falls into a danger frost zone. Coarse-grained ash can be classified according this criteria as mild frost soil. **Fine-grained ash is not satisfactory, coarse-grained ash is ok.**

5) Permeability of floated ashes are as follows:

- fine-grained ash → k_f= 4,5.10^{-7}m.s^{-1},

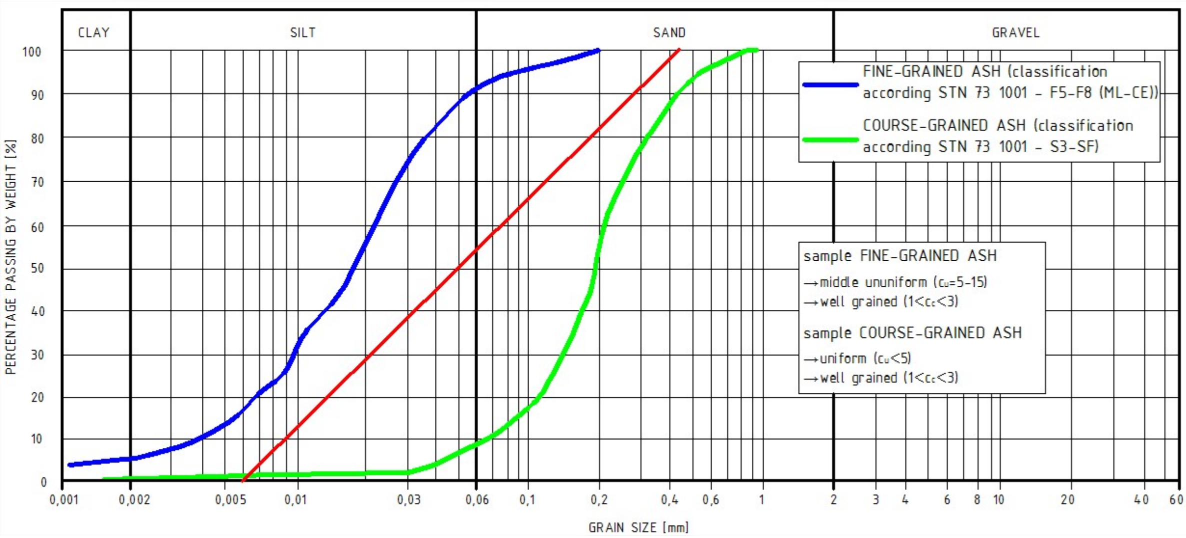

Fig. 3: Grain size distributions of representative samples of fine and coarse-grained ash [3].

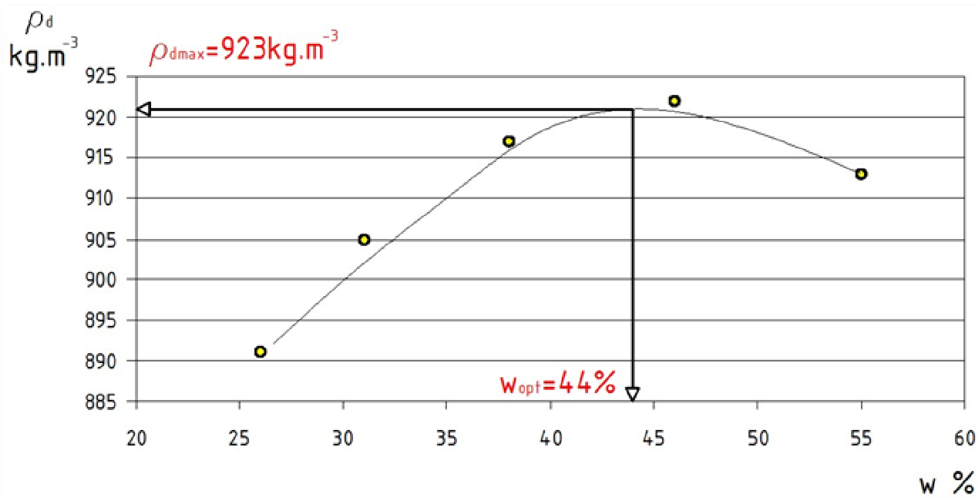

Fig. 4: Compactability parameters of fine-grained ash [3].

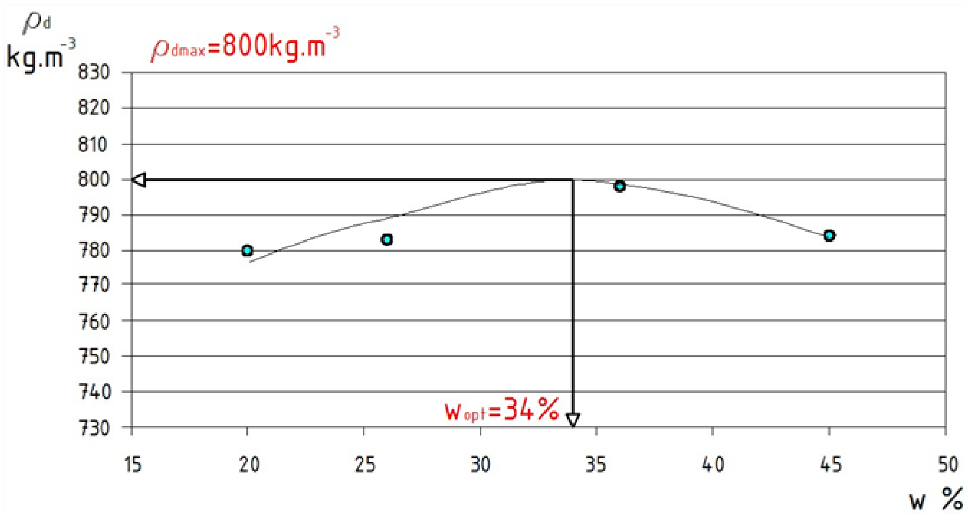

Fig. 5: Compactability parameters of coarse-grained ash [3].

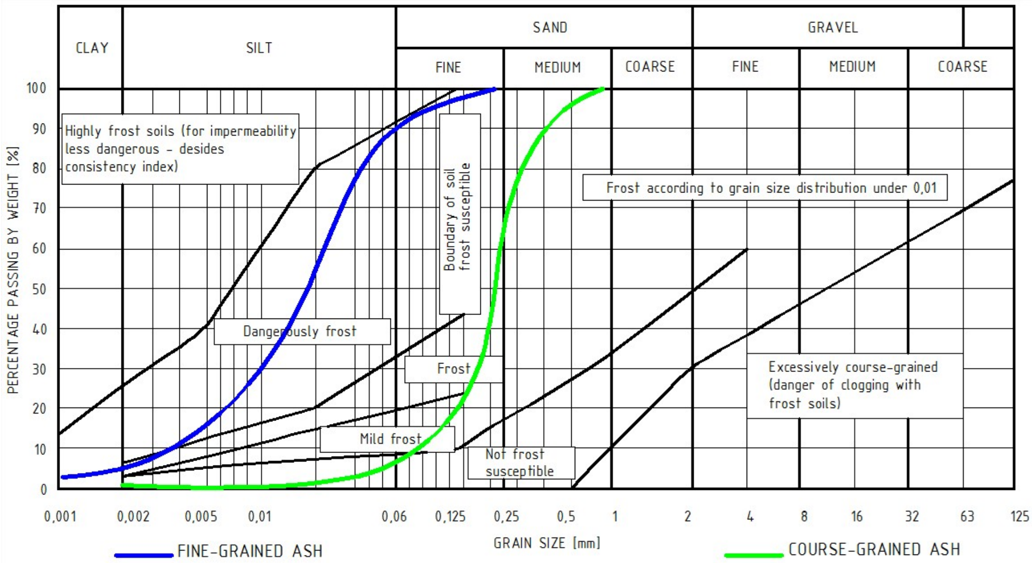

Fig. 6: Frost susceptibility criterion for soils according grading (modified Scheibl criterion) [5].

- coarse-grained ash → k_f= 2,05.10^{-5}m.s^{-1}.

To the requirement of very good permeability is closer coarse-grained ash.

2.3 Summary

Based on the assessment of two types of ashes deposited into the tailings impoundments (coarse and fine-grained ash) appears to be a preferable alternative to use coarse ash into the embankments. It results mainly from the grain size distribution which is in case of coarse-grained ash preferable, particularly in terms of low proportion of fine-grain fraction, less frost susceptibility, high permeability. Silty fraction of fine-grained ashes has the ability to bind water, what is inappropriate especially in terms of frost susceptibility.

3 Proposal of Reinforced Embankment of Highway R2 Zvolen East – PstruŠa Using Ashes

3.1 Engineering Solution and Boundary Conditions of Geotechnical Calculations

Proposal of reinforced embankment of highway R2 Zvolen East-Pstruša was carried out in km 7,369 70 – Fig. 7. Cross section of the embankment in km 7,369 70 reaches a height of 13 m. Shape of the embankment is in the Fig. 8. The evaluation of geotechnical conditions of subsoil was based on results from exploration wells JP-62 and DPS-24, which were situated in a km 7,369 70 [6]. Type and thickness of subsoil layers are in the Fig. 9.

Geotechnical properties of subsoil and materials used in embankment are shown in Tab. **??**, and the shear strength line of compacted coarse-grained ash is in Fig. 10. Load from transport has been in [6] determined using two load models. The first load model taken from the STN EN 1991-2 considered into account the following parameters: location of loading lane, type of road, number and width of loading lanes. The second load model took into account the gravity of ground dimensions of the actual vehicles. The result is an uniform continuous load q [kN.m^{-2}]. The most adverse combination of load states from two load models was determined the combination during construction of the road (weight of land vibratory roller - 18 kN.m^{-3}).

Design of geosynthetic reinforcement was based on the static assessment of the structure, whereby uniaxial polyester geogrids Secugrid® R6 with a characteristic short term tensile strength from 60-200 kN/m were chosen.

* geotechnical properties of compacted coarse-grained ash according PS ** soil of embankment originally projected

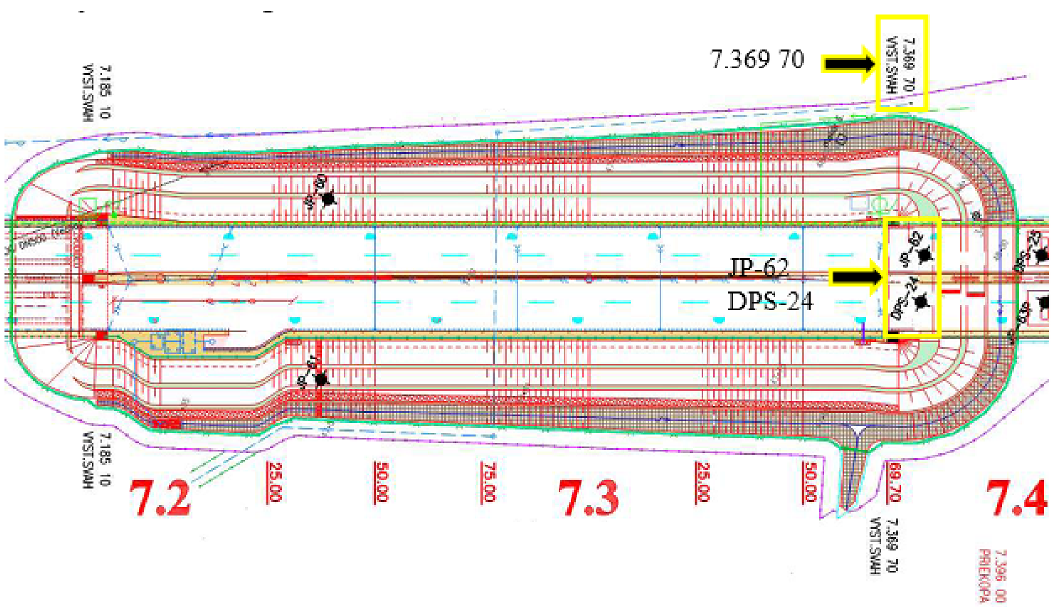

Fig. 7: Situation of highway R2 Zvolen East – Pstruša (km 7,185 10 – 7,369 70) [6].

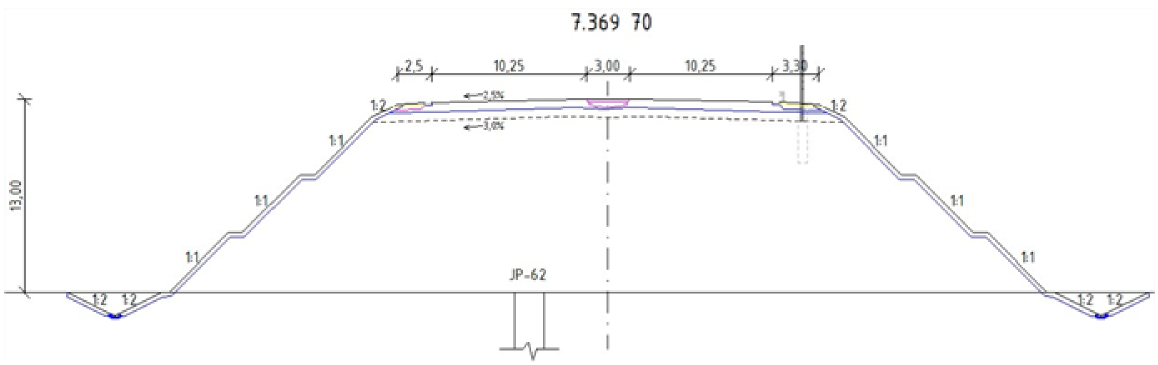

Fig. 8: Shape of embankment in km 7,369 70 [6].

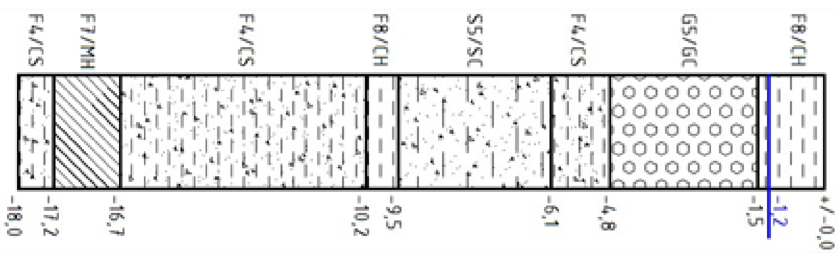

Fig. 9: Subsoil layers [6].

Tab. 1: Geotechnical properties of materials used in geotechnical calculations.

name of soil	classif. STN731001	n [-]	g [kN.m^{-3}]	f$_{ef}$ [°]	c$_{ef}$ [kPa]	I$_c$ [-]	E$_{oed}$ [MPa]
subsoil: clayey sand with gravel	S5-SC	0,4	18,5	27,0	8,0	-	29,9
subsoil: clayey gravel	G5-GC	0,3	18,5	33,0	2,0	-	93,3
subsoil: sandy clay, stiff	F4-CS	0,4	18,5	23,0	11,0	1,0	15,8
subsoil: silt with high plasticity, stiff	F7-MH	0,4	18,5	17,0	16,0	1,15	24,6
subsoil: clay with high plasticity, stiff	F8-CH	0,4	20,5	17,0	15,0	0,9	12,3
embankment: coarse-grained ash*	S3-SF	0,27	10,7	37,2	0,0	-	20,0
embankment: gravel with fine-grained soil**	G3-GF	0,25	19,0	31,5	0,0	-	114,0

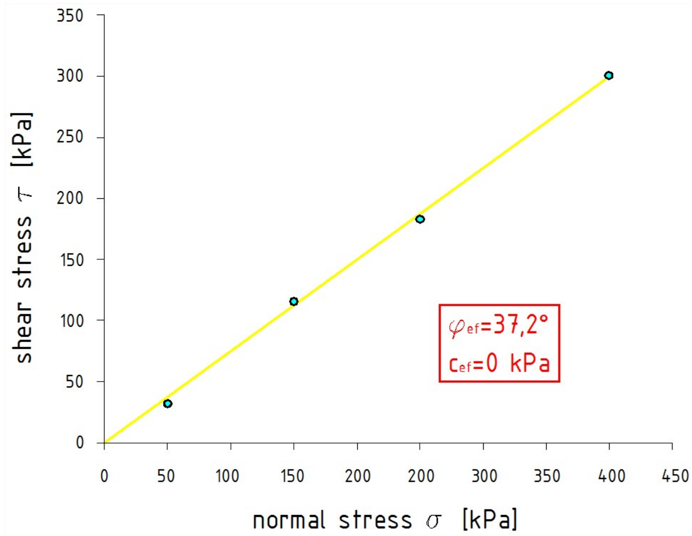

Fig. 10: Shear strength line of compacted coarse ash [3].

3.2 Geotechnical Calculations

I. LS → Within the geotechnical calculations was firstly performed an assessment of reinforced embankment from compacted coarse ash in the program GEO5 - module *Reinforced embankments*. In module reinforced embankments was the structure assessed to topple and displacement. The vertical load-bearing capacity of subsoil, internal stability (breakage and rip of reinforcement from embankment block) and global stability according to Bishop (Fs 1,5) was assessed. In module *Slope stability* was subsequently considered external stability of embankment according to Sarma (Fs 1,5). The results of the assessment are summarized in Tab. **??** and proposed types (according to short-term tensile strength), number and length of reinforcements are in Fig. 11.

The reinforced embankment from compacted coarse ash meets all assessments within I. LS.

II. LS → In module *Settlement* was according to the methodology STN 73 1001 calculated the final settlement of subsoil and effective stresses under the proposed embankment from coarse ash. Calculated final subsoil settlements under the embankment from coarse ash were compared with the final subsoil settlements under embankment which would be constructed according to the original project proposal (from soils G3-GF).

In Fig. 12 are calculated final subsoil settlements under embankment from compacted coarse ash. Subsoil settlements below the center of the embankment reaches up to 150 mm, what is despite of low weight of compacted coarse ash relatively high value. In comparison, the final settlements under embankment originally proposed (soil G3-GF) reaches up to 350 mm - Fig. 12.

Tab. 2: Results of geotechnical calculations.

assessment	results
topple	M_{res}=17693,52 kNm/m 561,55 kNm/m = M_{ovr}
displacement	H_{res} = 644,47 kNm/m 85,45 kNm/m = H_{act}
vertical load-bearing capacity	R_d= 396,38 kPa 146,25 kPa = σ
reinforcement breakage	R_t = 23,74 kN/m 2,08 kN/m = F_x
reinforcement rip	T_p = 191,30 kN/m 1,54 kN/m = F_x
stability according to Bishop	F_s= 1,69 1,5 = $F_{s,lim}$
stability according to Sarma	F_s= 1,55 1,5 = $F_{s,lim}$

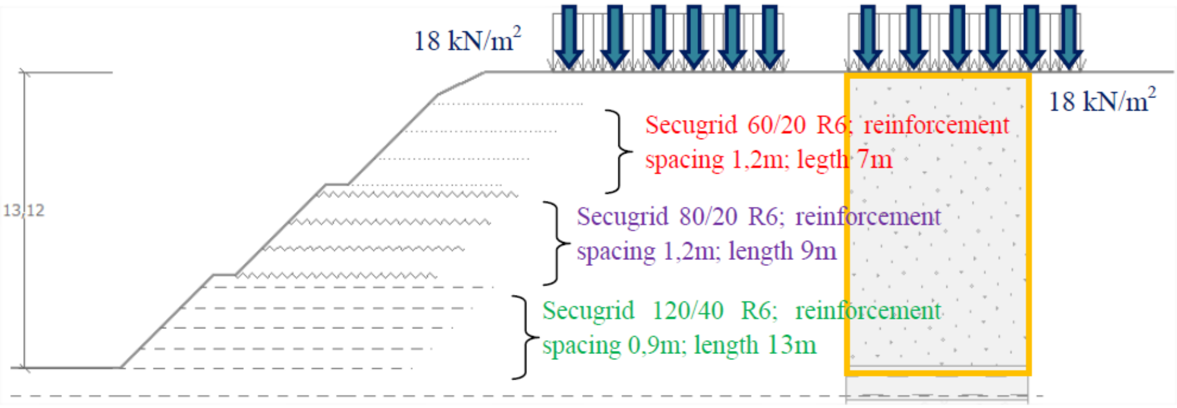

Fig. 11: Reinforcing elements placement in the body of embankment from compacted coarse ash.

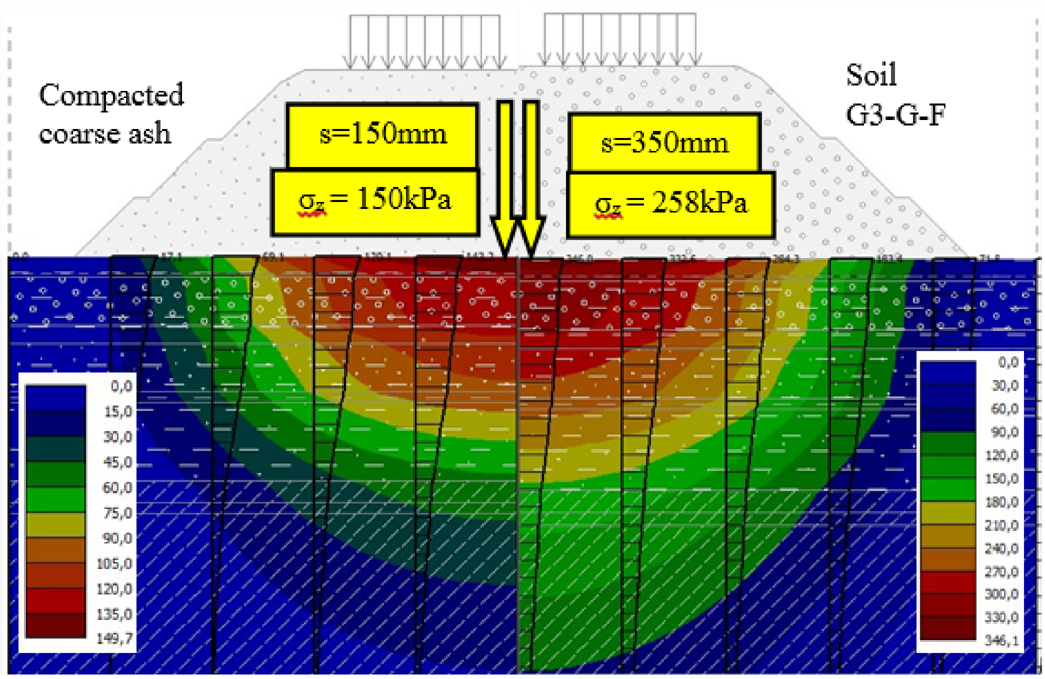

Fig. 12: Settlement isosurfaces of embankment from compacted course ash / from soils G3-G-F.

4 Conclusion

The low unit weight and a high value φ_{ef} of ashes are very favorable properties with respect to their use in embankments of road constructions. Based on the assessment of suitability of using ashes in embankments coarse ashes appear to be more appropriate than fine-grained ashes, especially in terms of their low frost susceptibility, the low proportion of fine grains and relatively high permeability. Purely fine

ashes are in many aspects inconvenient. According to this statement, the separation of coarse ashes from the fine particles would be necessary, what can be ensured by cycloning, or by extraction of coarse ash near the outlets, where is the biggest concentration of the coarse ash.

From comparative calculation of settlement of the subsoil under embankment of "light" compacted coarse ash and of the originally projected soil G3-GF resulted significant differences. The final settlement of subsoil using ash would be approximately half compared to using soil G3-GF into the embankment.

Slovak standards do not specify requirement of assessing suitability using ash in embankments of road construction in terms of ecology i.e. *chemical properties* of the embedding material. Czech standards indicate this requirement. According to the technical requirements, Ministry of Transport, Department of Road Infrastructure (TP 93 - Design and operation of constructions of communications using fly ashes and ashes [7]) specified the maximum amount of leached chemicals (elements). In case of tailings ashes would be useful to assess the arsenic extraction, which is probably in these materials in increased quantities. In this article, the interest was not paid to this assessment.

According to [8] until the 90s of the last century was using of ash in civil engineering limited to only ground backfilling and leveling near by the source. Since the 90s of the 20th century in Czech Republic using ash in civil engineering particularly in embankments of roads highly increased. In some years the processed amount exceeded 100 000 tons per year (e.g. construction D11 Osičky - Hradec Králové in 2009).

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Application of Coal Combustion By-Products in Levees

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Abstract: Levees are geotechnical structures (among the earth structures of water management structures) built along rivers especially. Embankment dikes require processing of large volume of material, and because of that there is effort to find a suitable replacement for traditionally used materials. One option is the use of Coal Combustion By-Products (CBB). Levees with CBB were built in several quantities in Czech Republic. Realized structures generally with problem-free function are documented. In some cases, relating to flooding during June 2013 there has been a failure. The paper shows some cases of applications of CCB and summarization of the results of usage CBB in earth structures of water management in Czech Republic.

Keywords: Levee; Failure; Flood; Coal Combustion By-Products.

1 Introduction

Floods are a common part of the natural and water cycle in nature. Building of protective structures and the level of protection are arising from public demand and of values of the protected area. Flood protection is technically possible in principle always, the question is economic acceptability. In connection with the floods in recent years (in 1997, 2002, 2013) is given special attention to flood protection dikes or levees that fulfills its function intermittently over a prolonged period of time [1]. These types of levees and the option of using alternative materials is becoming to be in demand, however their building require processing large volumes of materials.

One possibility is the use of solid products of coal combustion, known as Coal Combustion By-Products (i.e. CCB). They include: fly ash, slag, cinder, bottom ash and flue gas desulfurization (FGD) gypsum. For some of these materials, it is necessary prior to their application of the structure to assess susceptibility of unwanted effects. This is especially a relatively small resistance to repeated contact with water and freeze (i.e. frost resistance) reporting volume changes and the risk of failure to meet hygienic and environmental requirements [2]. In addition, alternative materials must exhibit equal or better geomechanical properties than conventional materials.

Most experience of using CCB in earth structures of water management provides building protective dams (dams step-up) on fly ash tailing ponds/storages.

Due to the completion of the recovery of individual blocks of power plants are assumed long-term operation of thermal power plants. It is obvious that the production of CBB in Czech Republic will be still high, so the construction of dikes contains CCB will be in effect in the upcoming years.

2 CCB Properties

For determining the properties of CCBs are primarily coal qualities, from which the CCB produced, but also the temperature and combustion conditions and the method of separation of coal ash from the flue gas. The temperature of combustion depends on the technical parameters of the combustion device, and determines the emergence of various mineral products. Individual types of CCBs differ in chemical and mineralogical composition and granulometry, which affects their use.

Of combustion and desulphurization technologies used in power plants in the Czech Republic formed below CCB:

- at wet limestone scrubber: cinder (in the case of grate boilers), slag (in the case of granulation boilers), ash and gypsum fraction;
- at semidry scrubber : cinder (in the case of grate boiler) slag (in the case of granulation boilers), ash fraction and desulphurization product;
- at fluidized bed combustion: filter ash, bottom ash and to a lesser extent ash from cyclone.

The largest producer of CCB in Czech Republic is company ČEZ, Inc., what is the largest producer of electricity in Czech Republic. The largest producer of electricity from coal in Slovakia is Slovenské elektrárne, a.s. (Slovak Power Plants, Inc.), which operates two thermal power plants Nováky and Vojany. CCB of these plants has potential uses in earth structures of traffic constructions including structures of levees. In Slovakia most of the CCB production is stored in tailing ponds, as alternative material is still not used in levees.

As CCB are also considered treated solid residues after combustion of coal, which are referred to as stabilizate or agglomerate.

As stabilizate we understand the mixture of ash and desulphurisation products or ashes from fluidized bed boilers, which is mixed with water with the addition of additives (lime, cement) to take advantage of the ability of the ash to solidify and harden like for example cement. Some of the stabilizers may lead to the formation of ettringite which causes volume changes. Ettringite formed in the fly ash stabilizate from soluble compounds of Ca, Al and S in a moist alkaline environment.

CCB are usually considered to be waste materials, which contain a higher concentration of contaminants against consentration in standard building materials, such as soil. Therefore, they must comply with the criteria, i.e. the quality of liquor and mass activity Ra_{226}.

CCB is one of the lightweight building masses. Dry bulk density reaches 650 to 900 kg.m^{-3}. CCB well compacted with optimum humidity (20-35%), bulk density reaches 1 100-1 200 kg.m^{-3}.

Within the experimental work that was further studied in the Department of Road Structures, Faculty of Civil Engineering in Prague frost resistance and water, including the reporting of volume changes on some fly ash mixtures. Stabilizate is possible in principle to use the construction of levees, but due to the negative results of the cyclic test of resistance stabilizer against freezing and thawing and significant absorbency using certain types of fly ash stabilizer does not seem too promising [3,4].

3 CCB Application Possibilities and Examples of Usage in Earth Structures of Water Management Structures

Ash stabilizates or other types of CCB can be used in dikes by several ways (Fig. 1). Alternative material can form a homogeneous body of the dam with protective side stir (levees Vrdy) where compacted CCB provides a stabilizing and sealing function, or can serve as a sealing core dikes (levees Pardubice), or the CCB used only to base layer structural layers of the road (Vrbno near Melnik).

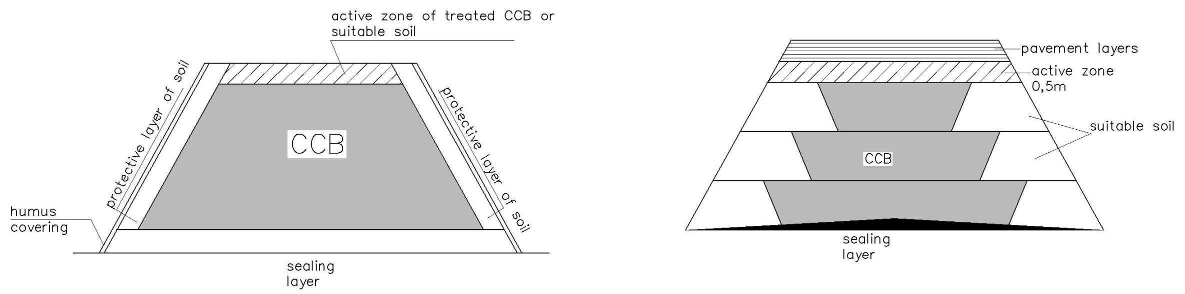
Natural protective side stir are necessary for the protection of the fly ash material from direct contact with water, which readily undergoes erosion.

Application CCB allows building protective earth dams in areas with poor soil bearing capacity, which is common in flood plains, which cover consists of fluvial sediments.

3.1 Examples of Use CCB

In the village Rohatec near Hodonin levees were built along the Morava River protecting the village against flooding (Fig. 2). Dam crest is headed 0.5 m above the water level at the centennial water on the Morava River (Q_{lOO}). The height of the dam corresponds to similar dams (Skalická) on the Slovak side of the river. The dam is 1.271 meters long, inclined slope of 1:2 and a dam crest has width of 4 mm. On the dam crest is built a lightweight construction of road (bike trail), width of 3.0 mm with 0.5 mm verge. The dam is designed as a continuous liner body with an inner solid core and the slopes of soil. Until the dam was applied to fly ash stabilizate REHAS EHO from near Hodonin power plant. Is the ash mortar, i.e. moistened mixture of bottom ash and fly ash (from fluidized bed combustion technology). The total volume of embankments amounts to 20.670 cubic meters.

In the town of Hradec Kralove protective levees were implemented protecting the southern and southeastern part of the built-up area of the village before the flows in the river eagle until Q100. The total length of the dam



(a) use CCB in simple body structure levees (sealing and stabilizing function)

(b) the internal compozition of the dam construction used as a traffic road (roads, cycling)

Fig. 1: Possible applications of CCB in levees, including road construction on the dam crest.

Fig. 2: Levee with asphalt bicycle trail near the village Rohatec in the Hodonín (http://standa1977.rajce.idnes.cz).

is 530 mm, height of about 1.20 to 1.80 meters. The investor of structure is the "Elbe River Basin", state office (Fig. 3).

Ash from the Heating Plant Malešice in Prague was used in the construction of levees in Rohanský ostrov (Rohan Island) in Karlín, part of Prague. Wold before the levee was later sprinkled up to the crest in order to increase the vertical alignment of the ground above flood flows, so it is no longer a barrier in the ground so evident.

In Pardubice construction of the dam was used to seal the core of the CCB from a nearby power plant Opatovice. After the dam crest is led by asphalt road. The core of the dam is protected side stir. The amount of stored fly ash stabilizer amounted to 52.500 tons (Fig. 4).

After previous experiences of urbanizated area of town Vrdy near Čáslav there was built levee in 2012, whose body is made up from ash stabilizate (Fig. 5). The dam has used 2.000 tons of fly ash stabilizate. In 2013, it successfully passed the river Doubrava spill and protect the village from flooding.

3.2 Causes of Hydraulic Failure

Earth dikes can exhibit a wide range of hydraulic failures. Fault conditions associated with crossing one of the limiting states. Breach of the dam may be due to loss of stability, overtopping the dam, filtration deformities, erosive action of water and sabotage, war and terrorism [5,6].

Most of the failures of earth dikes in the Czech Republic are due to overflow of the crest of dike during

Fig. 3: Completed levee near the village Nepasice (http://reka-orlice.sije.cz/trebechovice-hradec-kralove).

(a) levee during construction

(b) levee after construction

Fig. 4: The levee with bicycle path using CCB in Pardubice (Power Plant Opatovice, 2014).

the floods (40%), when there is a failure of the body of the dike, or even to its rupture. Embankment dikes are rarely designed as a spillway dam and their resistance to failure surface erosion is limited. While at low dikes constructed on a low flood flows with a higher frequency of repetition is the probability of exceeding the design flow higher. Increase the safety of the dike can be achieved by appropriate technical measures (site assessment and appropriate controlled spillway dike construction solutions in these areas). It is important to draw up emergency plans and warning system. A necessary condition for increasing the safety of dikes, is their monitoring and ongoing maintenance [6].

3.3 Failure of Protective Dikes

An example of the use of CCB, albeit to a limited extent, is also a dike near the village Vrbno near Melnik on the left bank of the Vltava near the confluence of the Vltava, Labe and navigation channel "Vraňansko-hořínský" (Fig. 6) [3]. Its primary function is partial protection of agriculturally managed area. The dike is about 2200 mm long, at the highest point of the dike is about 3 mm high.

In 2009-2010 it was built cycle path Horní Počáply-Vliněves-Zelčín a length of 18.6 km, which partially runs along the crest of the levee. During the construction has been found weak subsoil for the bike path on large parts of the structure including the levee. To improve the bearing capacity of the subsoil of asphalt pavement was used special stabilizate in the form of a mixture of fly ash and cement clinker from a nearby power station

Fig. 5: Vrdy - construction of levee using CCB (Power Plant Opatovice, 2014).

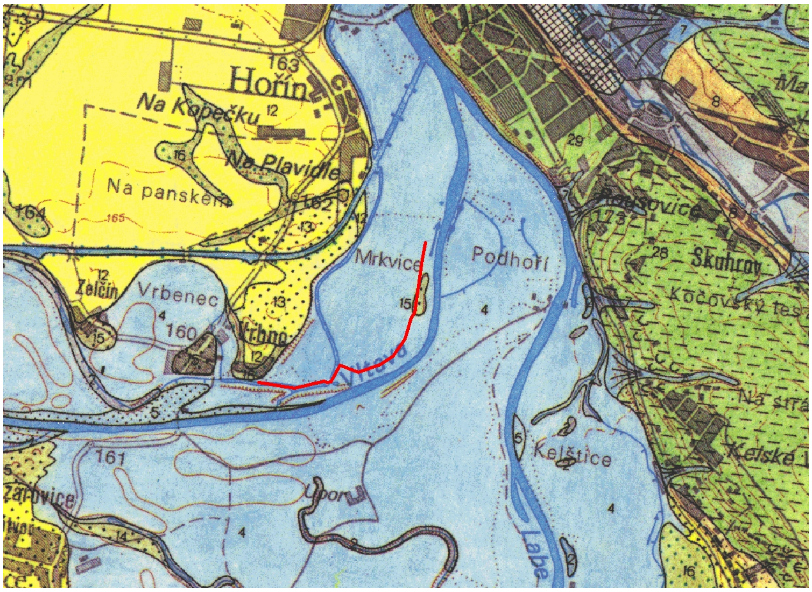


Fig. 6: Location of levee in the geological map 1:50 000, sheet Mělník 12-22.

in Mělník [7].

During the floods in 2013 there has been a breach this levee by overflow when exceeding of the design flow rate in the Vltava River. Water overspill violated in erosive action of downstream face of the dam (Fig. 7). In several places there was a shearing failure and landslide of the downstream face. Slide surface at the greatest failures to hit below the crest of the dam, respectively under construction layer of asphalt bike trail. The total number of failures dam reached 90, but most were minor disturbances. On the slide surfaces is apparent that is different resistance against shearing failure in fly ash stabilizer (higher) compared with the soil layers when saturated with water.

(a) the greatest of the many failures of downstream face of the levees in the length of approximately 25 m

(b) detail of exposed interior of the levee, including layers from fly ash stabilizate

Fig. 7: Failure of levee because of overtopping during the flood in 2013 (Mráz, 2013).

During the flood in 2013 there was overtopping of dikes and other places in the watershed of Vltava and Labe, respectively traffic roads using CCB as construction material (Fig. 8). There was no significant failures occurred, however. The difference between levee in Mělník and other structures consisted in using fly ash stabilizate greater extent, i.e. layers min. 40 cm or more. Layer of CCB at levee in Mělník is to maximal thickness of 25 cm.

Fig. 8: Flooding of base layer of still unfinished cycle paths from CCB during flooding (ČEZ a.s., 2013).

4 Conclusion

In the Czech Republic there are already a large number of examples of successful use of coal combustion by-products in embankments and dikes. At the same time, however, there has been documented cases where some construction problems occurred. It can say that is a clear causal ignorance of some specifics CCB or their understatement.

Using CCB to earth structures of water management can achieve their substantial price reduction. From the properties CCB used in the presented examples that the geotechnical properties can be better compared with soil especially in case of the shear strength, unconfined compressive strength and also the overall settlement of the dam crest. CCB advantage is the possibility of storage even at low temperatures.

The limiting factor for the use of certain types of CCB is a relatively small resistance to repeated contact with water and frost and reporting volume changes. Failures of constructions often stem from not detecting these properties due to neglect perform the required laboratory tests. Rarely does the problem of the possibility of leaching of toxic substances such as heavy metals.

Use CCB in earth structure of water management while reaching geotechnical and other desired properties can be recommended. Emphasis must be placed on the quality of the design which should be based on laboratory tests, the particular material used, or from in situ measurements on test sections.

The annual production of CCB is so high that it is still necessary to seek alternative ways of their use. The main advantage of using CCB is the replacement of natural raw materials (stone, natural gypsum, clinker partially), which has a positive impact on the environment, and lower, eventually zero costs for storing CCB as waste.

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Experimental Investigation of Durability of Concrete with Rubber Powder Exposed to Freezing Temperatures

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Abstract: This research is focused on concrete with rubber powder content exposed to low (freezing) temperatures. Rubber powder serves as air-entraining agent and should provide better freeze-thaw protection. On the other hand, rubber powder limits maximum compressive strength of concrete. The main purpose of this research is to find an optimal rubber powder content in order to satisfy needs for the minimal loss in strength of concrete as well as high freezing-thawing resistance. A freeze-thaw test was carried out on five concrete mixes with different contain of several rubber fractions to determine the optimal rubber grading. Specimens with rubber powder were tested on compressive strength, splitting tensile strength, tensile strength and moduli of elasticity.

Keywords: Freezing; Thawing; Rubber Powder; Compressive Strength; Air-Entrainment

1 Introduction

When temperature of concrete saturated by water drops below zero, the water held in capillary system of concrete freezes and expansion of concrete starts. After re-freeze of concrete, further expansion takes place. That repeated cycles of freezing and thawing has cumulative effect on concrete. There are several options how to make concrete less vulnerable to frost damage. The use of mix with lower water/cement ratio provides small capillaries. Such concrete has a low permeability and does not absorb so much water in wet environment. It also results in less water in pore structure that is likely to freeze. Other possibility on how to prevent severe frost damage to concrete is use of air-entrainment. In order to prevent concrete deterioration rising from freezing and thawing, the pore structure of concrete has to be modified. This ability is determined by amount of air voids within the cement paste. The larger pores filled with air are in concrete mass, the more durable concrete is. Freezing point varies with the size of pore.

Entrained air produces cavities in the cement paste so that no connections for water are formed within and the permeability of concrete should not be increased. The cavities never become filled with the products of hydration of cement as gel can form only in water. Air voids can be created by adding air-entraining agents. However, a few papers show successful attempts with granulated rubber as a new air-entraining agent. This could positively influence freezing/thawing resistance of concrete.

Since the water in large cavities starts to freeze, the formed ice generates surface tension which puts smaller pores into pressure. The pressure is higher the smaller the pore. So that freezing starts in the largest cavities and gradually extends to smaller ones. Gel pores are too small to permit the formation of ice, only there is temperature below -80 °C, so that in practice no ice is formed in them. However, the difference of entropy between gel water and ice, the gel water acquires an energy potential enabling it to move into the capillary cavities containing ice. The diffusion of gel water leads to a growth of the ice.

Thus, there are two source of dilating pressure. First one, freezing of water results in increase of volume (of approximately 9 percent) within the pore structure of concrete. The hydraulic pressure developed depending on the resistance to flow, i.e. on the permeability of the cement paste between the freezing cavity and a void which can accommodate the excess water.

The second dilating force in concrete is caused by diffusion of water. This diffusion is caused by osmotic pressure brought about by local increases in solute concentration due to the separation of frozen (pure) water from the solution. For instance, s concrete slab freezing from the top with also water access at the bottom will

Tab. 1: The numbers at the column of “Rubber powder content” represent the dosage of rubber fractions by percent (sorted by the size of the fraction).

Mix type	Description	Rubber powder content [%]
Reference	plain	0-0-0
Mix 1	with rubber	10-20-70
Mix 2	with rubber	20-50-30
Mix 3	with rubber	50-40-10
Mix 4	with air entrainment	0-0-0

be seriously damaged since its total moisture content could become greater than before freezing due to osmotic pressure.

There are two material characteristics that this research is trying to optimize when using rubber powder in concrete, the objectives are follows: to provide high freezing and thawing resistance concrete without any great loss in compressive and tensile strength (maximum up to 10%). This can be done by optimum dosage of rubber powder as an additive to concrete mix.

2 Mix Design

A standard C30/37 concrete mix was chosen as the reference. Three different concrete mixes were tested to determine optimal proportion of different rubber fractions. The fine (0-4mm), (4-8mm) and coarse (8-16mm) aggregates were used.

Three size of rubber powder, obtained from mechanical shredding of car tyres, were used. Combination of three different factions of rubber powder were used as an additive to concrete mix : 0-0,4mm; 0,4-0,8 mm ; 0,5-1,5 mm. The last fraction can be classified more as “rubber sand” then rubber powder.

Rubber powder was considered to be an additive to the designed mix, not a sand replacement. Replacing sand with rubber powder has the effect of reducing the compressive strength and elastic modulus [3].

Every concrete mix has the same batch of rubber powder, only proportion of three used rubber fractions differs in each batch. This paper derives partly from previous research on concrete with rubber content, where optimum rubber content was investigated. The optimum dosage was set at 0,8 % of weight of aggregate. The same dosage was used in this research. However, more research on optimal dosage is still needed.

3 Testing Methods – Summary

Standard cube form (150mm) was used for compressive strength test, splitting tensile strength test (after temperature cycling). Beams (100/100/400) were used for moduli of elasticity test (ultra-sonic test), tensile strength test. All specimens were stored, in water tank for minimum 28 days.

4 Air Content and Slump Test

Entrained air pockets provide a relief system for internal ice pressure by providing internal voids to accommodate the volume expansion caused by freezing water.

At this point, it is important to note that entrained air is not the same as entrapped air. Entrapped air voids are created during improper mixing, consolidating and placement of the concrete. These kinds of voids have severe effects on strength and durability of concrete.

The goal is to develop a system of uniformly dispersed air voids throughout the concrete.

Measuring of air content was done by pressure type air meter. Slump test was executed by ČSN EN 12350-2 standard. Mix proportions and the effect on the workability and air content of concrete are shown in table Tab. 2.

Tab. 2: Summary table of tests.

Test	Test Method	Specimen	Curing	Number of specimens
Compressive strength	ČSN EN 12390-3	Cube	Curing under water (28 days)	3
Splitting tensile strength	ČSN EN 12390-6	Cube	Curing under water (28 days)	3
Freeze/thaw test	ČSN 731322, ČSN EN 12390-6	Cube, Beam	Curing under water (28 days)	3
Moduli of elasticity (sonic method) + tensile strength test	ČSN EN 12504-4, ČSN EN 12390-6	Beam	Curing under water (28 days)	3
Air content	ČSN EN 12350-7	-	-	-
Slump test	ČSN EN 12350-2	-	-	-

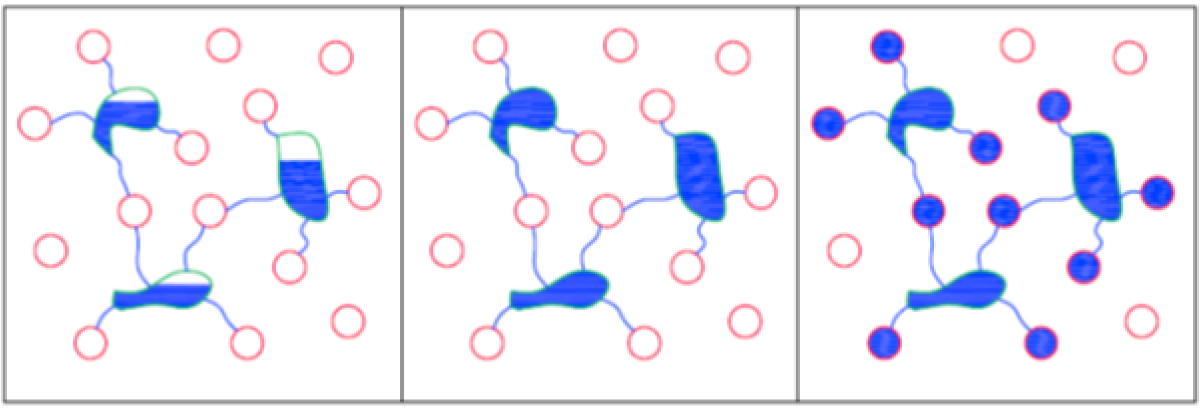

Fig. 1: Basic scheme of air-entraining principle in concrete.

Tab. 3: Table of specimens for slump test.

Mix type	Slump [mm]	Air content [%]
Reference	50 mm	4,2 %
Mix1 (10-20-70)	140 mm	5,8 %
Mix2 (20-50-30)	170 mm	6,0 %
Mix3 (10-20-70)	160 mm	5,8 %
Mix4 (air-entraining)	200 mm	5,8 %

(a) reference mix

(b) mix 1

Fig. 2: Slump test (ČSN EN 12350-2).

Tab. 4: Preliminary results of cube strength test and dynamic modulus of elasticity. Values are related to the reference specimens.

Mix type		Splitting tensile strength	Compressive strength
Reference mix	100 %	100 %	100 %
Mix1 (10-20-70)	98 %	85 %	82 %
Mix2 (20-50-30)	98 %	71 %	77 %
Mix3 (10-20-70)	97 %	71 %	84 %
Mix4 (air-entraining)	96 %	62 %	81 %

The result of the slump test indicates that fresh concrete with rubber powder content achieved a reduced slump, so the workability of fresh concrete rises. The results obtained from air content test also point at the fact that rubber content brings more air to the mix.

The reason for the improvement of workability by entrained air is probably that air bubbles act as a fine aggregate of very low surface friction.

5 Results

All tests are still running so only preliminary results are available at the moment of writing this paper. Only a few specimens have been tested so far. Especially tests with temperature cycling take more time (for determination of freezing/thawing resistance, 200 temperature cycles were settled). However, cube strength test are presented in Tab. 3.

The reduction in overall density indicates the presence of internal voids in concrete.

Compressive strength of specimens with rubber powder was reduced approximately about 17 % for specimens with rubber powder. Very similar results are obtained for specimen with air-entraining agent. The reduction again pointing at influence of air-entering which rubber powder brings.

Splitting tensile strength of specimens with rubber powder was higher than with specimens with air-entrainment. It was shown (see Tab. 4) that amount of air in both mixes is very similar. Yet, the loss in tensile strength is lower with rubber powder. That's a good advantage for rubber powder as a possible entraining agent.

Dynamic modulus of elasticity wasn't practically changed. These results will serve for the oncoming comparison with values of dynamic modulus after temperature cycling.

6 Conclusion

This paper has shown that there is a potential for using rubber powder as entraining agent as well as freeze/thaw resisting agent in concrete. The use of rubber powder in concrete has sustainable credentials in that it uses a waste product to enhance the performance of concrete.

All tests are still running so only preliminary results were presented.

The data of freezing/thawing resistance of concrete with rubber powder and air-entraining agent, which will provide ongoing research, are crucial for the next research. The results showed in this paper confirmed that rubber powder can be used as an air-entraining agent which can bring similar amount of air to the concrete mix as commonly used air-entraining agents.

Acknowledgement

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Recycled Aggregate and the Method of Mechanical Activation

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Abstract: This paper is a literature review on topic of recycled aggregate (RA) and mechanical activation method (MA). Basic recommendations for utilization of RA are summarized and examples of successful application of RA in road structures are presented. A method of MA is presented as well. This method can be utilized as secondary treatment for selected materials, especially concrete mix compounds.

Keywords: Recycled Aggregate; Mechanical Activation.

1 Introduction

Concrete is nowadays the most common building material for roads, buildings, bridges and other infrastructure [1]. In average, approximately 1 ton of concrete per capita is produced every year [1, 2]. Alarming reality is that sooner or later this volume is going to become a waste. In 2010 in countries of the European Union Construction and Demolition Waste (CDW) represents one third of entire waste production [3]. To act in line with sustainable development in certain areas, natural material is being partly or completely replaced by CDW. However, apart from the waste issue it is necessary to realize that for 1 ton of concrete 300 – 450 kg of cement is necessary. Unfortunately, cement is one of the materials with highest embodied CO_2 emissions [1]. Cement industry is responsible for 5 – 7 % of anthropogenic carbon dioxide production [1,2]. Therefore, there is global trend to develop so-called “green concrete” where in general cement is partially replaced by another material with lower embodied CO_2 emissions in comparison with other materials [2]. To deal with mentioned issues, there is an effort to re-use CDW in civil engineering again. Concrete waste is being used in the form of recycled aggregate (RA), other waste such as fly ash or granite powder are being used as concrete additives.

Unfortunately, current trend in utilization of recycled aggregate is rather “down-cycling” – material which originally had high quality is re-used for inferior purposes. This is not very efficient, therefore there are several research programs which try to prove quality of recycled materials and also studies which try to find better way of re-use of high-quality waste. In this paper, current knowledge about usage of recycled aggregate and few examples of good employment of RA are captured. The method of Mechanical Activation (MA), which may enable re-usage of concrete waste as cement replacement, is presented as well.

2 Recycled Aggregate and Its Employment in Road Infrastructure

Recycled Aggregate (RA) is gained from concrete waste by a process of several treatments. Blocks of material are crushed and reinforcement is separated. As a result, coarse and fine aggregate is obtained. The utilization of both components is limited and differs one from the other. Results from several studies are captured below.

The road constructions are suitable for utilization of RA for two main reasons. Firstly, road constructions represent huge volume and therefore it is possible to use large volume of RA. Secondly, the road reconstructions are great opportunity to recycle and re-use the material in-situ. Avoiding transportation is a benefit to environment and it means cost savings as well. Several possible employments of the RA can be found in road construction. RA or CDW in general is widely used in subgrade layer (Fig. 1), however for RA this is an example of down-cycling. In foreign countries there are several examples of utilization of RA in subbase course which means better evaluation of RA [4].

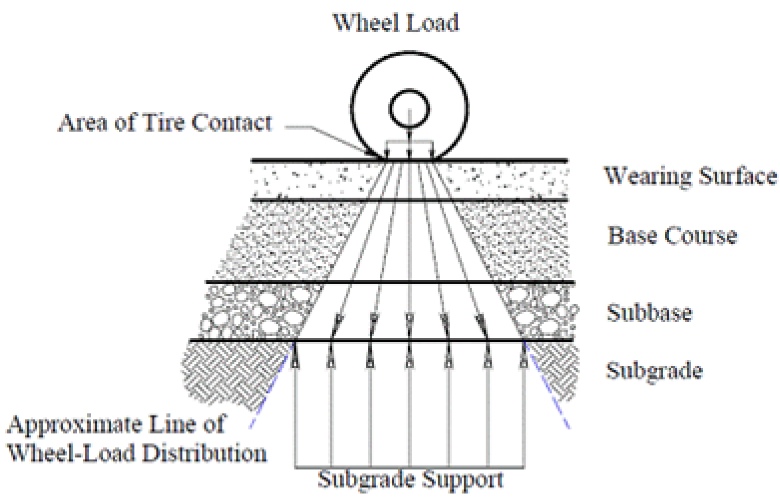

Fig. 1: Road structure profile [5].

The recycled aggregate can be further divided into two parts – coarse and fine aggregate. In general, concrete with recycled aggregate has worse rheological performance. This is a result of higher water-cement ratio which is needed if RA is used (compared to natural aggregate). The necessity of higher w/c ratio is caused by the rests on cement paste remaining on coarse aggregates. Also the presence of fine component raises the necessary volume of batch water. As these factors lead to worsen quality, several studies have been conducted and several rules or recommendation to utilization of RA were formulated.

A research from Great Britain shows that there is significant difference between replacement of fine recycled aggregate (FRA) and coarse recycled aggregate (CRA). From the results displayed in Fig. 2, it is obvious that natural aggregate (NA) can be replaced up to 60 % by CRA without important impact on the compressive strength. However, FRA noticeably influences the strength already in case of 20 % replacement NA by FRA. It has been proven as well that crushed bricks have very negative impact on final mechanical characteristics. Therefore its volume should be restricted to 10 % maximum [6].

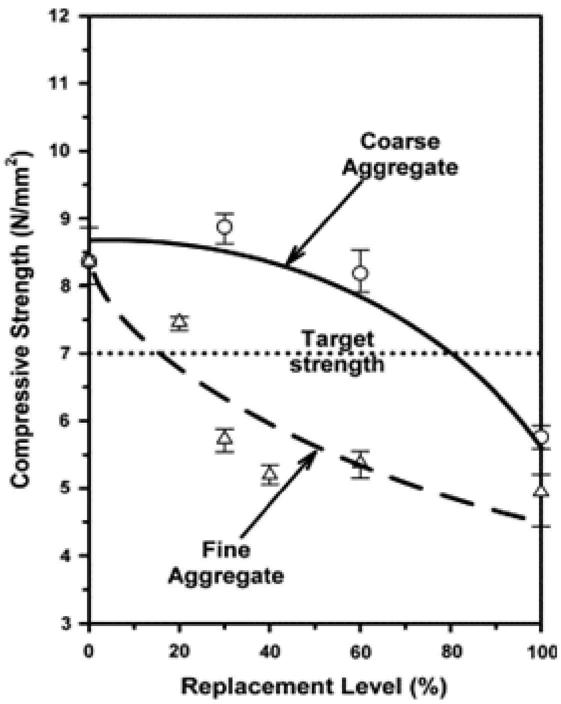

Fig. 2: Replacement of natural aggregate by fine and coarse recycled aggregate (adopted from [6]).

Since the worsen quality of concrete mix with RA is caused by cement paste remaining on surface of the aggregate, it is possible to improve the quality by secondary treatment of RA. The aim of such treatment is to remove the remaining cement paste from the surface of RA. There are several methods which can be used [7]:

- Mechanical removal by abrasion
- Combination of mechanical and thermal treatment
- Electrodynamic or electrohydraulic methods

Detailed description of methods can be found in cited literature. Other treatments are described in other papers, for example "two stage mixing approach" [8] and others.

3 Examples of Employment of Recycled Aggregate in Road Structures

In some countries, recycled aggregate has been successfully used in upper layers of concrete road structure. That shows that it is possible to meaningfully utilize RA.

An interesting example is reconstruction of A1 highway in Austria, where 100 % of material was re-used in new road structure. Material from old surface was treated, washed out of fine particles and sorted into several fractions (Fig. 3). These fractures were stored near to the site and used later to cement stabilized foundation slab and subbase concrete. Recycling in-situ saved 1,7 million km of heavy transportation and it prevented production of 1445 ton of carbon dioxide [9].

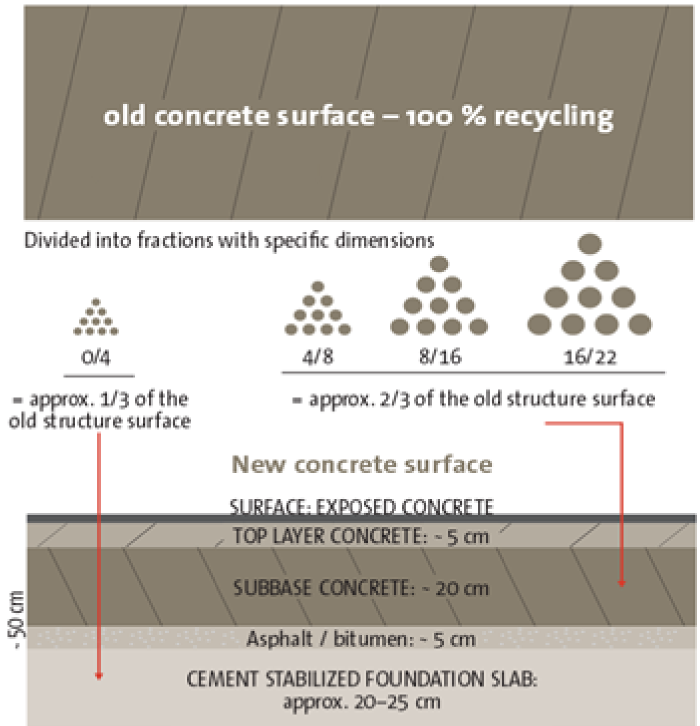

Fig. 3: Utilization of recycled aggregate in road profile of highway A1 in Austria (adopted from [9]).

In Nordic countries they focus a lot on research of utilization of recycled aggregate in road structures. In 2004, RA has been introduced in their national construction standard for road structures as construction material. Many realized projects proved that employment of recycled concrete is suitable solution for road structures, despite the fact that the material often does not accomplish the requirement of mechanical strength. Traditional testing methods are not convenient for this type of material, correct evaluation should be done according to methods respecting functional behavior of material.

A specific project has been realized in Norway. Trial section has been built between years 2003 and 2004 as a part of highway E6 in Melhus close to Trondheim. The highway is the main connection line between the south-east and the north of Norway, therefore it needs to support heavy traffic (ATD = 12 500). Crused concrete of two different fractions 0/100 mm and 20/100 mm has been used into sub-base structure of asphalt pavement. The recycled material came from eliminated prefabricated concrete panels, thus had high quality.

The structure has been monitored during the construction and after finalization as well and an increase of modulus of elasticity has been observed. From initial values E = 350 – 650 MPa (higher values are for fraction 20/100 mm) after one year and half of operation of the trial section of E6 highway, the modulus of elasticity grew almost twice E = 800 – 900 MPa (fraction 0/100 mm, values has been calculated retrospectively out of

Fig. 4: Material for recycling (adopted from [10]).

Fig. 5: Paving (adopted from [10]).

data from measurements of specimens executed by deflectometer FWD). High elastic stiffness values compared to natural material (ordinary gravel or crushed rock material) has been observed as well [10–12].

4 The Method of Mechanical Activation

The method of Mechanical Activation (MA) is a way of secondary treatment of materials. This method resides in mechanical crushing of materials. The effect is that more fine material with higher specific volume is gained. This increase of specific volume (meaning also fineness) has direct impact on reactivity of the material. Since the surface area is significantly increased, larger surface is open to chemical reactions. This consideration can be justified on behaviour of cement. Cement's specific surface is in the range from 250 to 600 m^2/kg. It is well known that cement with higher specific surface hydrates faster and better than more rough cement.

On example of coarse raw fly ash particle (Fig. 6) it is possible to observe positive impact of MA. By grinding, smaller reactive particles and smaller less reactive particles are obtained. Smaller reactive particles can better react in chemical reactions, less reactive ones can become a good filler between cement particles.

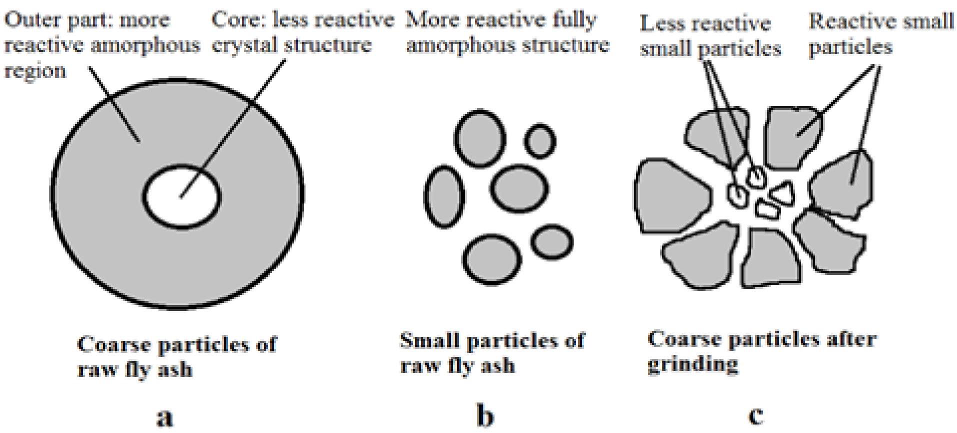

Fig. 6: Process of grinding (adopted from [13]).

However, higher specific volume brings along some less positive effects as higher w/c ratio while preserving consistency. Unfortunately, higher w/c ration has in general unfavourable impact. Therefore, the best balance between all aspects always needs to be found. , In research regarding MA of fly ash from Czech authors [13], the fineness close to the cement fineness (250 – 600 m^2/kg) has been defined as the most convenient one.

Result of MA strongly depends on type of the mill as well as on speed and duration of milling. There are various types of mills – hammer mill, ball mill, attritor mill, jet mill etc., Fig. 7. They differ in the mean of grinding. Detailed analysis on economical effectiveness could prove its different rating, but this is not a subject of this article. The majority of mills use some milling medium – hammers, balls; Medium is placed together with the initial material in a roller or another convenient container. The medium or entire container is then set in motion and the collision of milling medium causes crushing of the material. It is necessary to invest adequate power to actually cause the crushing. That is the condition of speed and grinding time and it highly depends on specific mill and material. As an example, research concerning mechanical activation of granite powder can be taken. The sample of granite powder grinded for 10 minutes with 350 revolutions per minute did not achieve the activation; the sample grinded for 60 minutes with 600 revolutions per minute was activated [13, 14].

Several experiments related to this issue were already conducted in different parts of the world. In general it is possible to observe that all of them have proven a positive impact of MA treatment on additions such as granite powder, slag or fly ash. In comparison with reference specimens containing non-treated addition, specimens with MA addition achieved higher both early age and final strength. The Young's modulus of elasticity has not been evaluated within these experiments [4].

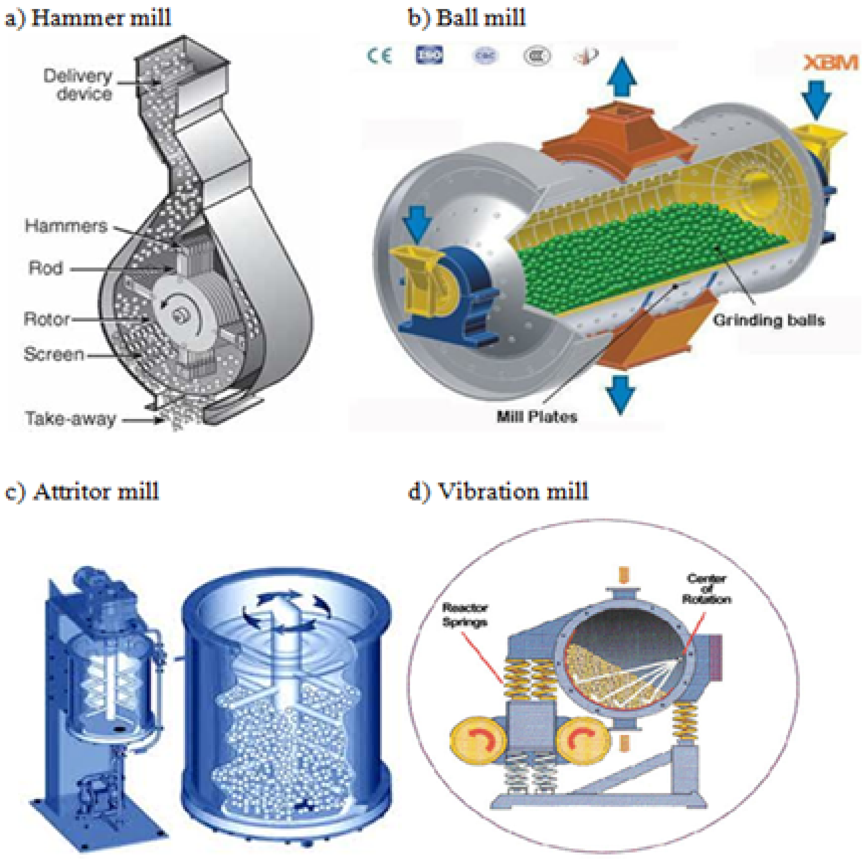

Fig. 7: Mill types (adopted from [4]).

The method of MA seems to be a suitable solution for secondary treatment of recycled aggregate as well. There is a hypothesis, that mechanically activated recycled aggregate could be used as partial cement replacement in concrete recipe. This will be an objective of further research in Czech Technical University in Prague.

5 Conclusion

The utilization of construction and demolition waste (CDW) is necessary in order to behave in line with sustainable development. Nowadays construction CDW is not utilized as it could be. Recycled materials are used mainly for "down-cycling", however there are already some flagships proving that meaningful employment of good quality recycled material is possible. CDW is currently used in the form of recycled aggregate (RA). It is necessary to make difference between fine and coarse recycled aggregate (FRA and CRA). While CRA can replace up to 60 % of natural aggregate without important impact on final strength of concrete, FRA negatively influences final strengths already in small volume (20 % replacement). Mechanical characteristics of concrete with RA can be improved by several treatment methods which lead to reduction of cement paste remaining on surface of the aggregate which is the source of worsen mechanical characteristics.

The method of mechanical activation (MA) means other possible option for employment of CDW/RA in concrete recipe. MA is a secondary treatment method which crushes material into fine ash with high specific volume. The possibility to use the material (RA) treated by this method as partial cement replacement will be an objective of further research in Czech Technical University in Prague.

Acknowledgement

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Influence of Selected Chemical or Nanochemical Additives (combined with Cement) and Alternative Hydraulic Binders for Treatment of Local Soils on Compressive Strength

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Abstract: The paper focuses on alternative solutions for processing locally available conditionally suitable soils treated by new types of binders or by selected chemical or nanochemical additives, which improve cement based soil treatments. It will be shown how new binders and selected additives can change final properties of stabilized soil materials used for pavement base layers or subgrade improvements. The paper focuses on assessment of these treated materials by means of traditionally applied test methods and requirements given in Czech technical specifications. Treated soils were tested with respect to required parameters for their future application in pavement structures – primarily utilized in base layers respecting the climatic zone and conditions in Central Europe. The tests performed were compressive strength after 7,14,28 and 60 days of curing and cyclic test of resistance to water and frost. All tests were performed on two types of soil (sandy, clay) with different dosage of used binders.

Keywords: Nanochemical Additives; Alternative Hydraulic Binders; Compressive Strength; Water and Frost Resistance.

1 Introduction

The economic parameters of a construction project still remain one of the determining aspects of road construction work. At the same time, more and more emphasis has been put on improving the non-renewable resource exploitation recently, including the use of less suitable but locally available materials; we have focused intensely on the project's impact on the environment, carbon footprint generation during construction and other aspects that were not usually as significant in the evaluation of construction structures and projects. The trend has been noticeable and, for a number of years, developed primarily in relation to reuse of materials incorporated in the pavement; materials that used to be classified as unsuitable for further processing, or materials we used to consider as waste materials even not so long ago. This paper pays attention primarily to the processing of unsuitable and conditionally suitable soils, using new (alternative) binders and additives to modify the traditional methods of soil treatment and stabilization by hydraulic binders. In the recent years, additives have been encountered which modify the hydraulic binder setting process or change the parameters of the soil used, thus allowing to process local materials even under extreme conditions when such materials would have been ruled out of application in the pavement structure otherwise. The new technologies have been embraced particularly in developing countries where good-quality road construction materials are scarce and, therefore, exploitation of on-site materials is desirable. It is currently striven to utilize such modern technologies in the conditions of the Czech Republic where alternative binders and chemical additives are applied to locally available soils that are presently classified as unsuitable, or conditionally suitable for further processing (usually due to a higher proportion of clay particles). Thus we try to change the existing soil treatment technology by hydraulic binders, particularly the commonly available Portland cement or slaked lime. The process of soil treatment by hydraulic binders has been known in the Czech Republic since the 1960's. The modification is presently used mostly for soils the mechanical physical properties of which fail to meet the requirements for processing within the pavement structure and which, therefore, must be modified, or replaced by a more suitable new material first. This process is always more demanding both economically and in terms of time. The current standard (ČSN

73 6133) distinguishes the soils which are suitable or unsuitable as construction materials, or determine the cases where the soils cannot be used as a construction material. Unfortunately, the standards do not reflect the option of applying new, alternative technologies which facilitate economical processing of even extremely unsuitable soils. Theoretically, there are no obstacles for processing almost any sort of soil; the modification of unsuitable soils by hydraulic binders has been perceived as too expensive so far and, in most cases, the soil is removed and replaced by a more suitable new material. However, this is changing under the increasing pressure for recycling and implementation of new technologies to allow processing even materials that would not have been possible to use in the past.

2 Modifying Additives and Hydraulic Binders

Modern alternative binders and additives focus not only on improving the existing methods but also on implementing completely new technologies which allow processing a broader range of materials. Alternative binders or selected chemical additives have several modes of operation. The first one is modifying the chemical environment of the material processed, the soil in our case, to make the material economical for further modification by standard hydraulic binders. Another option is modifying the hydration process as such to eliminate the effects of any undesired substances present in the soil. This results in modified final properties of the structure where higher strengths and improved resistance to frost and water are achieved. Two foreign additives, TerraSil and UPD, were selected for the comparison; they primarily prevent water from entering the structure, i.e. make the soil hydrophobic. Also Doroport, a Czech originating slow-setting hydraulic road binder the composition of which decelerates the increase of strength characteristics in time, was tested; the experimental study also included a ternary binder, Sorfix, which is developed in the Czech Republic by the Czech Technical University in cooperation with the Czech power company (ČEZ). As usual, a mix with no additives bound solely by cement was chosen as the reference mix. The project aimed to verify the expected (or, in other cases, declared) benefits of the individual additives in the conditions of the Czech Republic.

2.1 TerraSil [1,2]

This substance is a nanotechnological, water-soluble, 100 % organic chemical additive. The benefit of the additive can be seen particularly in hydrophobization of the mix where the resulting layer is highly resistant to water infiltration or not; this results in improved technical design parameters of the treated soil, particularly in case of frost and water susceptibility. The siloxane bond (Si-O-Si) facilitates the formation of a very thin, permeable membrane on the surface of soil particles, which is stable and chemically resistant while serving as waterproofing solution at the same time.

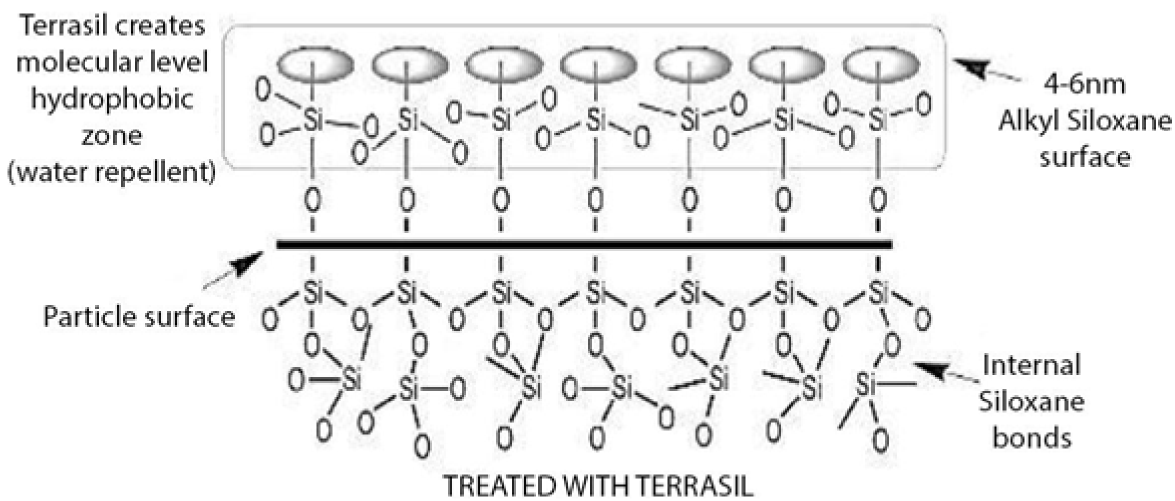

Fig. 1: Surface treated by TerraSil [1].

This type of modification is possible for most soil types. The additive itself is mixed with water and, most often, applied to the compacted pavement surface which it impregnates, thus rendering the surface hydrophobic.

The other option is mixing a diluted solution of the additive with the soil; in this case, the entire layer rather than just the surface is treated.

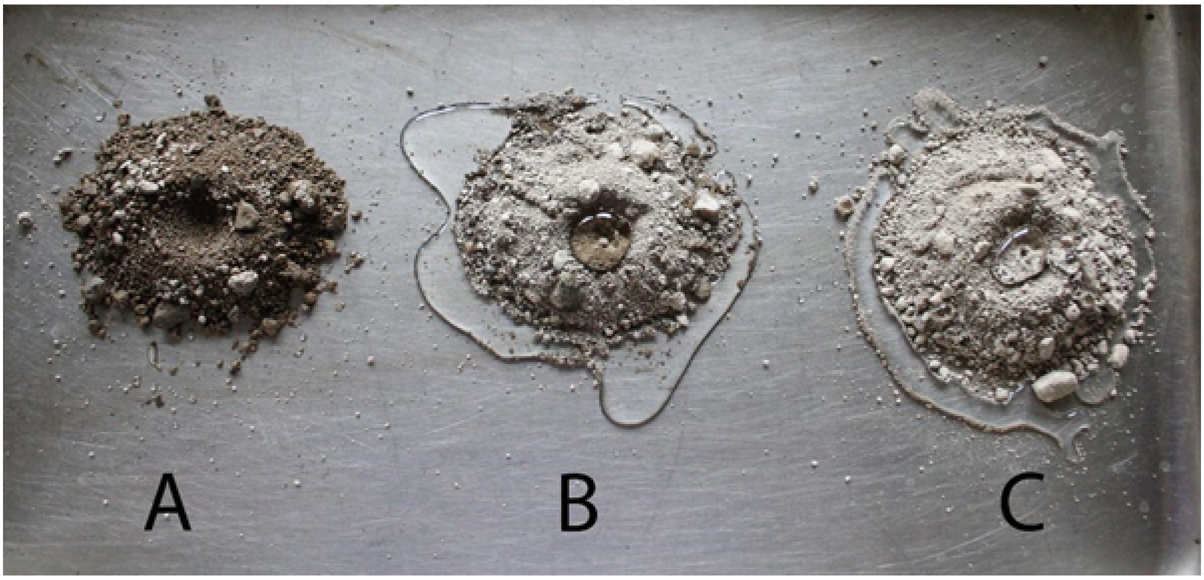

Fig. 2: Mixture treated by Cement 3 % and TerraSil A) 0 % B) 0,01 % C) 0,1 %.

2.2 UPD [4, 5]

This is a liquid additive working together with cement. Through ion exchange, it neutralises any undesired chemical environment in the soil while preventing water from entering the pavement structure. This allows processing even chemically polluted soils when leaching of harmful substances into the environment is prevented. A typical example is treating soils polluted by fuel, oil, lubricants and other undesired petrochemical substances which occur on the sites of old warehouses, industrial premises, airports etc. Such soils are currently handled as dangerous waste; according to the applicable legislation, they must be removed and stored at special landfills which is rather costly. Layers bound by UPD can be used most often in the pavement base and subbase. The improved resistance to cracks and propagation thereof to upper structural layers of the pavement as declared by the manufacturer is also a significant advantage. Higher cement content can be used without the need for providing expansion joints which are always a problem point in any structure (especially if found in the base and subbase). The additive declares the ability to process a broad range of soils; the only limit in this case is organic substances contained in the soil – these should not exceed 5 % which corresponds with the current recommendations of European standards. The additive is added straight in the mixing drum of the milling machine along with water.

2.3 Georoc Doroport TB25 [3]

This is a road binder type adjusted specifically to the conditions in the Czech Republic. The additive is a powder constituted by a suitable combination of ground clinker and hydraulic binders, developed for road construction purposes. It is currently used most often in base and subbase layers for soil stabilisation and improvement. Main advantages of Doroport include high resistance to sulphates, extended period of mix workability and a major benefit is the slower strength increase in time. The gradual binder hydration helps eliminate the possibility of shrinkage cracking and the binder proportion in the mix can be higher. A material advantage in CZ is the existence of practical experience where Doroport is perceived as a full-fledged tool for soil treatment. Like other hydraulic binders, Doroport is most often spread over the surface of the soil to be treated and a milling machine is used to mix it with the soil.

2.4 Ternary Binder - Sorfix

A ternary binder based on various types of fly ashes was developed in CZ based on fly-ashes typical for this country. The binder composition is covered by a patent. The binder is currently going through a further development stage. A sample was obtained within the framework of the CTU Prague research project. Most often, the combination involves cement and other components, in this case fly ashes from coal-burning power plants where the main effort is recycling the waste material while using it as a valuable binder for structures in road construction.

3 Evaluation of the Declared Characteristics

The practical part of the project focused on the treatment of two types of soils and subsequent evaluation thereof. The concerned soils were classified, according to the currently applicable national standard (ČSN 73 6133), as sand with a proportion of fine-grained loamy particles (S-F) obtain from section 9 of the modernisation of main Czech motorway D1 on the 72nd km, as well as clayey sand (SC) extracted at the construction site of the 3rd railway corridor in Veselí nad Lužnicí, Czech Republic. Depending on the soil classification, CBR and identified optimum moisture content, the quantity of the hydraulic binder was set to 3 % for the sand with a proportion of fine-grained soil and 6 % for clayey sand. The contents of the additives, UPD (0.15 l/m^3 of soil) and TerraSil (0.01 % per 100 % soil) which are most often added in combination with cement were determined on the basis of previous findings and technical consultations. In the case of Doroport hydraulic binder and Sorfic ternary binder, the contents were chosen on the basis of the quantity of cement applied to the reference mix to allow comparing the variants to one another.

3.1 Compression Strength

Compression strength and resistance to frost and water immersion were selected for the purpose of verifying the declared properties of the additives; this is in accordance with the current requirements of European standards EN 14227-10 Hydraulically bound mixtures – Specifications – Part 10: Soil treated by cement. Specimens were prepared on a Proctor compactor in compliance with standard EN 13286-50 Unbound mixtures and mixtures bound by hydraulic binders, Part 50: Methods for test specimen preparation by Proctor compactor or vibration plate. Compression strength was measured after 7, 14, 28 and 60 days, specimen classification is governed by the aforementioned standard EN 14227-10 while observing the slenderness ratio of 0.8. Freezing occurred during 13 cycles under the temperature of -20 +/-21 °C.

Cure	3% cement	3% cement+ 0,1% Terrasil	3% Doroport	3% cement+ UPD	3% Sorfix
	compressive strength				
7 days	2,89	2,13	2,27	2,52	0,20
14 days	3,22	2,74	2,54	3,09	0,38
28 days	3,14	3,39	2,83	3,07	0,68
60 days	4,28	3,24	3,60	3,66	1,05
resistance to water and frost	1,66	2,73	1,59	2,34	0,28
	52,85%	80,59%	56,22%	76,21%	28,04%

Fig. 3: Strength characteristics of sand with a proportion of fine-grained loamy particles.

The values measured for sandy soils with fine-grained loamy components clearly demonstrate the benefits of TerraSil and UPD which focus primarily on hydrophobization of the mix where the treated mix has a better resistance to water in the structure, and should prevent water from entering the individual grains at all. The fact was confirmed during the test of resistance to water and frost where the specimens treated by TerraSil showed roughly 30 % higher strength than the reference mixes bound solely by cement. In the case of UPD, the improvement was roughly 25 % against the reference mix. Also a slower but constant increase of strength in specimens bound by Doroport was observed. The application of the ternary binder appeared to be absolutely unsatisfactory when, compared to cement, the binder demonstrated significantly lower compression strengths and failed to meet the requirements for water and frost susceptibility.

The strength characteristics measured for treated clayey sand rather corresponded with the predicted behaviour of individual additives. Thanks to the high doses of the binders applied to the mixes, the reference mix showed partial decreases of strength. This phenomenon is probably caused by the too rapid strength increase in time and the occurrence of micro-cracks associated with the hydration heat release. A similar problem also

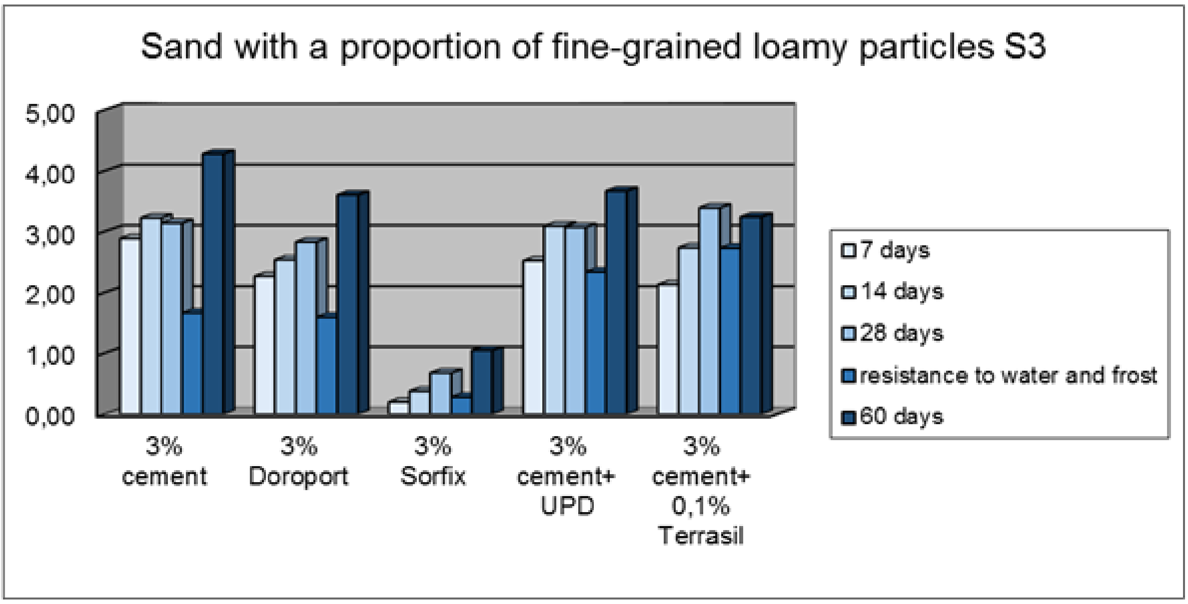

Fig. 4: Strength development in time for sand with a proportion of fine-grained loamy particles.

Cure	6% cement	6% cement+ 0,1% Terrasil	6% Doroport	6% cement+ UPD	6% Sorfix
	compressive strength				
7 days	3,24	3,25	3,13	3,78	1,74
14 days	4,06	4,75	4,25	4,77	1,79
28 days	3,69	4,36	6,07	5,78	2,05
60 days	4,87	7,24	6,55	8,90	2,34
resistance to water and frost	3,93	4,33	5,24	4,67	0,30
	106,50%	99,45%	86,42%	80,84%	14,57%

Fig. 5: Strength characteristics of clayey sand.

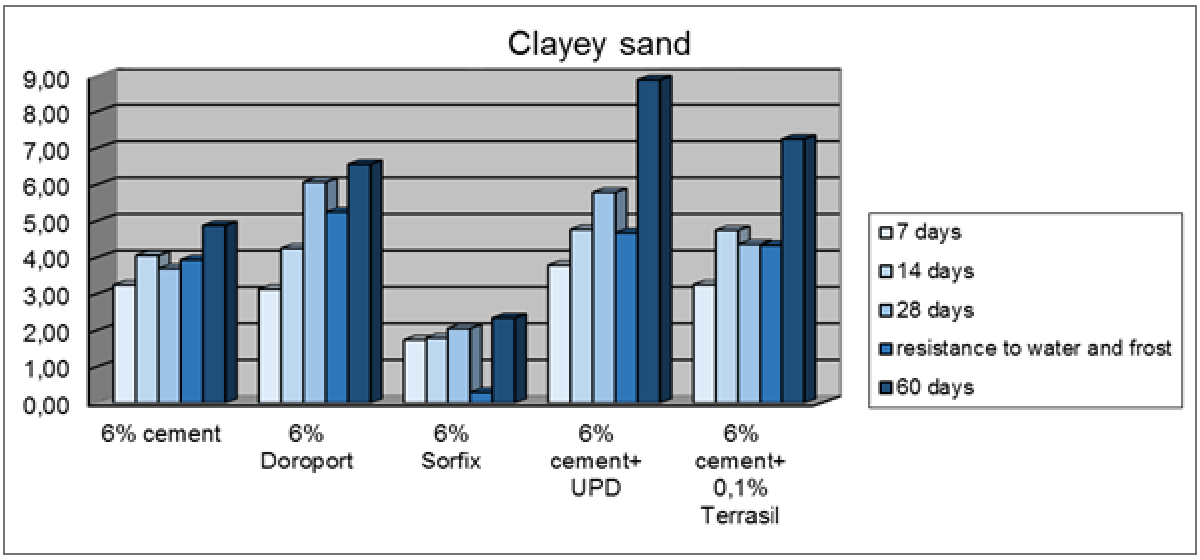

Fig. 6: Strength development in time for clayey sand.

occurred in the case of the mix treated by TerraSil where the additive focuses primarily on improving water susceptibility of the mix. From the point of view of resistance to water and frost, all specimens (with the exception of the samples bound by the ternary binder) met the applicable European standards. What is still surprising is the increase of strength in the reference mix where the strength after freezing exceeded the strength after 28 days. Compared to the other additives, the strength measured for the reference mix were ten times lower. The slow-setting road binder, Doroport, proved its potential again when the strength of the other soil increased constantly, too. Even for the other soil, the mixes bound by the ternary binder were shown to be unsatisfactory when a significant impact of water on such mixes was proven again.

4 Conclusion

With the increasing requirements for construction structures and economical aspects, environmentally compatible approach to construction thereof, modification of the existing soil treatment technologies presents a viable solution. This paper points out some of the possible alternative soil treatments by hydraulic binders where modern additives show a significant potential. For TerraSil and UPD, the impact on mix hydrophobization was verified where the additives showed a positive effect on resistance to frost; the impact on cement hydration as such where the additives had a beneficial effect on strength characteristic development in some cases was of interest as well. Prior to any possible practical use, it will be desirable to verify the effect of the additives on any specific soil types. The slow-setting hydraulic binder Doroport met the expectations and its contribution was proven even with higher doses of the binder when the mix did not degrade due to micro-cracks. Further, Doroport-treated mixes showed a slower but constant increase of strength in time. The results were also stable with respect to all parameters measured for both soil types. The ternary binder was observed to be unsuitable for fine-grained soil modification where a major role plays the water absorption power of the soil itself which cannot be influenced by the binder at all. Compared to the other binders, the specimens bound by the ternary binder demonstrated minimal improvements of strength characteristics in comparison to unmodified soil. This option failed to meet the requirements for resistance to frost and water completely. The binder is extremely unsuitable for fine-grained soil treatment; it will not be included in any further experimental work in its present form. In the future, the portfolio of the modified soils will have to be expended to allow more thorough evidence of the declared benefits of the individual additives. Similarly, it seems beneficial to obtain more additives and binders for a broader comparative test.

Acknowledgement

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PROCEEDINGS OF THE 1st INTERNATIONAL CONFERENCE RISEM pp. 44–49
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Screw Spike Pullout Test of Recycled Plastic

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Abstract: Plastics or composites with plastic matrix are widely used in industry as well as in railway engineering. In this paper is introduced ordinary recycled plastic used in the fabrication of storage pallets. The main part of this paper includes the screw spike pullout test of recycled plastic and its verification, whether this material would be suitable for the production of the railway sleepers. The outcome is ultimate strength of material, at which the screw is pulled out. The test was also conducted on conventional oak wood for comparison of the recycled plastic. The last part describes the recommendation for the use of chosen recycled plastic in the production of railway sleepers.

Keywords: Sleeper; Railway Superstructure; Recycled Plastic; Consumer Waste; Screw Spike Pullout Test.

1 Introduction

The article deals with railway sleepers, which are beside the slab track the most common type of railway support. The attention in the article is paid to a sleeper screw as a crucial part of fastening and also to the resistance of screw to pulling out from a sleeper. As is shown in Fig. 1, the sleeper screws fasten the rest of fastening on a sleeper.

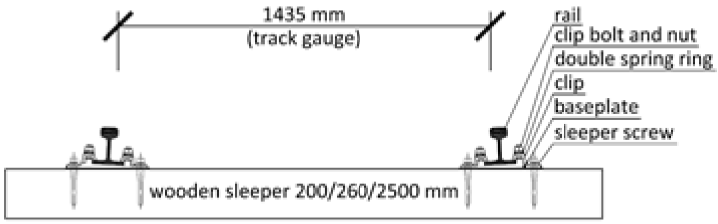


Fig. 1: Basic nomenclature of railway sleeper [1].

In the production of railway sleepers in the European Union the traditional materials are used e.g. as hard wood, concrete, steel and also the plastics as a new material for railway sleepers and bridge sleepers.

1.1 Why Is the Test Important

In a railway track a screw is strained from vertical force caused by influence of passing railway vehicles. Vertical force is generated from following reasons:

- the weight of a railway vehicle causes the lift of rail around the wheel in a distance from third to sixth sleeper from the wheel (Fig. 2). This is the case, where the screws carry the tensile stress. The rail lifting is consequence of track bed elasticity.
- In case with wooden sleepers fitted with inclined base plates the outer screws are strained by tensile forces. It is caused by excessive forces occurred by rail inclination (1:40 or 1:20). A steel base plate is laid on a wooden surface is laid a steel base plate. It can press down the wood on inner side of railway string [2].

Insufficient screw spike pullout strength or failure of fastening would lead to safety hazard. It caused many railway accidents in the past. For example the bad condition of railway fastening brought the derailment of four carriages in Kladno in the Czech Republic in 2013 [3].

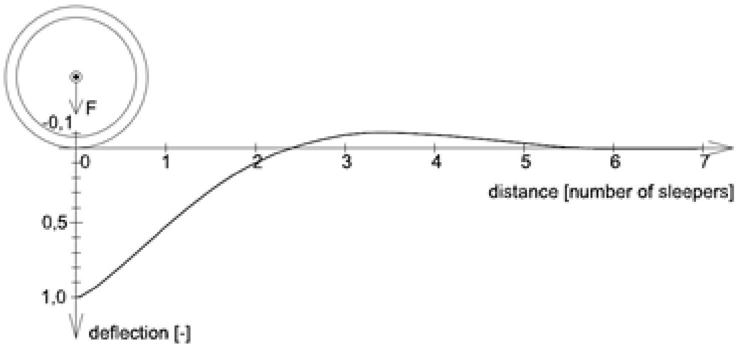


Fig. 2: The deflection of a rail string in the adjacent area of the wheel load [2].

2 Screw Spike Pullout Test

In this test the railway screw is vertically extracted from the specimens. The outcome is ultimate strength of material, at which the screw is pulled out.

2.1 Methodology

In accordance to EN 13481-2 the test is usually performed on a specimen prepared as a whole railway sleeper; however it was not possible in this paper in accordance to the test equipment of a laboratory. For this research the specimens were cut as parts of plastic pallets and wooden sleeper so as to prevent unwanted deviations in results. The advantage is the easy handling with the specimens. There was expected, that a screw is mounted in the adequate distance from edges of the specimens [4].

2.2 Recycled Plastic Chosen for the Test

The source of recycled plastic chosen for this test is mainly obtained as consumer waste placed in the containers and as technological waste from industry production. The material contains mainly polyethylene (PE), polyethylene terephthalate (PET), polypropylene (PE), polystyrene (PS) and other substances which contaminate the materials such as paper, textile or metals. This recycled plastic is based on PE (65%) [5]. Input plastic is formed in granules and is molded by the extrusion process with temperature about 220° C and the pressure about 100 MPa. The homogenized plastic melts down and is put in the steel forms. The shapes of the forms are presented as the finished products in Fig. 3 [4].

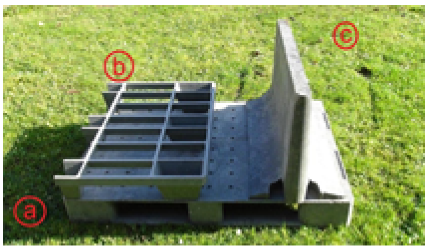


Fig. 3: The basic products made from the chosen recycled plastic: (a) a storage pallet, (b) a lightweight storage pallet, (c) a barrier.

2.3 Test Set Up

The test device was divided on following main parts:
- loading frame,
- set for mounting of screw (Fig. 4).

For loading was used the laboratory press EU 40 with maximum load of 200 kN attached with a string dilatometer for the measurement of displacement. The screw spike pullout test was performed with screw R1 (Fig. 4). Screw was mounted with the set of tools, which is shown in Fig. 4. The set is consisted from four steel parts: a load distribution plate, a block with hole for screw, a locking pin and bar, which is fastened into the loading frame. The important set up of the test is the length of screw shank screwed into the specimen. This factor could straight affect the ultimate strength of the material. It is worth mentioning that the typical length for real fastening in a railway track is about 122 mm. The set of tools in the test enabled the use of screw shank only with length of 115 mm.

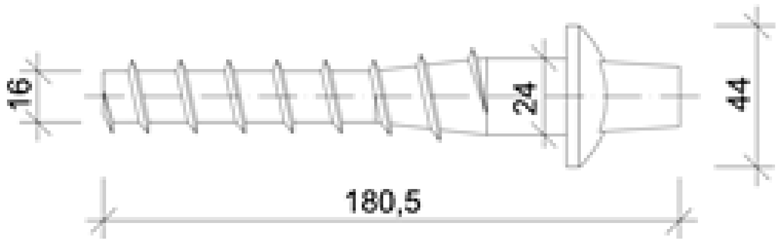


Fig. 4: Screw R1 – most common screw used in fastening in the Czech Republic (in accordance to SŽDC terms) [1].

(a) disassembled set for mounting screw

(b) assembled set for mounting screw

Fig. 5: The set for mount of a screw in a specimen and in the loading frame.

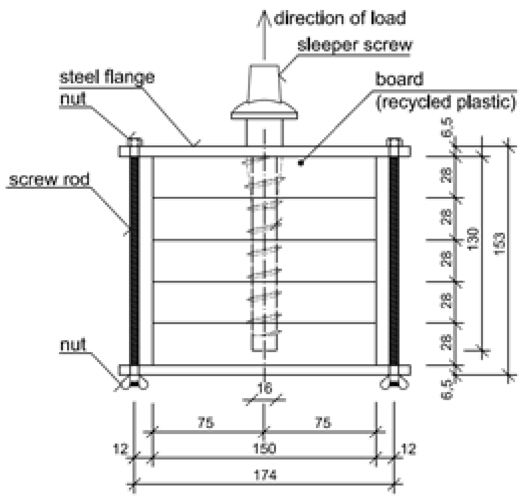
The screw spike pullout test was performed in the same way for recycled plastic as well as for oak wood with the following steps:
- loading frame,
- set for mounting of screw (Fig. 4).
- insertion of screw R1 into the specimen,
- mount of tools set on the specimen,

The load rate was performed manually in two following load steps:
- insertion of screw R1 into the specimen,
- mount of tools set on the specimen,

2.4 Specimens Variation

The specimens from recycled plastic were prepared in similar way as it is defined for concrete in EN 13481-2. The screw spike pullout test of other materials used in the production of sleepers such as concrete is performed directly on concrete sleepers or on specimens in accordance to technical standard EN 13481-2 which requires the distance of 100 mm between screw and edges of specimen [4].

The preparation of the recycled plastic specimens is described in the following text. It was complicated due to weaknesses in technology. In the process of recycling the plastic there is a need to fill the melt in the robust steel forms together with the simultaneous action of high pressure and temperature. The preparation of specimens was subjected to the amount of mass and availability of shapes. Since the company has got only forms for plastic pallets, the specimens had to be prepared from the pallets. And as there was the insufficient of plastic mass in the storage pallet, the samples were prepared from five stacked boards, which were cut directly from the storage pallets. As is shown in Fig. 4, the specimen consisted of five boards which were closed with steel flanges and screw rods. The screw-edge distance was about 75 mm. There was the assumption, that this construction made from boards is able to substitute the mass of material.



(a) design of the specimen [6]

(b) the specimen prepared for the test

Fig. 6: Specimen made of recycled plastic for the screw spike pullout test.

The second set of specimens was sampled from new oak sleeper generally used in railway tracks. In accordance to SŽDC (infrastructure manager in the Czech Republic) the shape of wooden sleeper was E1. It was produced and impregnated with creosote oil in 2008. The dimensions of specimens were 350 x 260 x 150 mm, with a screw placed in the centre. In accordance to SŽDC regulations the screw was placed more than 150 mm from the edge because of longitudinal cracks, however the specimen ends were fitted with gang-nail plates to strengthen the ends of specimen due to cracking.

For both tested materials total of three specimens were prepared. In relation to R1 screw (Fig. 4) and to the technical regulations of SŽDC the hole in the specimens was drilled with a diameter of 15 mm.

3 Results Evaluation

In this section the mean values of screw spike pullout test of chosen recycled plastic and oak wood are evaluated. The results of screw spike pullout test of other recycled plastics used in production of railway sleepers abroad are cited. It is necessary to mention, that results in Tab. **??** were not achieved with the same technical regulations i.e. conditions of the tests probably were not the same.

In the Fig. 6 load-displacement curves of oak wood and recycled plastic for each specimen are presented.

The curves in Fig. 6 are clearly divided into two groups in accordance to the material. The continuous curves in Fig. 6 show the test of oak wood, dashed curves show recycled plastic. Wood exhibited less displacement for the same load than it shows the recycled plastic. The difference was about twice more.

4 Discussion

The measurements of the material resistance to pullout force show several interesting facts about the screw spike pullout test and about the preparation of the specimens.

Tab 1 presents mean achieved values for the specimen made of recycled plastic boards. The values of pullout strength show that the assumption made for preparing the plastic samples from boards were chosen well. Fig. 7 shows the failure of plastic specimens and the effect of layers. All three plastic specimens cracked in the top three layers; two others were not destructed. In case that the screw was in compact mass of recycled

Tab. 1: The results of the screw spike pullout test of recycled plastic and oak wood. Comparison with recycled plastics used in the production of railway sleepers abroad [7, 8, 9, 10].

Tab. 2: The results of the screw spike pullout test of recycled plastic and oak wood. Comparison with recycled plastics used in the production of railway sleepers abroad [7–10].

property	recycled plastic	oak wood	Axion	Lankhorst	TieTek	Integrico
pullout strength [kN]	58,4	57,0	31,6	35,1	36,3	74,8
technical standard	EN 13481	EN 13481	ASTM D6117	EN 13230	not published	not published
assessment	ok	ok	already developed and applied			

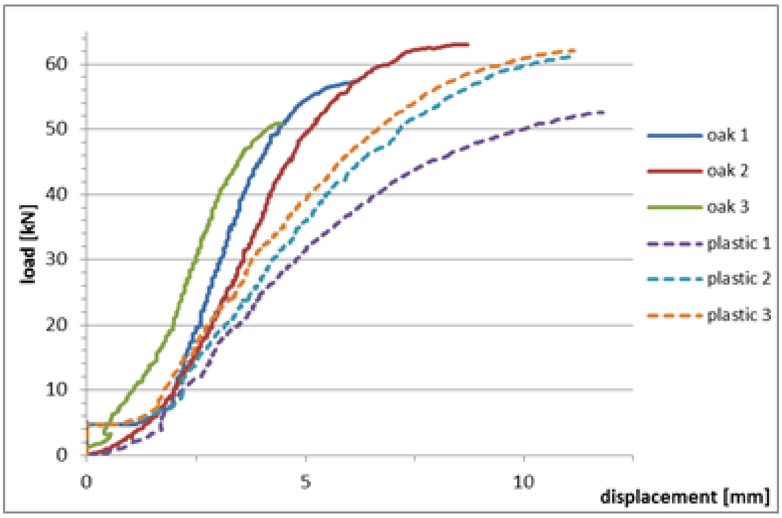


Fig. 7: The load-displacement curves of screw spike pullout test of oak wood and recycled plastic specimens.

plastic, the resistance to the extraction of screw would be even higher than the achieved value in this test. To other hand the greater mass in the production of plastic occur the greater proportion of cavities are in the porous core.

In Fig. 7 below the oak wood specimens are shown. There were no significant longitudinal cracks in the direction of the wooden fibers in the area of cracked hole. This means that the gang-nail plates at the ends of the test specimens do not affect the result of pullout strength. Their functions might occur during long-term tests in many years.

Fig. 8: The specimens made of recycled plastic and of oak wood after conducting the screw spike pullout test.

5 Conclusion

The initial test results show that the chosen recycled plastic used in the production of storage pallets might be suitable for the production of railway sleepers. The material has satisfactory ultimate screw spike pullout strength. It was proved in terms of ultimate force acted on a screw inserted in the recycled plastic samples. However the behavior of material requires special attention, because loaded screws exhibited considerable deformation of chosen recycled plastic. Together with high frequency of loading cycles under railway vehicles the excessive deformation could cause fatigue effect of recycled plastic. On the base of this fact, the additional test of plastics is recommended.

Acknowledgement

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Mechanical Properties of 100 % Recycled Half-Warm Asphalt Mixtures

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Abstract: In several countries bituminous mixtures are used in railway subballast instead of crushed stone because of their greater resistance to permanent deformation, durability, impermeability and better thermal insulating properties. Since the production of hot mix asphalts requires great amounts of energy and natural materials, a solution utilizing recycled asphalt pavement (RAP) only and demanding less energy was proposed and investigated. 100 % recycled half-warm asphalt mixtures (H-WMA) were produced by heating RAP up to 100 ±5 °C and were placed over a soft subgrade in a full-scale laboratory model of railway trackbed. After 500 000 loading cycles, specimens were taken from the bituminous layer to assess, whether any deterioration of the recycled H-WMA occurred due to the cyclic loading and to evaluate the mechanical strength of the mixtures as well as their resistance to water and frost. The tests revealed satisfactory mechanical strength and resistance to water and frost of the mixtures.

Keywords: Recycled; Half-Warm Asphalt Mixture; Railway; Subballast; Strength.

1 Introduction

The main function of railway track bed is to transfer forces induced by rail traffic from track superstructure further to track subgrade. Any extensive permanent deformations, which would lead to track geometry deterioration, shall not occur. Bearing capacity of railway track bed is to a certain extent dependent on bearing capacity of subgrade, parameters of which could be significantly affected by exposure to water and frost, especially if the subgrade is formed by water and frost sensitive soil or rock. An appropriate design of materials and thicknesses of railway track bed structural layers should thus:

- ensure sufficient load-bearing capacity of railway track bed,
- prevent rain water from penetrating to the subgrade,
- provide frost protection of the subgrade.

Furthermore, the material used in subballast should be itself resistant to water and frost, so it keeps its parameters stable.

In 1950's several national railway administrations started to investigate possible application of asphalt mixtures as a substitute or a supplement of granular subballast layers. In some countries, e.g. USA, Italy and Japan, the bituminous subballast has become a common solution, especially for high-speed or heavy traffic tracks. Experiences gained in last decades indicate that bituminous subballast contributes to optimal drainage of rainwater. The asphalt mixtures covered by ballast bed do not experience any excessive weathering or deterioration due to oxidation or extreme temperatures. Furthermore, any failures typical for road pavements were not observed. This fact is attributed to relatively low pressures induced on the surface of subballast by rail traffic in comparison with pressures induced by heavy trucks on the surface of asphalt pavement [1]. Several studies have also shown that higher resistance to permanent deformation and high durability of bituminous subballast contribute to decreased track maintenance costs compared to tracks with conventional granular subballast [2]. Moreover, if the bituminous subballast is used, the overall thickness of subballast and formation protective layers can be significantly decreased and thus notable savings in consumption of crushed aggregates can be achieved [3]. Consequently, total life cycle costs of the track could be even lower, than costs of a track with conventional subballast.

Nowadays, a significant effort is made to find out ways how to decrease amounts of energy and natural materials necessary for production of asphalt mixtures (i.e. aggregates and bitumen). Technologies aimed at maximum use of recycled materials as well as technologies utilizing lower production temperatures are being developed. Obviously, such important alternations in production process affect the resulting performance of asphalt mixtures. High percentages of recycled asphalt pavement (RAP) containing aged bitumen can contribute to worse compactability of the mixture which consequently leads to lower volume density and thus higher air void content [4]. The reason is gradual hardening of bitumen which is in the road pavement exposed to oxygen, ultraviolet radiation and changes in temperature [5]. Similarly, with lower production temperatures the mixtures become less workable and compactable. It was proved that in a temperature range between 40 °C and 120 °C the achieved volume density of asphalt mixtures containing only RAP increases linearly with the compaction temperature [6]. It is also well known that higher air void content may negatively affect mechanical performance of the mixtures as well as their resistance to water [5, 7]. Another negative effect is a greater permeability which is unfavorable from the point of view of subgrade protection against water. Air void content of 8 % is considered the upper limit for impermeable layers [1]. Presented study focuses on 100 % recycled half-warm asphalt mixtures (HWMA) comprising only crushed RAP and which are to be laid and compacted at a temperature of 100 $\pm$5 °C. In compliance with abovementioned experience a special emphasis was placed to investigate the effects of water and frost on performance of the mixtures.

2 Material and Test Specimens

Two full-scale laboratory models of railway trackbed with a layer of 100 % recycled H-WMA instead of granular subballast were built and subjected to cyclic loading. The recycled mixtures were produced by heating up RAP without any additives. The aim of the presented laboratory investigation was to assess, whether any deterioration of the recycled H-WMA occurred due to the cyclic loading and to evaluate the mechanical strength of the mixtures as well as their resistance to water and frost. Furthermore, for one of the mixtures permeability tests were carried out to assess, whether the 100 % recycled H-WMA can form an impermeable layer which would prevent rainwater from penetrating to the subgrade.

2.1 RAP Analysis

The RAP used in this study, was collected at the Středokluky asphalt plant at a covered stockpile of crushed and sieved material, fraction 0/22 mm. The binder content in RAP was determined by centrifuge extractor according to the CSN EN 12697-1 standard. The extracted aggregate was sieved and its particle size distribution was determined in accordance with the process given by the CSN EN 933-1. The softening point of recovered bitumen was evaluated from a ring-ball test (CSN EN 1427). The maximum volume density of the RAP was determined in compliance with the CSN EN 12697-5 standard.

Three samples of RAP were tested for binder content. It ranged from 5.5 to 5.7 %, with an average of 5.6 %. This value can be considered as standard. Only moderate variation of binder content among samples indicates good homogeneity of the RAP. The particle size distribution of the extracted aggregate is shown in Fig. 1 together with the limit curves for grading of hot recycling asphalt concrete for base layers (ACP). The content of fines smaller than 0.063 mm exceeded the upper limit by approx. 0.8 %. The ring-ball test revealed average softening point of the recovered binder of 68.1 °C, which is a typical value for aged bitumen in RAP. The maximum volume density of the RAP was 2469 kg.m^{-3}.

2.2 Preparation of Test Samples

The test specimens were core-drilled from layers of 100 % recycled H-WMA, which formed a part of two full-scale laboratory models of railway trackbed. The models allowed for assessing whether any excessive deterioration of the recycled mixtures occurred due to the cyclic loading. Furthermore, the models served to evaluate the mechanical strength of the mixtures as well as their resistance to water and frost when compacted in conditions similar to those in a real track (malleability of subgrade, compaction in a 150 mm thick layer).

The recycled H-WMA was prepared by heating the RAP up to 105.0 and 97.2 °C for application in the two respective models. After heating up, the mixture was placed and being compacted by a vibrating plate for 30 minutes. The composition of both models was as it is shown in Fig. 2. The subgrade was simulated by a

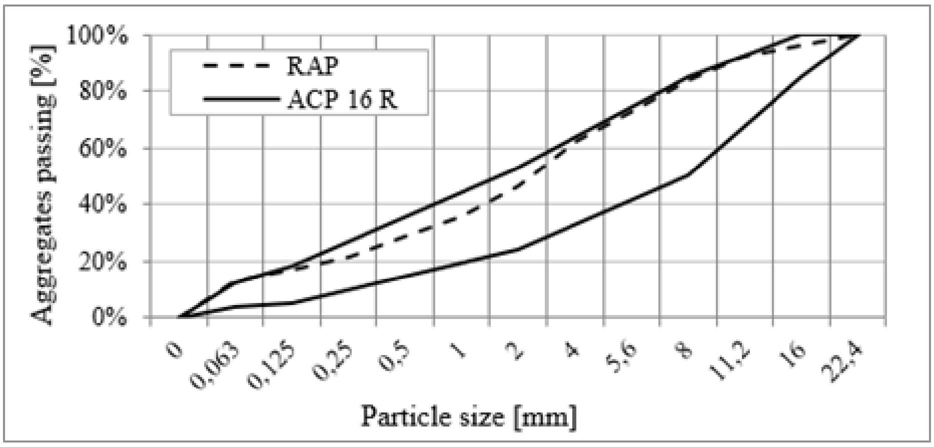

Fig. 1: Particle size distribution of extracted aggregate.

200 mm thick layer of low plasticity clay (CL), over which a 150 mm thick layer of 100 % recycled H-WMA was placed. The top layer was formed by railway ballast with a grain size of 31.5/63 mm. After completion the models were subjected to 500 000 loading repetitions each simulating passage of 22.5 t vehicle axis. During dismantling, cores were taken using a core drill with an inner diameter of 100 mm. The cores were then cut to achieve desired height of the specimens for laboratory tests described in part 3.

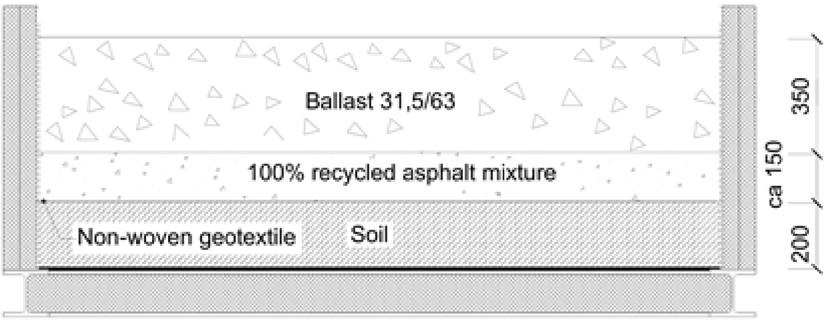

Fig. 2: Composition of full-scale laboratory models with a layer of H-WMA.

3 Test Methods

In selection of the laboratory test methods used in the investigation the intended use of the recycled HWMA layer and its placement within the railway trackbed as well as the nature of the material were taken into account. To assess the mechanical strength both compressive strength test and indirect tensile strength test (ITS) were employed. The compressive strength test (according to CSN EN 13286-41) is used in the Czech Republic to assess the mechanical strength of hydraulically bound mixtures in railway trackbed. The required minimum compressive strength of the mixtures is 2.5 MPa [8]. Since the parameters of asphalt mixtures change significantly with the temperature, the compressive strength tests were carried out at constant temperature of 20 $\pm$2 °C. The specimens used for the compressive strength test were cut to a height of 100 $\pm$2 mm. The ITS test is used worldwide to evaluate the mechanical strength of asphalt mixtures. Modified versions of the test apply for hydraulically bound mixtures and cold recycling mixtures as well. The test versions differ in test sample dimensions and sample conditioning prior to the test. In this study cylindrical specimens with a diameter of 100 mm and a height of 63.5 $\pm$2 mm were tested after being stored at a temperature of 15 °C for at least 4 hours. The minimum required ITS of cold recycling mixtures ranges from 0.3 to 0.7 MPa depending on the binder employed.

Tests for evaluation of resistance to water and frost were based on abovementioned test of mechanical strength. The freeze-thaw resistance test was performed according to the CSN EN 14227-1 standard. The freeze-thaw resistance is defined as a compressive strength of specimens, which are capillary soaked and then subjected to a given amount of freezing and thawing cycles. Each freezing and thawing cycle comprises a 6

Tab. 1: Compressive strength and freeze-thaw resistance of the 100 % recycled H-WMA.

	Compacting temperature	Air voids	Compressive strength	Freeze-thaw to water	
	[°C]	[%]	[MPa]	[MPa]	[%]
Model 1	105.0	11.7	7.50	-	101
		10.3	-	7.60	
Model 2	97.2	14.5	6.38	-	121
		12.4	-	7.7	

±0.5 hours long freezing period in which the sample is put in the freezer keeping a given temperature and an 18 ±0.5 hours long thawing period in which the sample is placed on a sorptive pad at a temperature of 20 to 25 °C. The sorptive pad is partly submerged in water so as capillary soaking of the sample is enabled. The number of cycles and the temperature of freezing depend on the intended use of the mixture. In this study 10 freezing and thawing cycles were applied with the lowest freezing temperature of 20 ±2 °C. According to the CSN EN 14227-1 standard, the mixture shall retain at least 85 % of its compressive strength after being subjected to freezing and thawing cycles. Furthermore, the regulation of the Czech Railway Infrastructure Administration SŽDC S4 [8] requires compressive strength after freezing and thawing at least 3.5 MPa.

The resistance to water was tested in accordance with the procedure given in Technical specifications of the Ministry of Transport of the Czech Republic TP208 [9]. It was expressed as a ratio between the ITS of a wet set of specimens and the ITS of a dry set of specimens (ITSR). The wet specimens were immersed in water at a temperature of 20 ±2 °C for 7 days prior to the test. The dry specimens were tested immediately after the water used for cooling the core drill bit had dried out. The minimum required resistance to water for cold recycling mixtures bound with cement and bitumen emulsion or foamed bitumen is 75 % of the ITS.

Besides the tests for mechanical strength and resistance to water and frost, the volume density and the air void content of each specimen were evaluated. Furthermore, permeability of specimens coredrilled from the model 2 was determined using the constant head test method described in CSN EN ISO/TS 17892-11.

4 Results

The compressive strength of 100 % recycled H-WMA core drilled from the two respective laboratory models (Model 1 and Model 2) is shown in Tab. 1 together with compacting temperatures, air void contents and freeze-thaw resistance. In the model 1, the asphalt layer was compacted at a higher average temperature of 105.0 °C, while in the model 2 the average temperature was by approximately 8 °C lower. This difference is a presumable reason of differences in air void content and compressive strength of the mixtures. The absolute average values of compressive strength of the core drilled specimens from both fullscale models greatly exceed the requirements for minimum compressive strength of bound mixtures for railway trackbed (2.5 MPa).

On the other hand, both mixtures showed relatively high content of air voids, which in the case of model 2 even slightly exceeded the maximum limit for air void content in cold recycling mixtures [9]. As it was already mentioned some previous studies have shown that high air void content can negatively affect mechanical strength of asphalt mixtures and especially their resistance to water [5, 7]. Therefore, there were concerns about the freeze-thaw resistance of the 100 % recycled HWMA, since in the test specimens are subjected to combined effects of water and frost. However, as it can be seen from the values listed in the last two columns of Tab. 1 any negative influence of freezing and thawing on the compressive strength of specimens was not observed. In model 2 the compressive strength of the set of specimens, which were subjected to freezing and thawing, was even higher than the compressive strength of the specimens which were not subjected to freezing and thawing. A possible explanation of this observation might have been the different average air void content of the respective sets of specimens. In Fig. 3 the compressive strength of individual specimens (including specimens subjected to freezing and thawing) was plotted against the air void content. Even though some exceptions are visible, a general trend of decreasing compressive strength with increasing air void is obvious.

The results of ITS tests are listed in Tab. 2 together with compacting temperatures, air void contents and

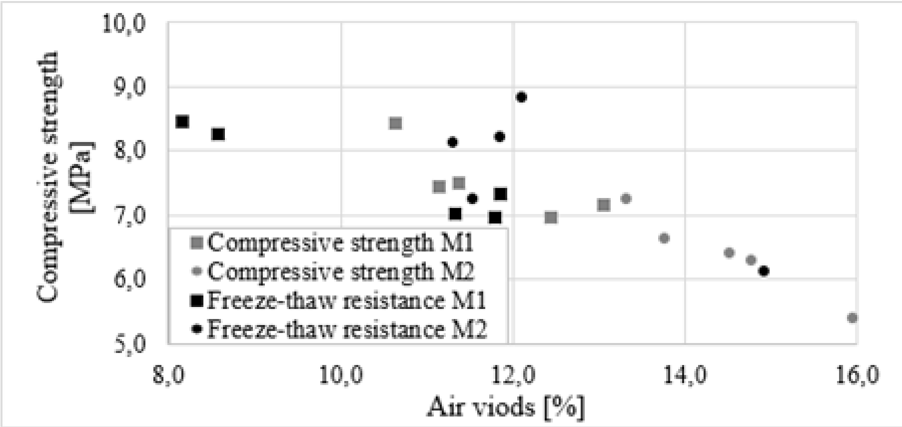

Fig. 3: Compressive strength against air voids.

Tab. 2: Indirect tensile strength and resistance to water of the 100 % recycled HWMA.

	Compacting temperature	Air voids	Indirect tensile strength	Resistance to water	
	[°C]	[%]	[MPa]	[MPa]	[%]
Model 1	105.0	11.7	1.43	-	134
		10.5	-	1.92	
Model 2	97.2	14.6	1.31	-	98
		13.2	-	1.29	

resistance to water. Model 1 showed again higher mechanical strength than model 2. The respective average values of the ITS were 1.43 MPa for model 1 and 1.31 MPa for model 2. As it was already mentioned, the minimum required ITS for cold recycling mixtures ranges from 0.3 MPa to 0.7 MPa. From this point of view, specimens taken from both laboratory models performed satisfactorily. Even though especially specimens from model 2 showed again relatively high air void content, their resistance to water was sufficient as it can be seen from the values listed in the last two columns of Tab. 2. The wet set of specimens taken from model 1 showed significantly higher average strength (by 34%) compared to the dry specimens taken from the same model. The results could be again influenced by variation in air void content; however it is probably not the only reason. Inequality of compacting temperature and binder content within the mixture might have also affected the results.

The permeability tests revealed an average coefficient of permeability of 6.0e-5. The soil permeability classification stated in [8] classifies materials with coefficient of permeability lower than 1.0e-6 as low permeability and materials with coefficient of permeability higher than 1.0e-6 as permeable. Based on obtained results the 100 % recycled HWMA can be classified as a permeable material. It is likely that the relatively high permeability of the mixture was given by higher air void content of the specimens. As it was already mentioned, air void content of 8 % is considered the upper limit for impermeable layers, while the specimens tested for permeability showed average air void content of 13.3 %.

5 Conclusion

Two full scale laboratory models of railway trackbed with a subballast layer of 100 % recycled HWMA compacted at a temperature of 100 ±5 °C were built in laboratory conditions and subjected to 500 000 load cycles simulating passage of a 22.5 t heavy axle. Afterwards, cores were drilled from the asphalt layer to evaluate the mechanical strength of the mixtures and their possible deterioration due to cyclic loading. Further tests were performed to assess whether the mixtures are satisfactorily resistant to effects of water and freeze.

Based on compressive strength and ITS test it is possible to say, that the mechanical strength of the 100 % recycled HWMA is satisfactory for use in subballast layers. Even after being subjected to 500 000 load cycles in a full-scale laboratory model of railway trackbed, the compressive strength of the mixtures was significantly greater than minimum compressive strength required for hydraulically bound mixtures to be used railway trackbed. The ITS of the specimens of 100 % recycled HWMA taken from both laboratory models exceeded the required minimum ITS applicable for cold recycling mixtures by 104 % and 87 % respectively.

A slight difference in mechanical strength of the specimens taken from the respective laboratory models was probably caused by a certain difference in compacting temperatures in both models (approx. 8 °C). In the model 2 the recycled HWMA was compacted at 97.2 °C and the air void content was close to 14 %, which is the maximum limit for cold recycling mixtures. However, in spite of the relatively high content of air voids, any negative effect of water and freeze on the mechanical strength of the specimens was not observed.

Based on abovementioned findings it is possible to state, that from the point of view of mechanical strength and resistance to water and freeze, the 100 % recycled HWMA are suitable for use in railway subballast. However, if compacted at 100 ± 5 °C, the mixtures do not form an impermeable layer and thus do not prevent the rainwater from penetrating to the subgrade.

Acknowledgement

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CTU in Prague, Czech Republic, September 23, 2015

Influence of Alternative Binders on Properties of Cement Composites

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Abstract: The paper deals with utilization of secondary products as a substitution of Portland cement in cementitious composites. This is a research focused on application of secondary products in building industry. Series of specimens were manufactured with partial substitution of Portland cement by micro-grounded recycled material, fly-ash, or their combination. Specimens with dimensions $40 \times 40 \times 160$ mm were subjected to flexural loading and fragments from flexural tests were tested in compression.

Keywords: Recycled Materials; Substitute Binder; Micro-Ground Recyclate; Fly Ash; Mechanical Properties.

1 Introduction

Utilizations of recycled materials is one of European Community priorities. Waste management plans established in the Czech Republic aim to achieve re-use of 75 % volume of the construction waste. The 7th Environmental Action Programme for Europe promotes increase of recycling and re-use of materials. In accordance with these requirements utilization of the secondary raw materials is investigated, such as recycled concrete. Crushed concrete is a typical example of construction and demolition waste it is generally used as substitution aggregate. It appeared that recycled concrete after grinding to very fine particles has also hydraulic properties. So that it could be used as an alternative binder. There are other alternative binders as geopolymers – slag, fly ash, etc. Questionable matter is influence of particular binders, their interference or synergy. The investigations focused on effect of micro-ground recycled concrete, fly ash and cement and their proportioning on mechanical properties of resulting composite.

2 Experimental Program

The experimental program relates to previous investigations concerned in utilization of secondary waste products in cementitious composites [1, 2]. Experiments focused in determination of basic mechanical properties of cement composites with partial substitution of Portland cement by micro-grounded recycled concrete and its combination with fly-ash. The specimen sets with substitution from 10 % to 50 % were manufactured. Each set comprised three specimens with dimensions $40 \times 40 \times 160$ mm which were at the age 28 day tested in bending. The fragments from bending tests were used to determine compressive strength. The results showed influence on compressive strength decrease both for micro-grounded recycled concrete and combination of micro-ground recycled concrete and fly-ash. Flexural strength for combination of substitutive binders increased compared both to Portland cement and micro-grounded recycled concrete.

This part of investigations focused in substitution by fly-ash only. Five sets of specimens were manufactured with substitution of Portland cement from 10 % to 50 %. Prism specimens $40 \times 40 \times 160$ mm were after 28 days of maturing in water dried, measured and weighed. The flexural strength was measured on prisms in a three-point bending test; compressive strength was determined on fragments of prisms in a similar way as in previous investigations.

Tab. 1: XDR phase analysis.

Compound name	Semi quant [%]
Quartz	63
Calcite	2
Portlandite	2
Chlorite-serpentine	2
Albit low	20
Gypsum	-
Muscovite	11

2.1 Origin of Alternative Binders and Their Properties

For manufacturing of specimens a micro- grounded recycled concrete from railway sleepers was used and fly-ash from electrical power plant Mělník. Description of both materials' properties follows.

2.1.1 Micro-Ground Recyclate

The input material for manufacturing of micro-ground recyclate was crushed concrete from railway sleepers. The sleepers originate from cancelled precast-plant railway. A jaw crusher Metso Nordberg LT 105 was used for crushing of sleepers.

Manufacturing of the micro-ground recyclate was performed in research centre of the company Ecological Investment Group s.r.o. using self-designed and self-built device TRITON M – II. The device is a common contra-rotating grinder with two vertical-axis rotors – so called disintegrator. The diameter of grinding rotors is 395 mm. Grinding was executed in two stages under different conditions. The main reason was that the rotors were not intended for grinding of concrete and were made from relatively mild steel. With bigger grains the abrasion of rotors was significantly higher. The first stage – rough grinding with the aim to decrease the grain size to less than 1 mm was performed with relative circumferential speed of rotors 160 mm/s. The relative circumferential speed in the second stage – fine grinding was 215 mm/s. Micro-ground recyclate prepared with this procedure was analyzed using laser granulometry (Fig. 1) to determine grain size distribution and X-Ray diffraction (XRD) analysis (Tab. 1) to identify particular phases in the fine–ground material.

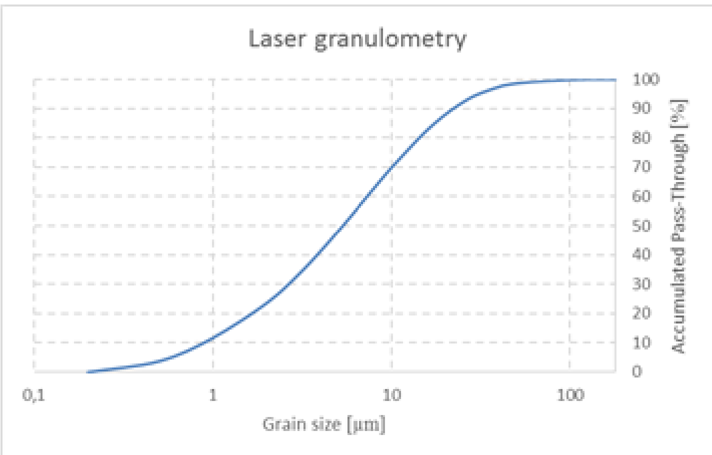

Fig. 1: Laser granulometry of tested micro-ground recyclate.

2.1.2 Fly Ash

In the investigation program fly ash from power plant Mělník was used. The Mělník fly ash is composed of 50.3 % Silicon dioxide SiO_2, 31.5 % Carbon dioxide (Al_2O_3), 2.33 % Calcium oxide (CaO), 6.96 % Iron(III)

Tab. 2: Composition of mixtures with water/cement ratio 0.4.

Blue	B0	B10	B20	B30	B40	B50
Portland Cement	100 %	90 %	80 %	70 %	60 %	50 %
Micro-ground recyclate	0 %	10 %	20 %	30 %	40 %	50 %
Fly ash	0 %	0 %	0 %	0 %	0 %	0 %
Green	G0	G10	G20	G30	G40	G50
Portland Cement	100 %	90 %	80 %	70 %	60 %	50 %
Micro-ground recyclate	0 %	0 %	0 %	0 %	0 %	0 %
Fly ash	0 %	10 %	20 %	30 %	40 %	50 %
Red	R0	R10	R20	R30	R40	R50
Portland Cement	100 %	90 %	80 %	70 %	60 %	50 %
Micro-ground recyclate	0 %	5 %	15 %	25 %	35 %	45 %
Fly ash	0 %	5 %	5 %	5 %	5 %	5 %

oxide (Fe_2O_3), 4.06 % Titanium dioxide (TiO_2), 1.02 % Potassium oxide (K_2O), 1.16 % Magnesium oxide (MgO) and less than 1 percent of other oxides.

2.2 Composition of Specimens

Particular mixtures comprised Portland cement, water, fly-ash, eventually micro-ground recycled concrete. Water/binder ration is same for all mixtures and equals 0.4. Composition of mixtures is given in Tab. 2.

2.3 Results

Results of the bulk density, compressive strength and flexural strength are bellow. The blue line states for mixture with addition of micro-grounded recycled concrete, the green line for mixture with fly-ash, the red line for combination of alternative binders.

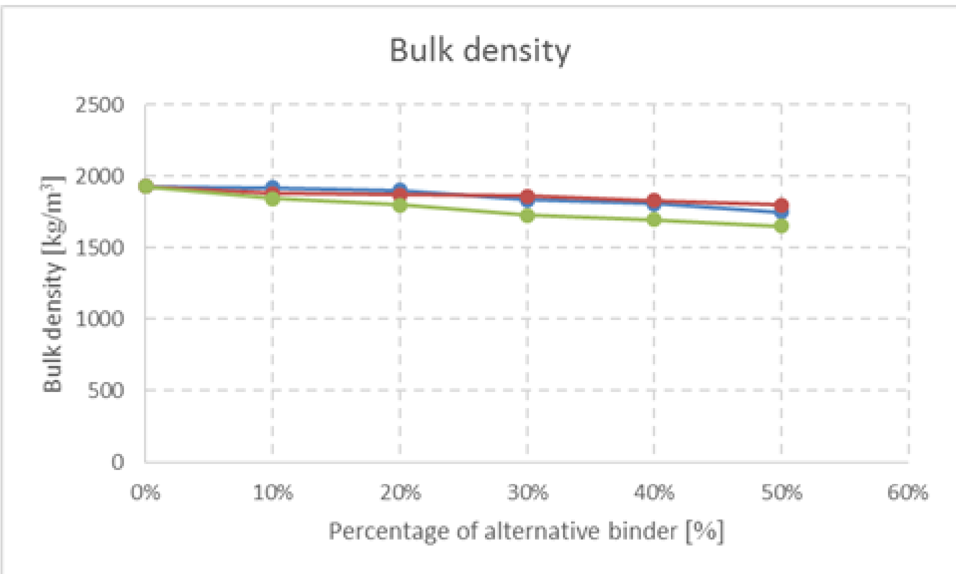

Fig. 2: Relation of bulk density on alternative binders content.

Specimens with alternative binders have lower bulk density compared to specimens with Portland cement only. The mixtures with fly-ash have lower bulk density than specimens with micro-grounded recycled concrete.

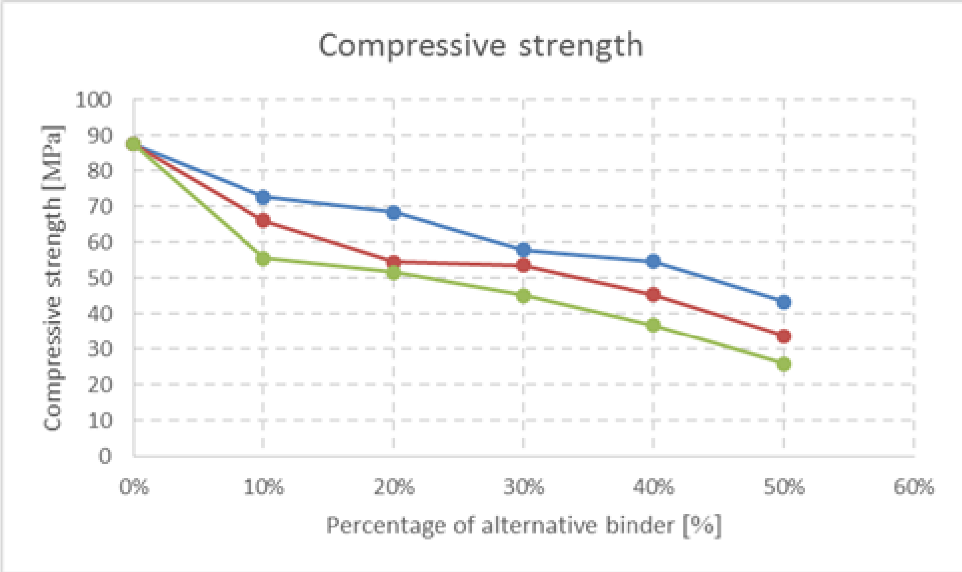

Fig. 3: Relation of compressive strength on alternative binders content.

From the graph follows that utilization of alternative binders lead to decrease of compressive strength, for fly ash the decrease is more significant.

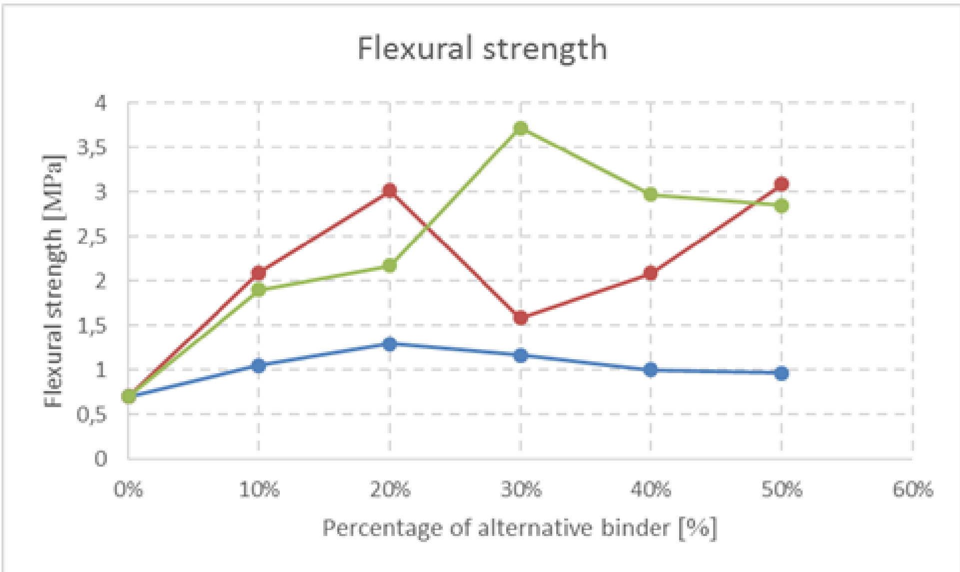

Fig. 4: Relation of flexural strength on alternative binders content.

Flexural strength increases for any type of alternative binders compared to specimens made from Portland cement. Fly-ash effect on strength is higher than effect of micro-grounded recycled concrete, their combination shows also good results. Combination of micro-grounded recycled concrete and fly-ash (substitution 30 % and 40%) provided high scatter caused obviously by shrinkage of the cement paste. This is proved also by nonstandard fracture plane (see Fig. 5). Further research of properties of combination of alternative binders is needful; it will provide larger set of results to investigate its effects on cement composite.

3 Conclusion

Use of micro-grounded recycled concrete and fly-ash adversely affects compressive strength of the composite but positively influences flexural strength. Fly-ash significantly decreases compressive strength whereas

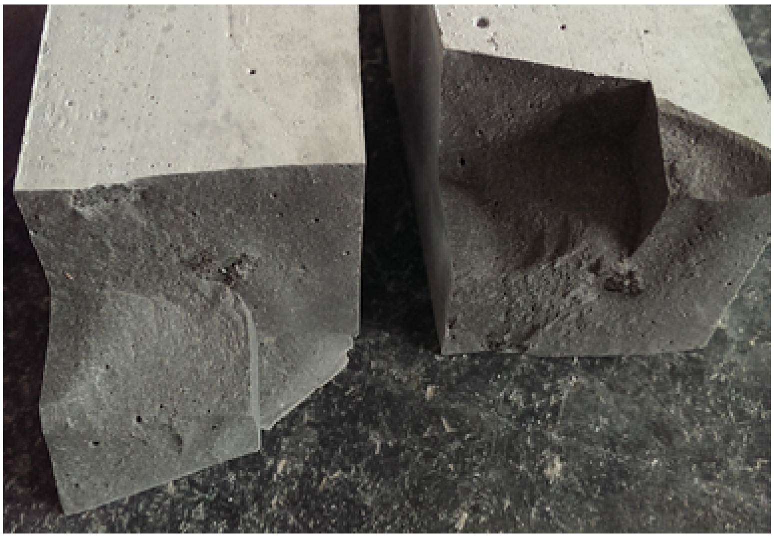

Fig. 5: Non-standard fracture plane after three-point bending test.

it increases flexural strength. Micro-grounded recycled concrete effect is opposite. Their appropriate combination could optimally affect both strength and it may become the best variant for application in construction industry.

By partial substitution of Portland cement by alternative binders the composite cost can be lowered and at the same time tribute to sustainable building by saving of primary sources.

Acknowledgement

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Deformation Properties of Asphalt Mixture with R-Material Addition

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Abstract: Plenty of current road structural materials use additives reached from recycling process of various sources, for example used tires or milled asphalt mixture. Using of these additives is conditioned by improvement of asphalt mixture functional properties. Paper is focused on stiffness measurement of asphalt mixture with r-material addition. Stiffness is property of asphalt mixture which describes deformation resistance of mixture. In this study was used four point bending stiffness test according European standards and for comparison was made measurement of elasticity modulus according Australian standards. For comparison there was also made stiffness measurement of asphalt mixture without r-material addition in same laboratorial and time conditions. In conclusion is clearly described influence of r-material on deformation properties of asphalt mixture.

Keywords: R-Material; Asphalt Mixture; Stiffness.

1 Stiffness of Bitumen Bonded Materials

Asphalt mixture is one the most used construction material for roads in almost every part of the world. For correct design of road structure are material characteristics needed. Behavior of materials bonded by bitumen is mainly visco - elastic for specific temperature conditions. Properties of asphalt mixture which are mainly studied are for example fatigue resistance, permanent deformation resistance, water resistance and also complex modulus of mixture.

Paper is focused on last of these properties, complex modulus or stiffness modulus describes material response on sinusoidal loading. Measurements and calculations of stiffness in the paper are according European standard [1]. According this standard, stiffness modulus is numerical value of complex modulus. Complex modulus *(E*)* is calculated from two elements, real element of complex modulus *(E_1)* and virtual element of complex modulus *(E_2)*.

$$E_1 = \gamma \times \left(\frac{F}{z} \times \cos(\varphi) + \frac{\mu}{10^3} \times \omega^2\right) E_2 = \gamma \times \left(\frac{F}{z} \times \sin(\varphi)\right) \tag{1}$$

$$|E^*| = \sqrt{E_1^2 + E_2^2} \tag{2}$$

where: γ - sample shape factor; μ - mass factor; F - force; z - displacement; φ - phase leg.

In this study was used equipment which is able to measure flexural stiffness and modulus of elasticity according Australian standard [2]. This measurement was chose only for comparison of complex modulus and elasticity modulus value. For elasticity modulus measurement was chosen only one frequency but it was done in every studied temperature condition. Equations of flexural stiffness *(S)* and modulus of elasticity *(E)* are below.

$$S = \frac{\sigma_t \times 10^3}{\varepsilon_t} E = \left[\frac{F.z}{\varphi.w.h}\right] \times \left[\frac{3Sw^2 - 4Lw^2}{4h^2} + k \times (z + \upsilon)\right] \tag{3}$$

where: σ_t - tensile stress; ε_t - tensile strain; w - width of sample; h- height of sample; Sw - support span width; Lw - loading span width; k - actual shear stress divided by average shear stress; υ - Poisson's ratio.

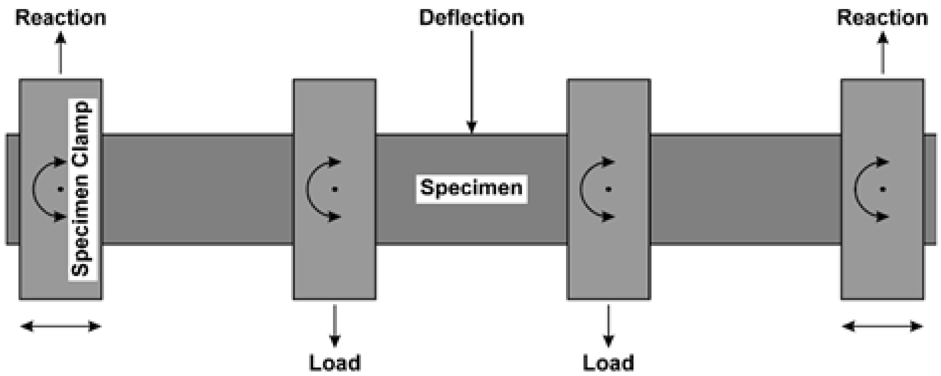

Fig. 1: Side view on loading scheme of 4-PB apparatus [3].

Tab. 1: Results of basic asphalt mixture measurements.

mixture	AC $_{11}$	AC $_{11}$ + R_{10}
binder content [%]	5.4	5.4
air void content [%]	3.0	3.9
void content filled by bitumen [%]	81.1	76.4
indirect tensile strength ratio [%]	85	86
wheel tracking slope [mm/10^3cycles]	0.06	0.06
proportional rut depth [%]	4.45	4.68

Both these standards uses four point bending method with prismatic beam specimen. Size of specimen was 380 × 50 × 50 mm and loading scheme of equipment could be seen in Fig. 1.

Beam is loaded in two inner points and it is fixed in two outer points in vertical direction. Load is applied with sinusoidal shape by inner clamps and frequency of loading could be set from 0.1 Hz to 50 Hz. Value of complex modulus is stiffness of beam in 100th loading cycle and control strain should be no more than 50 micro strains. Temperature conditions were set according Slovak design method TP 02/2009 [4] which are average temperatures during season of the year (winter 0 °C, spring and autumn 11 °C, summer 27 °C) .

2 Studied Materials

In this study was measured asphalt mixture which is one of the most used road construction material for surface and binder course of roads in Slovakia. Asphalt concrete with continual gradation curve has maximal size of grain 11 mm and used binder was road bitumen with penetration 50 / 70 with adhesive additives. There were made two variants of mixture, first variant was ordinary asphalt concrete (AC 11) and second variant was asphalt concrete with 10 % of r-material (AC 11 - R10). Depend on results there is option for using of asphalt concrete with r-material on heavy loaded roads, the most important criteria are deformation characteristics of mixture.

R-material was reached by milling of surface course of second class road. This surface layer also consists of asphalt concrete 11. Binder content in this R-material was 5.3 % of total mass. R-material was added to mixture in amount of 10 % of total mixture mass and r-material replaced part of aggregate with size 0 – 2 mm.

These mixtures were also tested on basic properties of asphalt mixtures according standards for type testing. Results of measurements could be seen in Tab. 1.

The results of measurement show that both variants of mixture are good enough for using in pavement construction also in heavy loaded road sections. Differences between variants of mixture are minimal and after these tests could be claimed that addition of r-material in asphalt mixture has good influence on characteristics of mixture. According these results there was assumption that also stiffness of these variants reaches similar values.

3 Evaluation of Measurements

The temperature conditions were same for both mixtures and both test of stiffness and test of elasticity modulus. Frequency conditions for stiffness measurement were chosen in recommended range (0.5 Hz; 1 Hz; 5 Hz; 10 Hz; 20 Hz; 50 Hz), frequency of elasticity modulus measurement was set up to 10 Hz because this frequency is close to standard recommended frequency 8 Hz. The tests were done on same specimen and by same testing equipment. At first was done stiffness measurement for every frequency with one temperature and after that was measured modulus of elasticity with same temperature. Strain amplitude 50 $\mu\varepsilon$ was used for stiffness and elasticity modulus measurements. The measured phase angle was higher during stiffness measurement (about 3 degrees) and also strain amplitude was little bit higher (about 0.1 MPa) than during elasticity measurement. For the both mixtures were tested four samples. The results were in statistical range $\pm$ 10 % of average value. In the Fig. 2 and Fig. 3 could be seen complex modulus for both mixtures in whole temperature and frequency range.

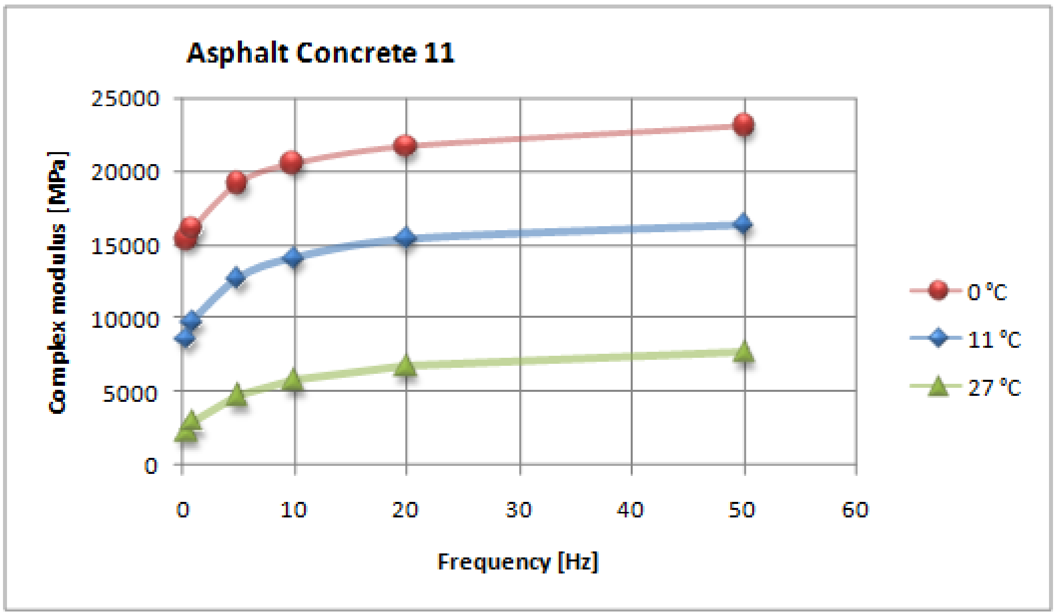

Fig. 2: Complex modulus of mixture Asphalt Concrete 11 in different frequencies and temperatures.

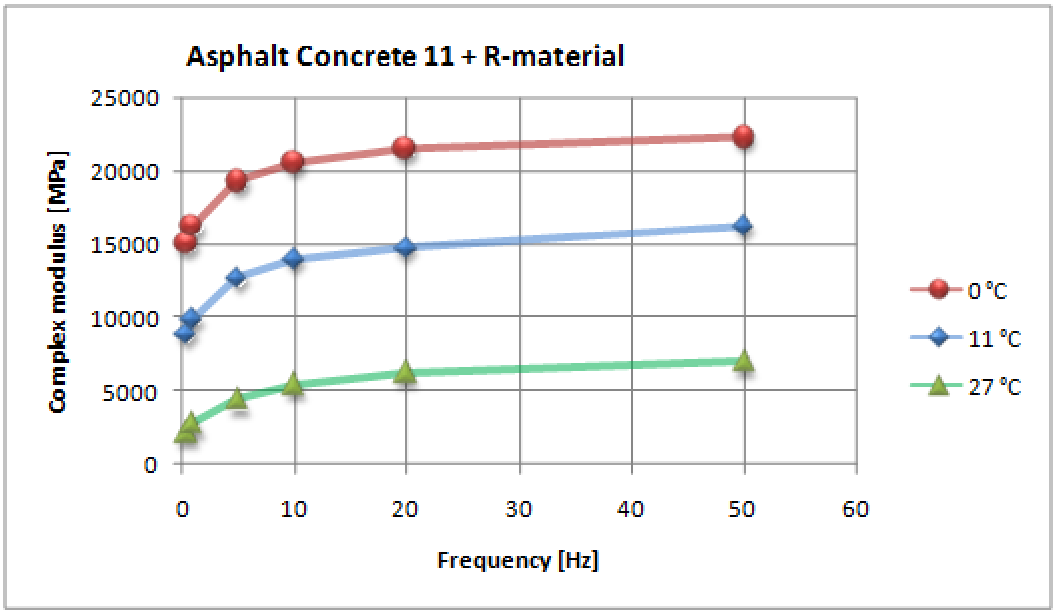

Fig. 3: Complex modulus of mixture Asphalt Concrete 11+R_{10} in different frequencies and temperatures.

According to these results, stiffness of mixture with r-material addition is little bit lower than stiffness of traditional reference mixture but it's still comparable. In every studied temperature, mixture with r-material had similar values of stiffness like the traditional mixture. From stiffness point of view, r-material has no negative influence on traditional asphalt mixture.

Comparison of complex modulus, flexural stiffness and modulus of elasticity is illustrated in Fig. 4 and Fig. 5. As could be seen modulus of elasticity is slightly higher than complex modulus but variance is only about 5 % of value. On the other hand flexural stiffness is lower than complex modulus and also with variance about 5 %.

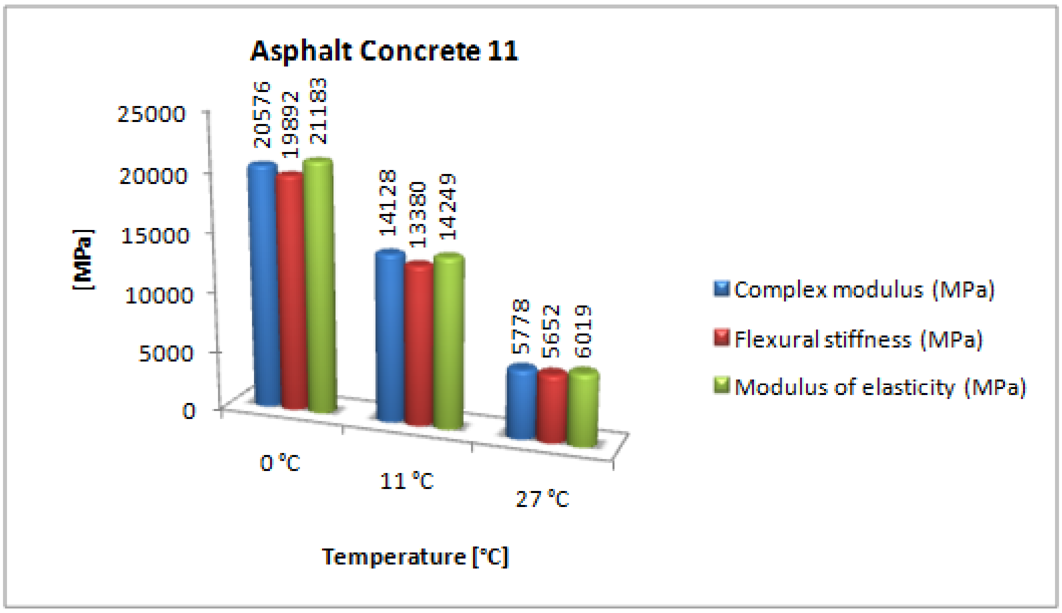

Fig. 4: Comparison of complex modulus, flexural stiffness and modulus of elasticity (AC 11).

Same comparison was made also on mixture with r-material addition and again there are no big differences. Values are little bit lower in comparison with tradition mixture but variance is no more than 10 %. Based on these results it seems that flexural stiffness is similar like real element of complex modulus and complex modulus is modulus of elasticity. Differences between results could be caused by standard measured error or error in calculation because equations are slightly different.

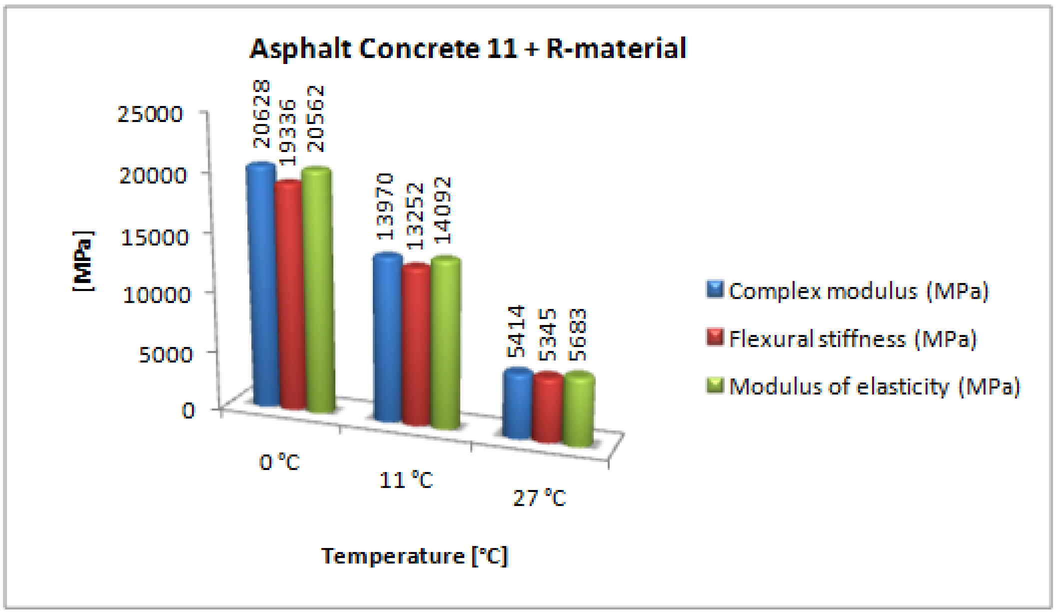

Fig. 5: Comparison of complex modulus, flexural stiffness and modulus of elasticity (AC 11+R_{10}).

4 Conclusion

In this study was observed deformation properties of asphalt mixture with addition of r-material. Amount of r-material was chosen according actual standard as 10 % replacement of aggregate, which is maximal allowed amount. Studied traditional asphalt concrete 11, had quality properties as wheel tracking resistance and also water resistance. By addition of r-material this properties were no negatively affected.

Attention in this paper was mainly focused on stiffness of these mixtures. Stiffness of traditional mixture was much higher than are table values in Slovak pavement design manual [4], but it is consequence of fact that table values are calculated and results in this paper are laboratory measured. Mixture with r-material has still good enough values of stiffness. Stiffness was slightly lower than stiffness of traditional mixture difference was about 3 % in average. The results definitely showed that addition of r-material reached from old pavement structures is appropriate material for new road layers bonded by bitumen. In case of comparison European standard and Australian standard (complex modulus vs. modulus of elasticity) there was no big difference between measured values.

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Impact of Multiple Recycling on Performance Characteristics of Asphalt Mixtures

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Abstract: Recycling of asphalt pavements is widely applied and established standard in road construction practice for many years. However, in this connection stronger attention has so far not been paid to evaluate the possibility re-using again once recycled material in the form of multiple recyclability approach of cold recycled mixes. Within the European project CoRePaSol this process was evaluated in order to determine the multiple-recycling characteristics and limits. At the CTU in Prague, selected types of cold recycled mixtures were designed, which differ in the type of used stabilizing agent/binder. All designed mixtures were observed in terms of the evaluation of the ageing effect and the influence of environment where the specimens were stored. The aged material was quickly frozen and immediately crushed and then used like a multiple recycled material in cold recycled mixes. Differences in the characteristics of mixtures were compared with the evaluation of the ageing impact and increased bitumen content in the final mixture.

Keywords: Recycling; Cold Recycled Mixture; Multiple Recycling; Stabilizing Agent; Ageing.

1 Introduction

The recycling of asphalt pavements in cold recycling mixtures is a widely applied and mostly approved procedure for full-depth rehabilitation measures. Whereas the mix design procedures for optimizing the properties of cold recycled mixtures are well researched, the end-of-life strategies for these types of pavement layers are not known so far. However, the design of modern pavement materials has to consider end-of-life characteristics of the material in order to avoid costly and environmental hazardous disposal of these materials in the future. In order to assess the recyclability of pavement layers composed of cold recycled materials, a laboratory campaign was conducted on newly produced cold recycled mixtures. After the accelerated aging of the materials in laboratory their applicability in new road materials was evaluated.

2 Laboratory-Simulated Reclaimed Cold-Recycled Mixtures

Cold recycling is a term used for re-use of a material from existing pavement without heating the recycled material or any of added binders, as is common for traditional hot asphalt mixes. Group of these technologies became, not only in the Czech Republic but mainly abroad, a trend particularly in case of in-place cold recycling. This group of techniques is well-established standard described e.g. in Czech Technical Specifications of the Ministry of Transportation TP 208. It offers broad possibilities of application; from reconstruction of thin pavement layers to full-depth recycling of a pavement structure. With respect to evaluation of the possibility to re-use again once recycled material 4 different types of cold recycled mixtures were designed. They differ in the type of stabilizing agent/binder and their variable content in the mix as shown in Tab. 1.

For the design and experimental production of cold recycled mixtures reclaimed asphalt material (RAP) 0/22 mm from the Stredokluky asphalt plant was used. Properties and composition of the RAP were analyzed in the laboratory and it included determination of bitumen content as well as of physical properties. Visual evaluation of the material allowed to determine if the RAP is active or inactive. This means assessment whether the RAP has definite own cohesion thanks to the contained binder. Extraction of the bituminous binder was done according to ČSN EN 12697 to reclaim aggregate from the RAP and to determinate the soluble binder

Tab. 1: Design of assessed cold-recycled mixes.

Mix	RAP 0/22 Stredokluky	Stabilizing agent content			Water content
		Cement	Bitumenous emulsion	Foamed bitumen	
A	91.0 %	3.0 %	3.5 %	-	2.5 %
B	90.5 %	3.0 %	-	4.5 %	2.0 %
C	94.0 %	-	3.5 %	-	2.5 %
D	93.5 %	-	-	4.5 %	2.0 %

Tab. 2: Characteristics of the RAP 0/22 mm from the Stredokluky asphalt plant.

Bitumen content (% by mass of RAP) according to the ČSN EN 12697-1	5.57 %
Softening point determined by the ring and ball method according to the ČSN EN 1427	77.4 °C
Penetration determined according to the ČSN EN 1426	14 dmm

content. Determination of the softening point is stated in the ČSN EN 1427 standard. The penetration test is defined in the ČSN EN 1426 standard. It is a very simple, but widely used empirical test which describes basic characteristics of bituminous binders. The needle penetration gives us one of the technical parameters of bitumen classification. Therefore the penetration test was used for the binder extracted from the used RAP. Gained characteristics of the RAP 0/22 mm, which were used for further mix designs are shown in Tab. 2.

Grading of the RAP from the Středokluky coating asphalt plant was determined according to the ČSN EN 933-1. A grading curve was plotted from the test results and the curve is shown in the Fig. 1 together with recommended requirements on grading envelope of cold recycled mixtures as specified in TP 208.

In compliance with the technical specifications TP 208 a cationic bituminous emulsion C60B7 was used for cold recycled mix design and its production, respectively. The straight-run bitumen 70/100 was used for production of foamed bitumen according to the ČSN EN 12591. Further on, Portland cement (specified as CEM II/B-M (V-LL) 32.5 R), manufactured in compliance with the standard ČSN EN 197-1 was used as a hydraulic binder. Portland composite cement containing total amount of silica ash (V) and limestone (LL) between 21 % and 35 % by mass was selected. It belongs in the 32.5 strength class and is characterized by its fast development of initial strengths (R).

2.1 Manufacturing of Test Specimens and Curing Conditions

Requirements set for production of cold recycled mix specimens differ country by country according to existing national technical specifications. Usually, a static pressure or gyratory compactor is used for compaction; Marshall hammer is used rarely in Europe. The differences in manufacturing do not concern just the equipment used, but more likely size of the test apparatus, number of revolutions, number of strokes etc. Results presented further assume that all specimens were manufactured by using a static pressure compaction. In compliance with the Czech specifications TP 208, a mixture is compacted by two pistons moving against each other. The pressure on a specimen is 5.0 MPa. When the load is applied, the vertical force needs to be balanced until it stabilizes at the value of 88.5 $\pm$ 0.5 kN for 30 seconds (for test specimens with 150 $\pm$ 1 mm diameter). Cylindrical test specimens with 150 $\pm$ 1 mm diameter and 60 $\pm$ 5 mm height were manufactured for each mixture using the mentioned compaction method. Bulk density was determined for each of the specimens (calculated from dimensions and weight of the specimen) in order to get the rate of compaction.

All four designed mixtures were observed to evaluate the effect of curing period and influence of environment where the specimens were stored. The curing conditions of cold recycled mix specimens are described in the TP 208 specifications. The document provides temperature, relative humidity and time necessary for storing test specimens. Using mixtures stabilized by bituminous emulsion or foamed bitumen (C and D mixture), test specimens are stored at 90–100 % relative humidity and temperature of (20$\pm$2) °C for 24 hours and then

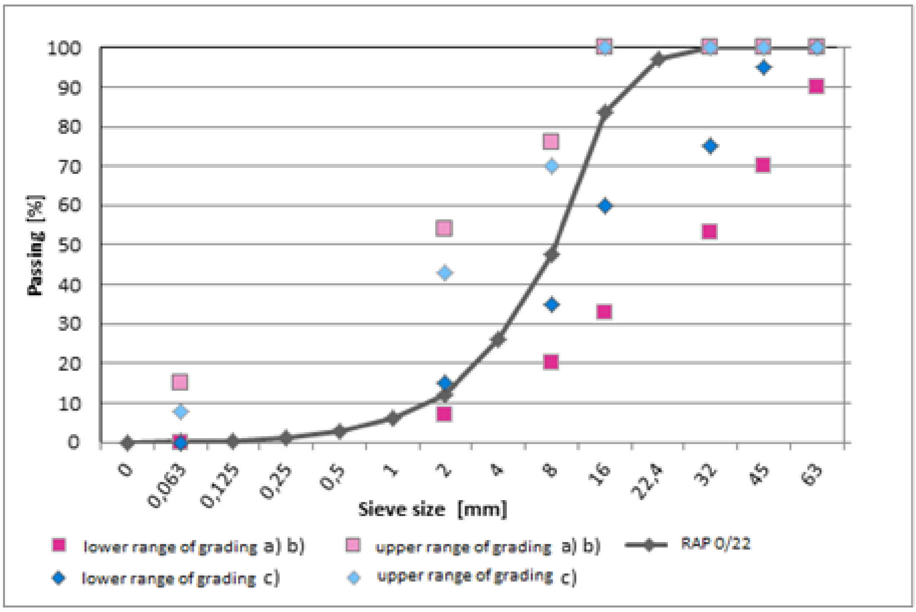

Fig. 1: Grading curve of RAP 0/22. Note: a) cement or other hydraulic binder, b) cement + bituminous emulsion/foamed bitumen, c) bituminous emulsion/foamed bitumen).

Tab. 3: Laboratory curing condition and impact of ageing on test specimens. Note: 90-100 % of relative humidity can be simulated by storing test specimens in sealed plastic bags. Relative humidity 40-70 % represents a humidity of a test specimen at regular atmospheric conditions in the laboratory (unsealed).

Cold-recycled mixture	1st cycle	2nd cycle
	Laboratory curing condition of the test specimens	Used ageing of test specimens
Mix A	24 hours at 90-100 % relative humidity and 20 ± 2 °C, 14 days of curing in dry conditions	9 days at temperature of 85 °C
Mix B		
Mix C	24 hours at 90-100 % relative humidity and 20 ± 2 °C, 72 hours accelerated curing at 50 °C	
Mix D		

exposed to the so called accelerated curing. This means that the specimens are moved to a heating chamber which keeps temperature of 50 °C for 72 hours. After elapsing this period the specimens are stored at 15 °C for at least 4 hours and then immediately tested for stiffness modulus. In the following cycle, the test specimens are exposed to ageing process in a heating chamber which keeps temperature of 85 °C for 9 days and then tested for stiffness modulus including indirect tensile strength at temperature of 15 °C (conditioning again for at least 4 hours). The ageing protocol was selected unanimously for the whole CoRePaSol project. In the case of cold recycled mixtures bound with cement and bituminous emulsion (A mixture), the test specimens were stored at 90-100 % relative humidity and temperature of (20±2) °C for 24 hours. Then the specimens cured at dry conditions with relative humidity of 40-70 % and laboratory temperature for additional 14 days. After this period the specimens are conditioned at 15 °C for at least 4 hours and tested for stiffness modulus. Another ageing cycle follows like for C and D mixtures; the specimens are heated at temperature of 85 °C for 9 days, then the stored for 4 hours at 15 °C and immediately tested for stiffness modulus and indirect tensile strength.

Testing procedures including the ageing process in case of all prepared cold recycled mixtures enable simulating conditions of recycled pavement material at the end of its life-time. The test specimens, exposed to the curing process, were further quickly frozen and immediately crushed using a laboratory jaw crusher. This laboratory aged RAP was then used like a multiple recycled material in cold recycled mixes with reduced content of newly added bituminous binder as well. An illustration of multiple aged and crushed RAP is presented in the Fig. 2.

Fig. 2: Multiple aged and crushed RAP.

Tab. 4: Test results for initial cold recycled asphalt mixes.

Cold-recycled mixture	Maximum density [g/cm^3]	Bulk density [g/cm^3]	Voids content [%]	ITS [MPa]		Stiffness modulus [MPa]	
				after 1st cycle	after 2nd cycle	after 1st cycle	after 2nd cycle
Mix A	2.403	2.162	10.0	0.76	1.12	3736	6539
Mix B	2.345	2.130	9.2	0.76	1.29	3406	7086
Mix C	2.416	2.140	11.4	0.67	0.99	2105	4775
Mix D	2.358	2.116	10.3	0.60	1.09	2487	5160

2.2 Results for Design Cold-Recycled Mixtures

Results gained for assessed cold recycled mixes containing bituminous emulsion/foamed bitumen or variations where also hydraulic binder is used, are described in following sub-sections. Tab. 4 summarizes basic physical properties of cold recycled mix with the comparison of three assessed variations of aged cold recycled mix. Bulk density and maximum density are important parameters, whereas the bulk density was later selected as a characteristic to which the most of the other properties are related. As is obvious, maximum densities for cold recycled mixtures containing bituminous emulsion and cement, as well as mixes containing only emulsion have similar values. Mix A and C shows slightly increased value. On the other hand maximum density for mix B and D where foamed bitumen and combination with cement were used, reaches lower values.

With respect to the voids content it is not possible to see any logical trend within the tested mixes. Highest voids content is reached for mix C where bituminous emulsion without hydraulic binder was used with an average value of 11.4 %-vol. Mixes A and D reach nearly same results. In case of mix A the voids content is 10.0 %-vol. and in case of mix D 10.3 %-vol. Mix B reaches lower values, 9.2 %-vol.

Fig. 3 illustrates results of indirect tensile strength test. If it would be assumed to compare ITS results with the requirements given for cold recycled mixes in Czech technical specifications following conclusions can be made. Technical specifications TP 208 prescribe requirements according to used stabilizing agent. For cold recycled mixes where bituminous emulsion or foamed bitumen is used the minimum indirect tensile strength after 7 days is set as R_{it} = 0.3 MPa. In case of mixes where bituminous binder is combined with cement, this required value has to be within the range of 0.3 MPa and 0.7 MPa, whereas the upper limit is selected with respect to avoid the risk on micro-cracking. In both cases the ITS is determined at 15 °C. Results of gained ITS values, were assessed also on aged test specimens, and compare with results on specimens after first cycle of laboratory curing. The quality of the asphalt cold recycled mix is therefore proven by reached required value of particular indirect tensile strength, eventually stiffness modulus. From the results it is obvious, that the highest values of gained ITS after cold recycled mix ageing are reached for mix B - value of 1.29 MPa, mix A and mix D – in average 1.10 MPa. In comparison to that mix C shows only strength values around 0.99 MPa.

For all four designed mix types stiffness modulus after first ageing cycle of test specimens was determined as well. The ageing effect depends on the assessed cold recycled mix type. For mixes with use of hydraulic

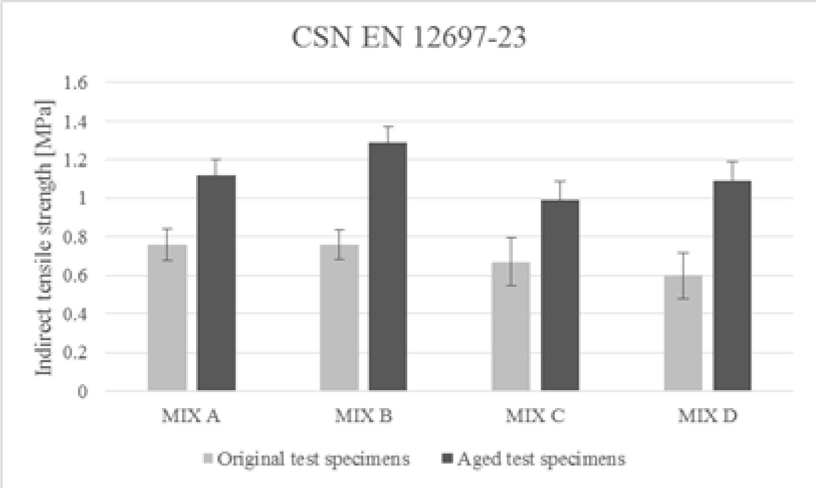

Fig. 3: Indirect tensile strength results of experimentally designed cold recycled mixes.

binders the stiffness was determined after 15 days curing, in case of mixes without cement this was determined already after 5 days (accelerated curing). Further the effect of test specimen ageing was reflected simulating material behaviour in the pavement structure which is for a certain time in operation. At the end of ageing the stiffness modulus was determined again and finally the indirect tensile strength was tested. Despite of the fact that Czech technical standards do not require the check on stiffness for this type of material and there is no minimum required threshold value, it is presented as a very important characteristic, which has a good predictability for describing cold recycled mix behaviour in a pavement similarly to the destructive testing of indirect tensile strength. At the same time stiffness represents an important parameter from the viewpoint of pavement structural design. Therefore for all mixes with unaged and aged material stiffness was always determined at the temperature of 15 °C by applying the IT-CY test method.

Comparison of stiffness modulus values for cold recycled mixes before and after ageing is illustrated in Fig. 4. From the gained results following findings and conclusions can be made. Stiffness modulus of mix A and mix B where bituminous emulsion (or foam bitumen) is applied together with cement shows highest average values before ageing (3.736 MPa and 3.406 MPa). On the other hand mixes C and D reach approximately values of 2.250 MPa. It is therefore possible to demonstrate again the influence of hydraulic binder content, which improves stiffness and ITS values. Ageing leads to increase in stiffness of tested mixes and the values correspond again well with results of indirect tensile strengths which were determined after ageing as well. Mix A reaches in average stiffness values of 6.539 MPa, for mix B a value of stiffness 7.086 MPa was determined. This means an increase of about 75 % of original value before ageing procedure. In case of mix C it can be stated, that stiffness modulus results are in average 4.775 MPa what is an increase by more than 125 %. Highest relative increase after ageing is nevertheless reached for cold recycled mixes with foamed bitumen (mix D). In this case stiffness was raised by more than 140 %. In general this corresponds well with results gained for indirect tensile strength. Only in case of mix D the ITS reaches in average higher increase of measured values.

3 Multiple Recycled Cold Mixes

Aged material from cold recycled mixes described in chapter 2 was re-crushed by a laboratory jaw crusher and used for new design of cold recycled mixes. The selected mix designs are summarized in following Tab. 5. For each proposed experimental mix two sets of test specimens were produced and compacted. Test specimens were left for 24 hours in sealed conditions at laboratory temperature and then moved to a climatic chamber with unsealed conditioned at 50 °C for three days. After this time period the test specimens were measured on their dimensions, weighed and divided in two groups. First group of test specimens was left at standard laboratory conditions for three days, second group was water saturated and conditioned for 3 days in a water bath at 40 °C. After 7 days curing stiffness modulus was determined on all test specimens at 15 °C. Lastly indirect tensile strength was set at the same temperature.

For each reclaimed asphalt material its grading has to be declared, i.e. relative mix composition showing the total mass of particular particle sizes including its expression in form of a grading curve. For the later

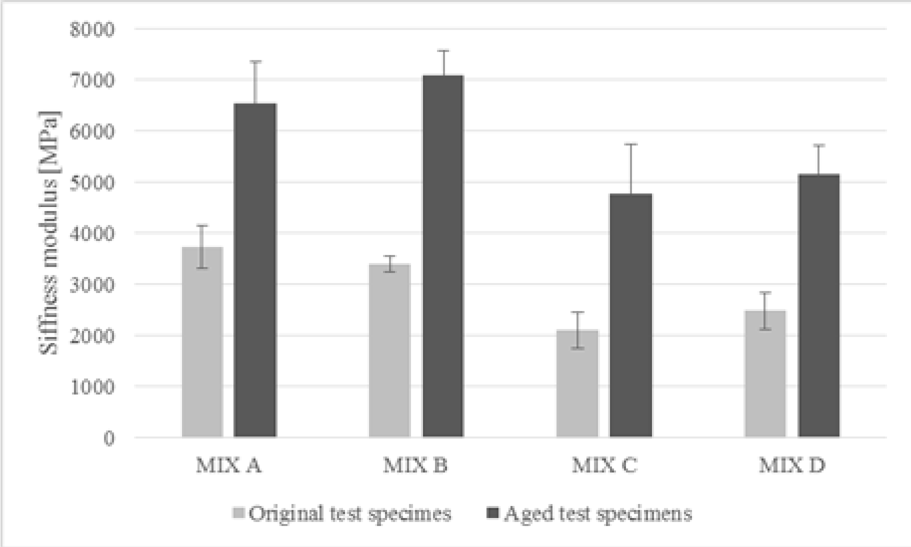

Fig. 4: Impact of ageing shown on stiffness modulus values.

Tab. 5: Design of multiple cold-recycled mixes.

Mix	RAP	Stabilizing agent content			Water content
		Cement	Bitumenous emulsion	Foamed bitumen	
SA	95.5 % – RAP A	-	2.0 %	-	2.5 %
SB	96.0 % – RAP B	-	-	2.0 %	2.0 %
SC	95.5 % – RAP C	-	2.0	-	2.5 %
SD	96.0 % – RAP D	-	-	2.0 %	2.0 %

mix designs and use of aged recycled materials in cold and hot asphalt mixes grading curves of all applied re-crushed and aged options of reclaimed asphalt material were calculated and plotted.

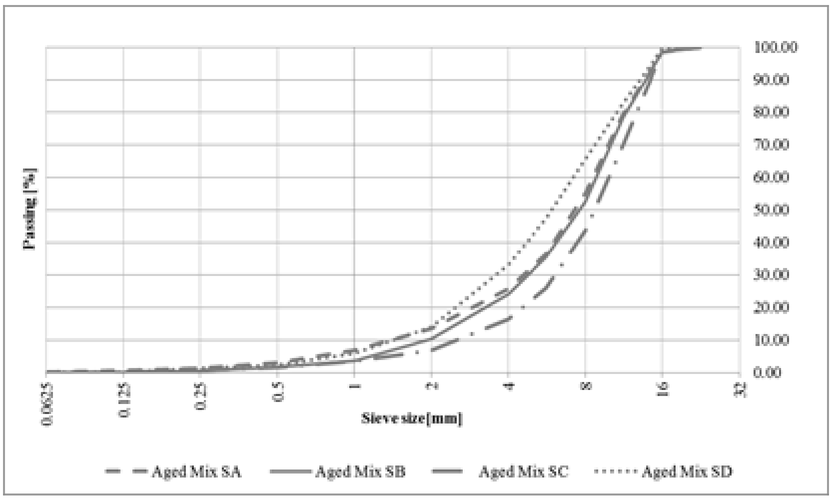

Fig. 5: Grading curves of aged and re-crushed material from cold recycled mixes.

Basic characteristics determined on test specimens of designed mixes are summarized in Tab. 6. From the results it is evident that voids content of the assessed mixes have similar trend like for cold recycled mixes produced and compacted before the artificial laboratory ageing. For mixes SA and SC resulting voids content is 13.4 % and 14.5 %, experimental mix with foamed bitumen as a binder shows lower value of 8.9 % and

Tab. 6: The basic empirical and mechanical characteristics.

Cold recycled mixture	Maximum density [g/cm^3]	Bulk density [g/cm^3]	Voids content [%]	ITS$_{dry}$ [MPa]	ITS$_{wet}$ [MPa]	ITSR [%]
Mix SA	2.328	2.017	13.4	0.796	0.666	83.8
Mix SB	2.290	2.088	8.9	0.745	0.755	101.4
Mix SC	2.407	2.059	14.5	0.798	0.546	68.8
Mix SD	2.300	2.078	9.6	0.834	0.526	63.9

9.6 %.

If we focus on comparing results of gained ITS values and display them in Fig. 6, then it can be concluded, that slightly higher values of 0.83 MPa were reached for the mix SD containing foamed bitumen. Sets of test specimens of mixes SA and SC reach in average similar results around 0.80 MPa. Indirect tensile strength for mix SB is 0.75 MPa.

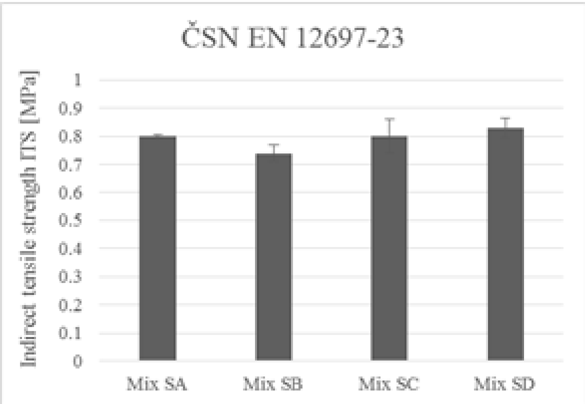

Fig. 6: ITS values of cold recycled mixes made with re-crushed and aged RAP.

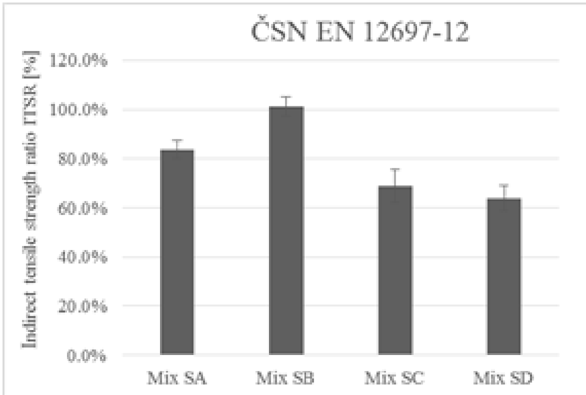

Fig. 7: ITSR values of cold recycled mixes made with re-crushed and aged RAP.

From the Fig. 7 where ratios of indirect tensile strength values are determined for dry specimen and water saturated specimen subjected to adverse impact of higher temperature of 40 °C it is obvious that there is a negative impact of water on the test specimens. From the results it is apparent that mix SA and SB containing combination with cement resist better the adverse effects of water if compared with the other two mixes. The mix is less water susceptible. Mixes SC and SD are reaching ITSR value lower than 70 %. Last but not least of the attention is paid to comparison of stiffness modules, the results confirm findings made for indirect tensile strength. Cold recycled mix SB resist again better negative impacts of water as against remaining three experimental mixes. Stiffness modulus of used dry test specimens for mix SA (3,161 MPa) and mix SB (2,991 MPa) and mix SD (3,185 MPa) –containing bituminous emulsion or foamed bitumen – show in average similar result, whereas mix SC (2,629 MPa) reached a lower value. In this case surprisingly the hydrated cement in the aged and re-crushed cold recycled material A and material B does not have any effect on the properties of newly produced cold recycled mix SA, the binder acts as a regular mineral part of the aggregates.

4 Conclusion

Multiple recycling of asphalt pavement layers is dependent on the ageing process during the life.time of this composite material which is used in the pavement structure and at the same time exposed to climatic conditions and effects of road traffic. Repeatedly use of reclaimed asphalt material is according to so far gained experience a possible and at the same time suitable approach for reduction of non-renewable sources exploitation (aggregates, bituminous binders). At the same time it is a possibility how to increase the environmental protection and securing savings of public budgets. This paper provided an assessment of impact of ageing if

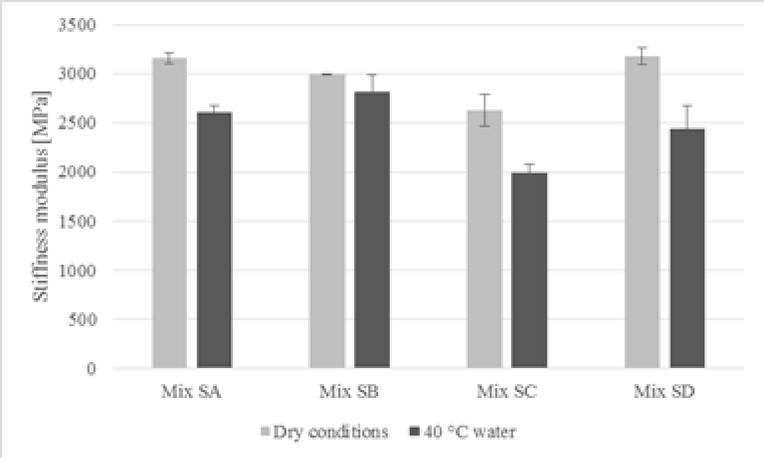

Fig. 8: Stiffness of cold recycled mixes with re-crushed and aged RAP, including water susceptibility.

simulated for cold recycled asphalt mixtures which then where repeatedly recycled in the same type of mixture just with a reduced content of used binder. From the gained results it is visible, that the use of twice recycled asphalt material does not show worsening of determined characteristics even with respect to slightly increased content of bituminous binder in the mix. Multiple recycling therefore lead to satisfactory results, nevertheless it is important to consider certain limit for the bitumen content. This is mainly if the repeatedly recycled RAP will reach too high level of residual active binder and such material would be then unsuitable for an application in a new asphalt mixture.

Acknowledgement

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Environmental Pavements

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Abstract: Recently, the interest in the preservation of the environment for future generations has increased. Particular environmental issues have become a serious problem of our civilization. We know that road transport has multiple effects on the environment. It should be realized that transport affects the environment in two ways, both positive and negative. The positive effect is that it ensures the movement of persons, goods and materials, thus ensuring the needs of society as well as the performance of certain services and contributes significantly to the growth of tourism. Negative effects is caused mainly by excessive load of road transport that damage and devalues the environment. The article is focused on road surfaces that reduce environmental impacts. It contains the results of the latest research of pavement surface properties, which contribute to improving the environment by reducing noise. The results will be presented graphically and in tables with a recommendation for their best use.

Keywords: Environmental Mixtures; Environmental Technologies; Noise; Re-Use of Asphalt Mixtures.

1 Introduction

The environment and its protection are highly discussed topics. Transportation engineering seeks new solutions for reduction of negative impact on environment. Major problems are caused by emission and noise pollution. Characteristics of pavement surfaces are either directly or indirectly influencing the environment. One of the most important criteria for road network design in urban as well as open areas is environment quality. This Paper is focused on pavement surfaces, which are able to reduce negative impacts on environment.

Slovakia has road network of approximately 17 970 km. Most of the roads use pavements with asphalt wearing course. Concrete pavements constitute only 1% of road pavements.

2 Environmental Technologies

In road engineering environmental technologies mean technologies of production and laying of cold asphalt mixtures and mixtures at lower temperatures (hot and semi-hot mixtures), cold material recycle and reuse technologies and technologies at lower temperatures. These technologies were invented as environmentally more acceptable alternative to conventional hot asphalt mixtures. Moreover, they are using various types of waste materials, energetic waste, new materials based on agricultural crops, etc.

Environmental technologies have considerable effects in several ways and their implementation and use has mainly positive influence on environment, in reduction of pollution and waste.

Environmental technologies include:

- end technologies for reduction of pollution (i.e. air pollution reduction, more efficient waste management),
- products and services which cause less damage to the environment and need less intensive use of natural resources,
- methods for more efficient use of resources (e.g. water supply, energy saving technologies).

As described above these technologies, pervade all economic activities and sectors, where they often reduce costs and improve competitiveness by reducing energy and materials consumption, thereby causing less emissions and waste. Environmental technologies present solution for sustainable growth of public and private markets [2].

2.1 Technology Végécol®

As the world's petroleum resources as the source for asphalt production are not unlimited, solution is sought in alternative binders. In this matter are active many research and development centres throughout all Europe.

Végécol is not classical asphalt wearing course but organic mixture based on plant base. It was developed and initially applied in France, where it is successfully used. Producer guarantees the mixture is an excellent alternative to conventional asphalt layers. It is characterized by improved physical and mechanical properties and ensures greater resistance against rut and fatigue cracks of pavement surface. It even has aesthetic advantages while the mixture can be coloured to the client's requirements. Colour scale does not yet contain full range but certainly enough for the client to choose from. However the white shade is more of colouring possibility demonstration than usable alternative. In Slovakia this technology was used in Myjava on town's roundabouts in the city centre (Fig. 1).

Végécol is really ecological product. Its precise composition is producer's secret, but it is known that the mixture is produced from renewable sources - plants commonly grown in European conditions. It does not contain any noxious agents that would negatively influence the environment and it is fire resistant [3].

Fig. 1: Laying of Végécol technology in Myjava [3].

2.2 Technology Valorcol®

Valorcol is cold mixture consisting primarily of asphalt aggregates mixed with, asphalt emulsion in the mobile mixing center. It is produced by Colas company.

In some cases it is produced as a 100 % asphalt binder recyclate. It can be used on pavements with moderate traffic and a high level of deformation.

In the field of environment protection Valorcol contributes by significant energy savings, binder and transport costs savings; emissions absence during production. Aggregate is made of natural stone so it is of good quality and economical at the same time [4].

Fig. 2: Production and laving of Valorcol technology [4].

2.3 ECO-MIX™

ECO-MIX uses a simple process, where the water is mixed with asphalt binder to create of microscopic steam bubbles 'foam'. Foamed binder has the same mechanical characteristics at a lower temperature than classical asphalt mixtures produces by hot technologies. This means that it may be laid at the same speed and with the same efficiency and quality as classical asphalt mixture.

ECO-MIX is transported and compacted by using the same conventional methods that are used on hot mixtures. It is 100 % recyclable and recognizably more environment friendly. It is a "hot asphalt" (WMA), which requires less energy for production and uses more recycled materials in the mixture. Reduces emissions, by which it is environment friendly and does not smell which is better for employees and clients [5].

Fig. 3: Laying of EKO-MIX mixture [5].

2.4 ECO-PAVE ®

This product is environmentally acceptable water-based binder. It is milklike white liquid which dries much faster than concrete. This product was tested and the results showed that it is ecological and environment friendly. It is quick and easy technology that can be laid in the range of 4-8 km per day. Eco-Pave is designed to achieve in combination with land or aggregate, including clay, a perfect combination and results. It is an ideal alternative to asphalt and other construction materials [6].

Fig. 4: Design EKO-PAVE in-situ [6].

3 Re-Use of Asphalt Mixtures

New mixture, where the old pavement materials were used, must have properties complying with the valid technical regulations. The possibility of using these materials and technologies creates a relatively large number of variations and combinations of pavement layers design [1].

This technology reduces energy requirements, economic demands related to the input materials processing and asphalt mixture itself, preserves natural non-renewable resources, fuel, reduces the volume of production waste materials. This technology can use up to 100 % of material obtained from the original pavement layer for new layer [7].

Fig. 5: Construction of new pavement with recycling machine by cold technology [14].

4 Noise

One of the negative environmental effects of traffic is noise. Noise exposure risk assessment is extremely urgent problem due to the enormous increase of acoustic energy in the environment at the present time. Traffic is the largest originator of noise in the world.

Solutions are possible in several fields:

- Modification of transport area surroundings (vegetation cover);
- traffic flow modification in favour of the noise reduction
- pavement structure designs reducing the origin of noise on tire-pavement contact
- re-use of old road construction materials as saving (economic, energetic, environmental etc.)
- parametric calculations of pavement structure with reduced noise design
- Measurement and evaluation of existing pavements in urban areas from the noise view.

This article is focused on noise reduction in tire-pavement contact. Increased traffic intensity leads to increased traffic noise. Road surfaces that can significantly reduce tire-pavement contact noise re currently development.

4.1 Surface of Noise Reducing Pavements

Asphalt pavements are the most common pavement types in urban areas and asphalt wearing course has many uses for traffic areas. As the asphalt wearing course are used asphalt concrete, mastic asphalt mixture, asphalt carpets (mastic, drainage, thin wearing course). Compared with conventional asphalt mixtures, the so called acoustic surface mixture can reduce noise by 12 dB.

Concrete pavements represent closed wearing course without significant macrotexture. [10] The noise can be easily reduced by adjusting the wear course. For macrotexture improving are used drawn ”brushes”, which show considerable roughness and noise. As a next option towable wetted burlap is used. This procedure is in decline because of their limitation against smoothing. Aggregate concrete is used to improve the texture too. The advantage of this surface adjustment is the durability of skid resistance based on the properties of aggregates, not on the properties of the cement mortar [11].

4.1.1 Porous Asphalt

The most often used noise reduction asphalt wearing course is drainage porous asphalt (PA). Thanks to drains the traffic noise is significantly reduced on pavement-tire contact. Compared to the asphalt concrete carpet wearing course the noise can be reduced by use drainage asphalt wearing course by 3-5 dB.

4.1.2 Stone Mastic Asphalt

Stone mastic asphalt (SMA) is mixture with discontinuos grading. The skeleton of crushed stone is bind with mastic asphalt mortar. SMA can reduce noise by 2 to 2.5 dB (A). The composition of the SMA is 70 to 80 % coarse aggregate, 8 to 12 % filler, 6.0 to 7.0 % binder, and 0.3 % fibre. SMA with its composition reduces the noise of traffic and it is sturdy and durable.

Fig. 6: Porous asphalt [15].

Fig. 7: SMA Stone mastic asphalt [16].

4.1.3 Very Thin Layer Asphalt Concrete

Very Thin Layer Asphalt Concrete (BBTM) is a mixture for wearing course with thickness of 20- 30 mm. Like SMA also BBTM has discontinuos grading in order to provide open texture surface between aggregate. To the group of very thin asphalt mixtures with noise reduction characteristics belong also BBTM A 5 and BBTM B 5.

The asphalt mixture BBTM A 5 reduces noise by 2 to 3 dB in comparison to the SMA 11. BBTM A 5 has better resistance to deformation and rut performance and excellent anti-skidding characteristics. It consists of a grain with maximum size 0/4 mm or 2/4 mm. The voids in the asphalt are 7 % to 10 %. BBTM B 5 can reduce noise by 3 to 4 dB in comparison to SMA 11 [11].

Fig. 8: Very Thin Layer Asphalt Concrete [17].

4.1.4 Viaphone ®

The company Eurovia CS developed in Czech Republic reduced asphalt mixture Viaphone®. Viaphone® is less prone to clogging and has good anti-skidding characteristics. The asphalt mixture Viaphone® can reduce noise by 6,5 dB as was proved by measurements in Prague.

Viaphone® has a grain size of 0/6 or 0/8 and the mixture has a high content of coarse aggregate of fraction 4/6 or 4/8 (5/8) [12].

Viaphone® effectively reduces rolling noise. Its characteristics are suitable for urban roads, maintenance of city streets, by-passes, circular intersections, access roads [13].

4.1.5 Open Graded Friction Courses

Open Graded Friction Courses (OGFC) is noise reduction asphalt mixture used in America. OGFC is composed from a mixture of fine aggregate (forms the pavement surface) and from a large percentage of air

Fig. 9: Viaphone ® [18].

voids. The advantage of these air voids is to improve the friction in wet weather and prevent aquaplaning, because of better drainage of the pavement.

Fig. 10: OGFC [19].

5 Conclusion

The aim of this paper is not to start the discussion about less or more preferred technologies, but to confirm the actual state in used environmental technologies in road building industry in means of noise reduction possibilities. Sustainable development and environmental protection are still relevant issues related to the use of modern environmental technologies in road construction and design. [8]

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Experimental Testing of Fly-Ash Stabilized Mixes

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Abstract: The application of fly-ash stabilizers, materials from fluidized combustion fly-ash and other solid coal-burning residues which are called coal combustion by-products (CCB) that have good potential for application in subgrade structures and roadbed materials of roads as well as in the structural pavement layers. One of the many factors limiting the application of some CCB sorts is the relatively low resistance in repetitive contact with water, volumetric changes and the risk of partly unsatisfactory hygienic and environmental parameters.In regard to the aforementioned negative characteristics of CCB which occurred primarily under the repetitive impact of water and freezing, the experimental examination focused on improving CCB resistance to frost and water, verification of volumetric changes and improvement of pozzolana characteristics of CCB by increasing the percentage of fine particles in the original material.Currently road construction industry strives to find a suitable substitute of the traditionally applied hydraulic binders as well as expand the existing base of the binders applied. The experience with using CCB as a binder or binder component has not been as extensive so far as to allow any generalisation of conclusions. Therefore, the possibilities of applying alternative additives as a replacement of the binders traditionally applies have been researched.

Keywords: Fly-Ash; Fluidized-Bed Fly-Ash; Mechanical and Chemical Activation; High Energy Milling; Roadbed Structures; Fly-Ash Stabilized Material; Chemical Analysis; Compressive Strength.

1 Introduction

All technically advanced countries have intensified their research and development of utilisation of various types of waste as secondary material sources in recent years. This trend also applies to the brown coal combustion residuals, great quantities of which are a waste in the power industry since they occur as solid residues from coal combustion and residue purification in power plants, heating plants or heat stations. The materials are called coal combustion by-products (CCB) and include e.g. fly-ash, slag, cinder, bottom ash, flue gas desulfurization (FGD) gypsum.

Those countries that support their power policy primarily by generating power in thermal power stations research the options of processing and subsequently effectively utilising CCB. Practical applications have focused primarily on the construction industry so far. One of the paths seems to be applying CCB in roadbed structures of roads, railroads or airfield structures as there is an assumption of processing larger quantities of CCB.

Another possible application of CCB gained after coal combustion is according to [1,2] use of such material for soil modification or improvements in the roadbed structure. Such modifications of soil usually lead to higher strength properties, improved workability and increased resistance to climatic effects. Other mechanical or physical characteristics of the original soil can be improved as well.

A limiting factor for the use of some CCB types is their relatively low resistance in repetitive contact with water and freezing [3, 4], volumetric changes and in some cases the risk of partly unsatisfactory health and environmental parameters [5, 6]. Particularly for fly-ashes from fluidized combustion, ettringite (high-calcium sulfo-aluminate mineral) might be formed in case of long-term contact with water [7, 8]. Extensive analyses of CCB chemical characterization have been collected and done, e.g. during the planning and preparation of embankment structure for a motorway project in the UK. CCB samples were taken from three different power plants. Leaching analyses have shown increased contents of arsenic, cadmium, chromium, mercury, selenium and sulphates. At the same time the pH value was increased as well as the concentration of polycyclic aromatic hydrocarbons [9].

With respect to the aforementioned negative CCB characteristics which occurred primarily under repetitive influence of water and freezing, the experimental research focused on improvement of CCB resistance to frost and water, verification of volumetric changes and improvement of pozzolana properties of CCB by increasing the percentage of fine particles in the original material (e.g. by means of mechanically and chemically activated fly-ash).

At the same time, the road construction industry strives to find a suitable replacement of the hydraulic binders traditionally used as well as extend the existing base of the binders applied. The experience with application of CCB as a binder or binder component is not as extensive so far as to allow generalisation of its conclusions. Therefore, the possibilities of alternative additive application as a substitute of the binders traditionally used were examined.

2 Materials

2.1 Coal Combustion By-Products

The samples chosen for the purposes of experimental research of stabilised fly-ash mixes were the so-called fly-ash from electrostatic separators (filters) of the Melnik power plant (hereinafter “EME”), often called high-temperature fly-ash, and bed ash from fluidized combustion from the Ledvice power plant (hereinafter “ELE”) (see Fig. 1a, 1b). The aforementioned power plants represent the two basic types of desulphurisation; each of them produces CCB of differing technical parameters. EME desulphurisation follows the route of the wet scrubbers. In ELE, CCB from furnace FK4 were used; this is desulphurised by the fluidized combustion method.

With respect to the combustion technology applied, the chemical and mineralogical composition of fly-ashes from fluidized combustion fundamentally differs from the composition of classic high-temperature fly-ashes. While the main phases of high-temperature fly-ashes consist of amorphous silicon dioxide, silica, both high-temperature modifications – cristobalite as well as tridymite and mullite – fly-ashes contain aluminosilicate phase, silica, insoluble anhydride II, free calcium oxide, and possibly calcium hydrate and calcium carbonate. As ensues from the above, high-temperature fly-ashes demonstrate the pozzolana properties exclusively while fly-ashes from fluidized combustion, thanks to the presence of calcium ions, have hydraulic properties even on their own [10].

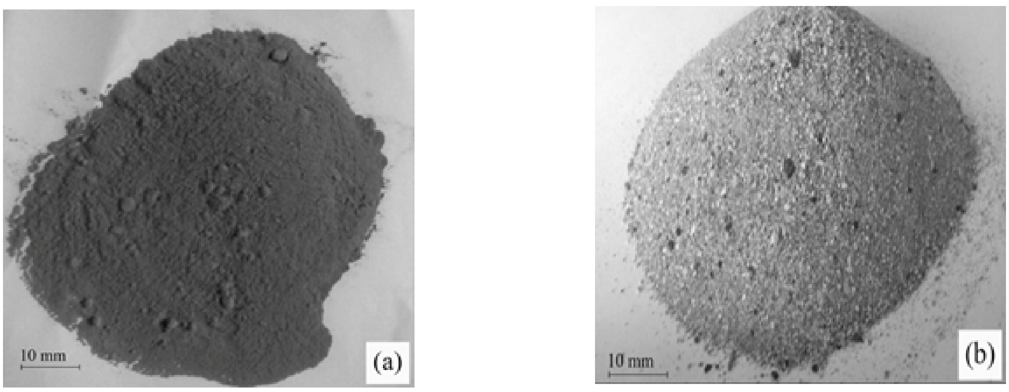

Fig. 1: Loose material; a) filter fly-ash EME, b) bed ash from fluidized combustion ELE.

Within the structural analysis of two selected types of energetic by-products assessment of internal material structure has been carried out by application of electron microscopy. Microscopy and microanalyses have been processed by the environmental scanning electron microscope (ESEM FEI PHILIPS), equipped by the set of electron detectors – scattered electron diffraction (SED) for morphology on micro level and BSED for phase contrast, both in high vacuum mode and environmental mode. The electron diffractions (ED) analysis of secondary X-ray spectra provides the quantitative chemical composition of the selected objects. The quantitative phase mineral composition both on micro and nano level is facilitated by OIM-BSED (Orientational Imaging Microscopy based on Back Scattered Electron Diffraction). This equipment gives the information about the quantitative phase mineral composition and structural orientation maps.

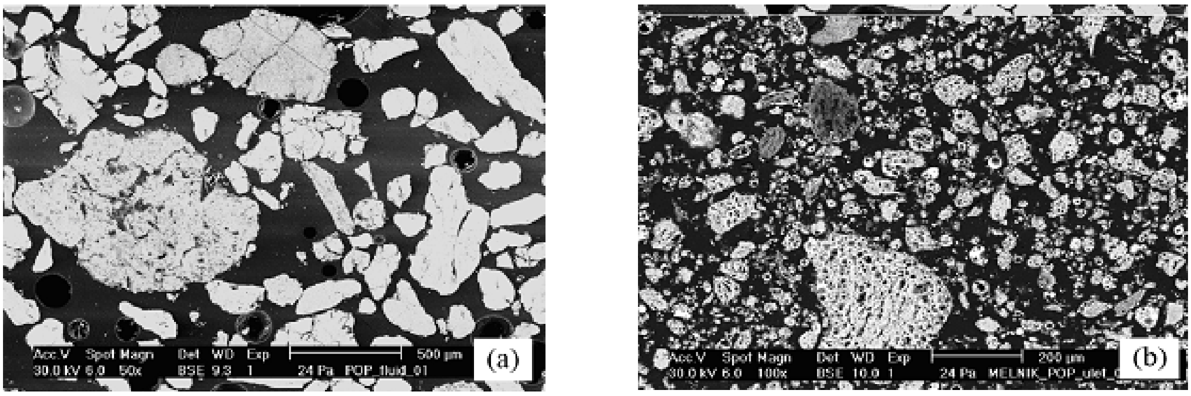

Fig. 2: Electron microscopy; a) bed ash from fluidized combustion ELE, b) filter fly-ash EME.

2.1.1 Sample Preparation Used for Electron Microscopy Analysis.

Before the electron microscopy analysis representative quantity of fly-ash sample (10 ccm) was mixed with low viscose epoxy resin. After hardening (at 25 °C), the sample was brushed (emery papers) and polished (diamond paste) in dry mode. Obtained surface was cleaned by pure methanol. This method of sample preparation prevents eventual reaction of water-sensitive fly-ash particles.

Methods of electron microscope assessment were based on phase contrast taken by back-scattered electrons detection (BSE) and energy dispersed X-ray analysis (EDS).

2.1.2 Analyses of Fly-Ash from Melnik Coal Power Plant

Fly-ash particles (FAP) are represented by the very porous slaggy debris some of larger particles are almost foamy. The character of FAP is very monotonous, nearly all particles have the same character: porous and foamy. The size of FAP varies from first microns up to several tenths of mm. The compact like glassy balls of FAP or iron oxides particles do not occur in this type of fly-ash from EME power station.

2.1.3 Analyses of Fly-Ash from Ledvice Coal Power Station

Fly-ash particles (FAP) are mostly represented by calcium sulphates (gypsum, hemihydrate, anhydrite etc.). Some grains are alumosilicates (mullite, quartz). Only a small portion of FAP is porous (in comparison with FAP from Melnik).

2.1.4 Analyses of Fly-Ash from Plzen Generation Plant

Fly-ash from fluidized combustion originated from Plzen generation (heating) plant which is equipped with the fluidized bed furnace technology that is installed in a number of power-generating operations in the Czech Republic. The types of fly-ash obtained are formed primarily during fluidized combustion of soft coal and limestone powder which, from the perspective of further application in the construction industry, are rather significant. The ash was mechanically activated concretely the material was driven between the rotors of a twin-rotor contra-rotating high speed mill – disintegrator – under mutual peripheral speed of the rotors of approx. 204 mm.s-1 and power consumption at the level of approx. 20 W per kg of pulverized ash.

The fly-ash from the Plzen heating plant contain unusually small quantities of free lime; such ash loses its self-binding ability and an addition of a certain quantity of lime or cement would be recommended in a certain stage of compacted mix production. The contents of the amorphous phase are rather high in such type of fly-ash. The results of XRD analysis for mechanically activated fly-ash is indicated in the following Tab. 1 and Fig. 3.

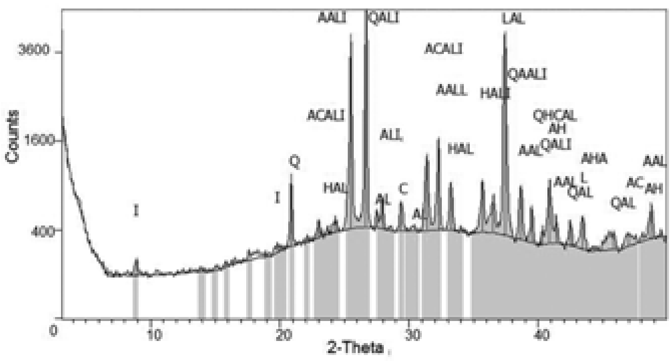

Fig. 3: XRD data record of mechanically activated fly-ash from fluidized combustion Plzen.

Tab. 1: Evaluation of phase composition of mechanical chemically activated fly-ash from fluidized combustion based on XRD data record.

Ref. Code	Compound Name	Score	Total Lines	Scale Factor	SemiQuant [%]
Fly-ash from fluidized combustion – generation plant Plzen					
01-085-0794	Quartz	69	7	0.994	24
01-074-2421	Anhydrite	63	16	0.722	31
01-079-0007	Hematite	46	7	0.136	3
01-078-0649	Lime	49	2	0.800	13
01-083-0578	Calcite	43	9	0.071	2
01-089-6423	Albite	26	83	0.076	9
00-026-0911	Illite-2\ITM\RG#1 [NR]	36	17	0.427	18

2.2 Mechanical Activation

The mechanical process or interference with the structure of a substance that increases its chemical reactivity can be called mechanical activation. Such an intervention in the substance structure can consist of grinding/pulverization. According to the classic interpretation, grinding is defined as mechanical dispersion of solid substances which results in reduced particle size and a simultaneous increase of specific surface and surface energy within the system; nevertheless, mechanical effects occurring in the course of dry grinding of solid particles might cause significant structural changes and chemical reactions in the material ground. The character of the surface, or morphology thereof, distribution of charges, chemical nature of the thin grain surface film have a very distinctive impact on reactivity as well [11–14].

Grinding might be a possible solution for rough fluid combustion separation process residuals. The grinding process causes large plerospheres (porous particles) to disintegrate and reduces particle roughness. Such reduction, together with the increased reactivity of the fly-ash, improves strength. The grinding of cenospheres increases density and fineness which results in higher pozzolana reactivity of the fly-ash. The grinding time affects the particle size, shape and, consequently, also the need for water [15].

2.3 Stabilized Fly-Ash Mix

The so-called fly-ash stabilizate was chosen as the first type of compacted mix for experimental verification. Fly-ash stabilizate is a solid mass which usually arises by wetting a mix of fly-ashes or ashes with a binder (e.g. lime or cement) or by wetting a mix of fly-ashes from fluidized combustion that demonstrate self-setting properties. Ettringite might form during this process which causes volumetric changes. Ettringite forms in fly-ash stabilizate from soluble compounds of calcium, aluminium and sulphur in wet, alkali environments. In some cases, its expansion ability might damage the solidified stabilizate. According to the composition of the mix and mutual component proportions, quantity of added water and the processing method, a mass whose strength and other physical properties (permeability, weight, and thermal conductivity) are similar to those of lightweight concrete.

A deponate is understood as non-solidifying mass (with no additional additives) which is only strengthened by dehydration, drying or thixotropy.

The following additives were applied to prepare the fly-ash stabilizates:

- cement CEM II/B-M 32,5R,
- lime CL90S,
- mechanically activated or combined mechanically and chemically activated fluid ashes (from the Plzen heating plant),
- mechanically activated dolomitic limestone (from the Krty u Strakonic quarry),
- mechanically activated or combined mechanically and chemically activated recycled mix from concrete (recycled aggregate generated by crushing and sorting of reclaimed concrete from the airport Ruzyne),
- chemical additives Iterstab, Zycosoil.

3 Evaluation Methodology for Compacted Fly-Ash Mixes

The Department of Road Structures, CTU, Faculty of Civil Engineering, determined the workability, strength characteristics and, with respect to the negative results in case of repetitive exposure to water, a test of resistance to frost and water immersion including the verification of volumetric changes of the mixes examined.

Mixes from both desulphurisation technologies and mixes with various binder proportions were tested. For CCB, the binders used as a standard were replaced by inorganic loose binders obtained by means of mechanical activation of fly-ashes from fluidized combustion, dolomitic limestone and reclaimed concrete materials. Besides such binders, the Iterstab (additive used primarily in soil improvement and stabilization) and Zycosoil (additive on a nanotechnology basis using silane groups) chemical additives were verified; the main benefit is preventing water from entering the mix. The objective was observing the same principles in CCB property modification as well.

One of the objectives of applying mechanically activated materials in CCB was eliminating ettringite formation which is generally one of the limiting factors of CCB application in embedding in roadbed structures of roads.

3.1 Compaction Assessment

Workability of fly-ash mixes was tested by the Proctor standard tests which simulate the compaction achieved by construction rollers very well. The laboratory test of CCB compaction quality is an important test for the assessment of applicability in road construction. CCB compaction effort is related to particle shapes and sizes. The mix compaction quality was examined by the standard Proctor test under CSN EN 13286-2 [16]. Compaction was started after a certain time elapsed from the wetting of the mix – this models the delays caused by transportation, spreading and other handling during real-life paving of the mix. Cement, lime or other additives were added to the dry CCB in samples with additives. The mix was dry-homogenised and wetted only afterwards.

3.2 Compressive Strength Testing

The laboratory test of compressive strength was performed according to the CSN EN 13286-41 [17] standard where a test specimen shaped like a right circular cylinder was loaded by the growing axial stress σ until its failure. The test principle consists in loading the test specimen of hardened energetic by-products with uniaxial compression with a simultaneous measurement of deformation. Strength characteristics after different curing times were studied in detail.

EME fly-ash and ELE bed ash were used to make test specimens by compaction in the laboratory with dimensions of R=100 mm and the height of 120 mm. The test specimens were cured for 7, 14, 21, 28, 60 and 90 days (for some mixes even for 1 year) in a laboratory environment in an airtight cover. The test specimen was also tested for immediate strength after compaction where the prepared specimen was cured at a laboratory temperature of 20-23 °C for approx. 2-3 hours.

3.3 Resistance to Frost and Water Immersion

The preparation and curing of test specimens followed the same process as in the case of compressive strength test. Once the 28-day curing was completed, the test samples were placed on a felt pad partly sunk in water and left to saturate through capillaries until the set weight so that the weight increment for at least 1 hour would not exceed 1 %. All test specimens were saturated in the course of 20 minutes from putting on the felt pad.

Subsequently, the test specimens were placed in a freezer box for 6 hours under -20 °C to -22 °C. After freezing, the test specimens were taken out of the freezer box and stored on a felt pad partly sunk in water for 18 hours to allow further capillary saturation. Simultaneously, de-frosting under +20 °C to +25 °C occurred. The test continued by another round of freezing and repeated 10 cycles according to the method stipulated in the National Annex NB CSN EN 14227-5 [18]. Once the last cycle was completed, a strength test was carried out according to the standard CSN EN 13286-41 [17].

3.4 Swelling Susceptibility of Fly-Ash Stabilizates

Monitoring of CCB volumetric changes is of crucial importance from the perspective of pavement structure durability. Volumetric changes might be demonstrated by shrinking or expansion and, subsequently, result in deterioration of the technical and environmental parameters or, often, complete destruction of the pavement structure.

Further factors affecting volumetric changes of CCB include:

- chemical and physical properties of the input materials;
- risk component content and variability,
- mix design,
- production technology,
- environment in which the CCB is placed (e.g. humidity and thermal parameters, pressure and combination of these factors),
- other specific factors [19].

In relation to the risk of undesired volumetric changes, this test must be viewed with more emphasis. The experimental research examined the impact of the mix on volumetric changes.

The subject matter of swelling measurements for fly-ash stabilizate was determining the linear and volumetric coefficient of swelling. Volumetric changes are understood as increasing of the fly-ash stabilizate volume caused by physical and mechanical processes ongoing in the material, or by additional water absorption.

A CBR bin and other equipment used for the preparation and facilitation of CBR testing under CSN EN 13286-47 [20] were used for the purposes of this test.

The mix saturated to w_{opt}according to the Proctor Standard test was compacted in the CBR cylinder by means of Proctor Standard (PS) energy.

Fly-ash stabilisers were cured for 7 and 28 in moulds under (20±2)°C in impermeable wraps and, subsequently, saturated by water until all deformations ceased to occur. Within the time intervals as mentioned, the changes of surface level of the compacted, saturated samples loaded by a weight were measured.

4 Results and Discussion

4.1 Fly-Ash Stabilized Mixes

4.1.1 Compaction

From the results of optimum compaction assessment done on fly-ash form EME implies, that optimal moisture content of fly-ash mixes with 6 % of CaO addition are only slightly dependent on the content of hydraulic binder in the mix and reach for guidance around 20 %.

Filter fly-ash without any additives showed optimum moisture content for compaction at 21 %, whereas filter fly-ash from EME with 6 % CaO reached optimum moisture content at 20 %.

Tab. 2: Compactability parameters of fly-ash stabilizates.

Mix	Fluidized-bed fly-ash from ELE without additive	Fluidized-bed fly-ash from ELE with 3 wt % of cement CEM II/B-M 23,5R	Fluidized-bed fly-ash from ELE with 6 wt % of pulverized dolomitic limestone	Fluidized-bed fly-ash from ELE with 10 wt % of pulverized fluidized-bed ash	Fly-ash from EME without additive	Fly-ash from EME with 6 wt % CaO
Maximum density [kg/m3]	1122	961	996	1084	1080	1060
Optimal moisture [%]	36,7	34,7	36,2	35,3	21,0	20,0

Compaction quality of fluidized-bed ash from ELE was reached with optimum moisture content about 35 %, if 6 % of pulverized dolomitic lime was added the optimum moisture content increased slightly to 36 %. In case of 10 % activated fly-ash from fluidized combustion the value went up to 38 %. Overall results of mix compactability are given in the Tab. 2.

4.1.2 Compressive Strength Results

From the Fig. 4 and 5 it is apparent, that best values of compressive strength are reached for mixes where mechanically activated fly-ash, dolomitic lime and cement are represented. These mixes fulfilled required threshold limits for compressive strength according to the technical specifications TP 93 [21]. By applying mechanically activated fly-ash form fluidized combustion, as well as dolomitic lime or pulverized recycled concrete the possibility of substituting traditional hydraulic binders by these alternative materials has been proven.

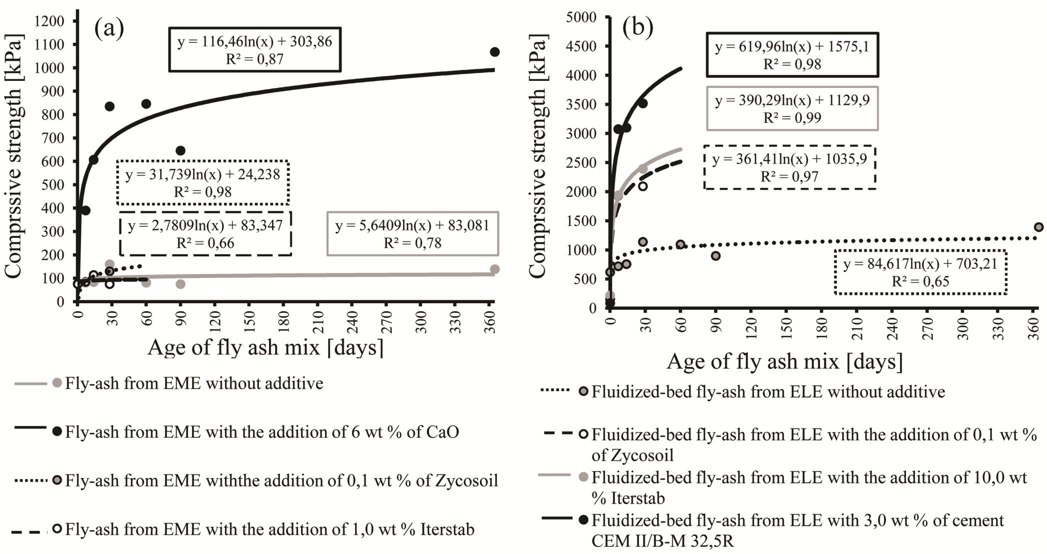

Fig. 4: Compressive strength results of compacted energetic by-products with different additives.

4.1.3 Resistance to Frost and Water Immersion

Resistance to freezing and water depends to a great degree on the composition of the original mix. Stabilizates (deponates) prepared with material from wet scrubber process mixes disintegrated after the first freezing cycle already, or even during the saturation stage. Stabilizates prepared with material from fluidized combustion technology disintegrated after 2 to 3 freezing cycles. Stabilizates made of compacted hydrated mix with

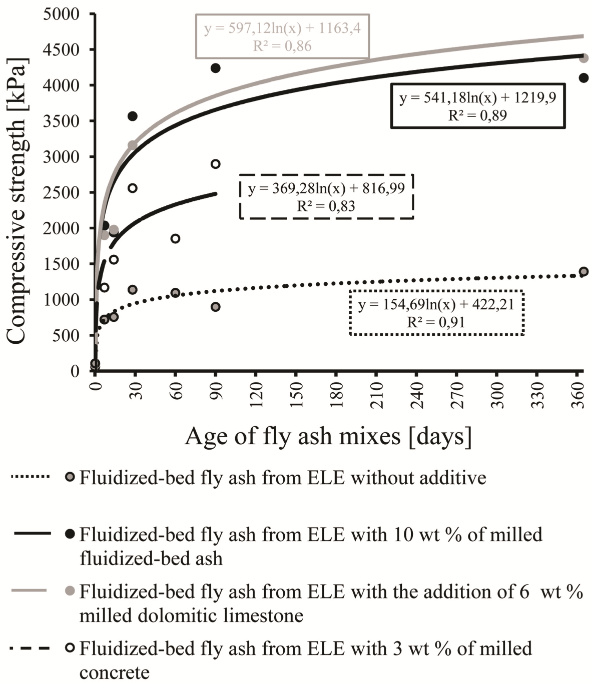

Fig. 5: Compressive strength results of compacted energetic by-products with added mechanically and chemically activated materials.

an addition of mechanically activated fly-ashes from fluidized combustion, dolomitic lime, reclaimed concrete or chemical additives improve the resistance feature of fly-ash mixes against the effects of water and freezing better than stabilizates with no additives. The drop in strength after freezing ranged from 20 to 30 %. For the stabilizate from fluidized-bed ash with the addition of 6 %-wt. mechanically activated dolomitic lime, the original compression strength even increased by 11 % after defrosting. The values must be confirmed again with more test specimen sets.

For the deponate prepared with fly-ashes from EME with no additives, the specimen collapsed in 50 minutes from placement on the felt pad partly soaked in water (see Fig. 6a). Specimens collapsed after the first cycle also in the case of application of chemical additives with 0.1 %-wt. Zycosoil and with 1 %- wt. Iterstab in fly-ashes from EME.

The stabilizate prepared from the EME fly-ashes with 6 %-wt. calcium oxide resisted 10 cycles; however, it demonstrated minor longitudinal and alligator cracks (see Fig. 6b). Compression strength after the last cycle fell to 0.41 MPa, i.e. approx. half of the strength detected in the design stage.

The stabilizate from fluidized-bed ash from ELE showed a transverse crack; the specimen broke after approx. 4 hours' freezing in the second cycle (see Fig. 6c).

In the case of the test specimen from fluidized-bed ash with the addition of 3.0 %-wt. cement CEM II/B-M 32.5R, compression strength fell by 26.5 % after freezing.

Stabilizates from fluidized-bed ash from ELE with the addition of 1 %-wt. Iterstab and 0.1 %-wt. Zycosoil survived 10 cycles and demonstrated the greatest resistance; nevertheless, the strength indicators fell by 19 % for Zycosoil and by 38 % for Iterstab. The stabilisers failed to meet the requirements of CSN EN 14227-14 [22] and, therefore, frost susceptibility according to CSN 72 1191 [23] has always to be determined.

For the stabilizates from fluidized-bed ash with the addition of 10 %-wt. mechanically activated fly-ashes from fluidized combustion, a transverse crack formed after the 10th cycle (see Fig. 6d).

The stabilizate from fluidized-bed ash with the addition of 6 %-wt. mechanically activated dolomitic lime (see Fig. 6e) which was stressed by a graded number of freezing and defrosting cycles demonstrated a value of strength even higher than the initial compression strength. These values must be reconfirmed with more specimen sets.

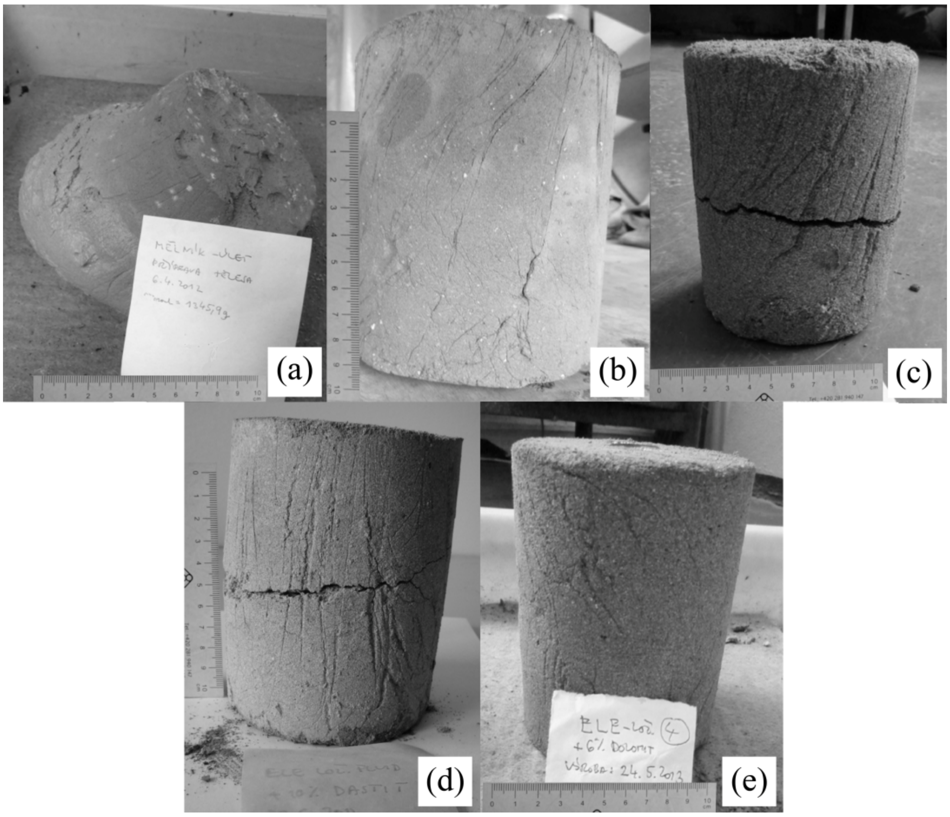

Fig. 6: a) filter fly-ash mix, EME, b) stabilizate EME + 6 wt. %. CaO, c) stabilizate from bed ash ELE without additives, d) stabilizate from bed ash ELE + 10 wt. % of pulverized fly-ash from fluidized combustion – generation plant Plzen, e) stabilizate from bed ash ELE + 6 wt. % pulverized dolomitic limestone.

4.1.4 Swelling of Fly-Ash Stabilizate

Based on the results of measurements of swelling during hardening and setting of the fly-ash stabilizates indicated in Fig. 7, it can be noted that the volumetric changes of the stabilizate amount to rather low values. The greatest volume increase (by approx. 1.5 %) was demonstrated by stabilizate from ELE with no additives after 7 days of curing. It shows no signs of setting even after 40 days. The remaining mixes show a slower volume increase, roughly linear with the hardening time. It can be assumed that the limit value $<$ 3 % of swelling in CBR cylinder under TP 93 [21] will not be reached by all fly-ash stabilizates tested even after a longer examination period. It is likely that a hydration reaction occurs once the stabilizate components are mixed with water and compressed; ettringite forms during the reaction. In contrast to other materials with hydraulic bonds (e.g. concrete, cement-stabilized soil), ettringite's slight tendency to swell is a prevention against shrinking during hydration.

5 Conclusion

The present article focused on the possibility of using CCB by-products directly or applying them after certain modifications (through improvement of the properties required) in roadbed structures of roads, or even as structural layers of the pavements.

Laboratory research of CCB has emphasised the dominant influence of the technology applied to desulphurisation on the resulting CCB characteristics. The results of long-term CCB testing have proven that besides the deposit from EME, mechanical properties of fly ash stabilisers improve significantly in time. The results obtained have confirmed that highest values of simple compressive strength are achieved by mixes with a representation of ground (mechanically and chemically activated) fluidized fly-ash, pulverized dolomite limestone or recycled concrete and, therefore, such mixes meet the minimum values required for simple compressive strength according to technical specifications TP 93, [21].

Based on the results yielded by the measurement of fly-ash swelling capacity, it can be concluded that the volumetric changes of the stabiliser amount to relatively small levels.

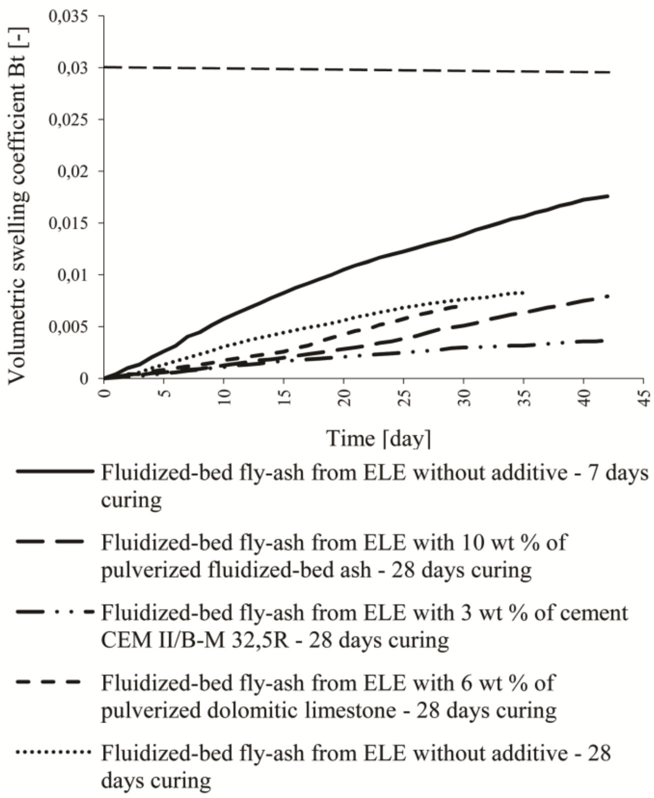

Fig. 7: Time dependent progression of fly-ash stabilizate swelling.

Frost and water susceptibility testing has indicated that the deposits and stabilisers tested are not resistant to frost and water. The majority of the samples tested disintegrated in the course of ten freezing and thawing cycles or, subsequently, compressive strength fell considerably after the last cycle.

A stabiliser made of compacted, slightly wet mix with the addition of mechanically and/or chemically activated mineral materials or chemical additives partly improves the characteristics of fly-ash mix resistance to water and frost when compared to a stabiliser alone (no additives). The application of mechanically and chemically activated materials or used additives facilitates elimination of some CCB problems, while indicating a possibility of substitution for lime, or cement.

Acknowledgement

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Complex Modulus and Stiffness Modulus of Cold Recycled Mixes

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Abstract: This article introduces part of the research done within the international project CoRePaSol focused on cold recycled mixes. Standard design for cold recycled asphalt mixes specifies the use of bituminous emulsions, foamed bitumen or hydraulic binders. In Central European countries, often the combination of cement and bituminous binder is used as the most preferable solution because of increased bearing capacity which can be provided by the final structural layer similarly to cement stabilized materials. For this reason it is expected that strength properties as well as stiffness are improved, nevertheless the strain-related behaviour explained usually by stiffness modulus, resilient modulus or complex modulus is not largely assessed. During the experimental study, cold recycled mixtures with bituminous emulsion or foamed bitumen and different cement content were assessed by the repeated indirect tensile stress test (IT-CY) evaluating different curing periods. Since the main objective of this paper is to compare stiffness and dynamic complex modulus, testing has been done for selected mixes using the four-point beam test (4PB-PR). The focus was oriented on possible correlations and comparability of values gained by these two tests characterizing the strain behaviour of the material.

Keywords: Stiffness Modulus; Complex Modulus; Strain-Related Behavior; Cold Recycled Mix.

1 Introduction

Cold recycled asphalt mixes are multiphase systems made from several components. Some of them have complicated internal structure and show increased thermal susceptibility. In fact, reclaimed asphalt pavement (RAP) consists of irregularly shaped aggregate, bituminous binder (usually of unknown origin and range of its ageing) and air voids. Despite the complex structure, the material behavior can be described by application of known theory of viscoelasticity [1–4].

One of the determining characteristics of bituminous materials, and thus also cold recycled asphalt mixes, is that they have time-dependent behavior, which can be observed especially when deformation (strain) behavior is determined. When these mixes are subjected to a very small loading, it results in a combination of elastic, delayed elastic and viscous behavior. In the range of low temperatures and high loading frequencies, these mixes behave like elastic material in a solid phase with almost fully reversible response. At elevated temperatures and low stress frequency, the behavior of these mixes is similar to viscous liquids with irreversible response. In the range of moderate temperatures and for the whole given frequency range, asphalt mixes exhibit viscoelastic behavior, or more precisely delayed response combined with viscous flow.

Rheological performance-based measurements, which result in characterization tools like complex (master) curves, depict the behavior of the cold recycled mix in the most complex way and provide information about functionalities e.g. between the complex dynamical modulus and stress duration and frequency of loading. The value of the complex modulus and the slope of the master curve can be considered as valuable information for prediction of mentioned functional properties of cold recycled asphalt mixes. This is the reason why it is recommended to perform assessment of pavement structures by applying preferably time-demanding and more difficult but simultaneously more complex performance-based tests. One of the methods, which can be considered for the assessment of deformation behavior, is four point beam bending test (4PB-PR). With this test it is possible to get values of complex dynamic modulus for given temperatures and stress frequencies [5].

Another simple performance-based test which is also suitable for characterization the ability of a mix to resist the effects of loading is determination of the stiffness modulus, usually by the repeated indirect tensile

Tab. 1: Evaluated experimental mix designs.

Mix	Bituminous emulsion	Foamed bitumen	Cement
		[% by mass]	
BCSM-BE	3.5 %	-	3.0 %
BCSM-BE (ref 1)	3.5%	-	1.5%
BCSM-BE (ref 2)	3.5%	-	1.0%
BSM-BE	3.5 %	-	-
BCSM-BE (ref 3)	2.5%	-	1.0%
BCSM-FB	-	4.5 %	3.0 %
BCSM-FB (ref 1)	-	4.5%	1.0%
BSM-FB	-	4.5 %	-

stress test (IT-CY). Both tests are not required for the cold recycled mixes in many countries, where cold recycling is regularly used. Therefore the testing procedures were assumed from relevant European standard for hot asphalt mixes [6].

The main objective of this paper is a detailed investigation of the cold recycled mix behavior by applying these performance-based tests. Findings from these tests were compared to the results of the indirect tensile strength test (ITS), which is usually required for declaring the quality of cold recycled mixes in most countries because of its simplicity. The output of this test is, nevertheless, just one empiric value for a given temperature, frequency and time of specimen curing.

2 Materials

For experimental evaluation of stiffness modulus and dynamic complex modulus different types of cold recycled mixes were designed representing possible options of used binders and their combinations. Tab. 1 summarizes the material composition of designed cold recycled mixes in terms of used binders with emphasis on mixtures, where either bituminous emulsion or foamed bitumen are used in combination with hydraulic binder (cement).

Within the cold recycled mix assessment it is differentiated which of the two binder types is dominant in the mix. Basic set for the laboratory evaluation consists of four mixtures, namely BCSM-BE (mixture containing 3 % of cement in combination with bituminous emulsion), BSM (mixture containing only bituminous emulsion), BCSM-FB (mix containing 3 % of cement and foamed bitumen) and BSM-FB (mixture containing only foamed bitumen). For these four basic mixtures the complex modulus at various temperatures and frequencies was tested as well. For the determination of the stiffness modulus by IT-CY this set was completed by four other cold recycled mixtures with different content of bituminous and/or hydraulic binder. In fact more cold recycled mix options have been tested including evaluating potential and impact of fly-ash or lime.

All designed mixes contained the same type of screened RAP with 0/22 mm grading; cement CEM II / B 32.5 and bituminous emulsion C60B8 were used. For the production of foamed bitumen standard bitumen 70/100 according to EN 12591 was applied. When preparing the foamed bitumen 3.8 % of water was added. The amount was determined in accordance with the procedure which is recommended for cold recycling technology by [7]. Foamed bitumen was injected in the mix at the temperature of 170 °C by means of the Wirtgen WLB10S laboratory equipment. The mix as such was mixed using a twin-shaft compulsory mixing unit Wirtgen WLM 30. The optimal moisture content of the cold recycled mix for the composition specified in the Tab. 1 was determined according to [8].

3 Methodology

3.1 Determination of Stiffness Modulus by the IT-CY Method

The bearing capacity of a pavement layer is usually characterized by stiffness or modulus of elasticity. Stiffness modulus is defined as a ratio of material stress and strain and it characterizes its ability to resist the effects of loading. Higher stiffness value means that the material is more resilient to traffic loading than the material with a lower value. It usually means that better resistance to permanent deformations can be expected by the mixes with higher stiffness, on the contrary it is more difficult to find a straight relation to fatigue life.

Stiffness modulus was determined according to repeated indirect tensile stress test (IT-CY) in compliance with [6]. It is a non-destructive performance-based test with good reproducibility and repeatability, during which e.g. the Nottingham Asphalt Tester device (Fig. 1) loads the test specimen by a vertical pulse characterized by the force (P), which causes horizontal deformation (Δ). Effects of the vertical force are transferred to the horizontal - perpendicular - direction by the Poisson ratio (μ), which is dependent on the type of material as well as on the specimen temperature. That is because the ratio of the perpendicular axial deformation or the ratio of orthogonal axial force varies at different temperatures. Stiffness modulus characterizes short term rheological behavior of asphalt mix taking into account deformations lasting only for tens or hundreds of milliseconds.

The cylindrical specimens of 150±1 mm diameter were prepared by putting the cold recycled mix in cylindrical moulds and compacting by the static pressure of 5.0 MPa. For all test specimens basic volumetric properties, as well as the indirect tensile strength according to [9] and stiffness modulus at 15 °C according to [6] were determined. Specimens were tested after 7, 14 and 28 days. All cold recycled mixes were stored for one day at 90-100 % relative humidity and temperature of (20±2) °C. Further the specimens were stored at laboratory conditions with 40-70 % relative humidity and temperature of (20±2) °C for the rest of their curing time.

Fig. 1: IT-CY test in Nottingham Asphalt Tester.

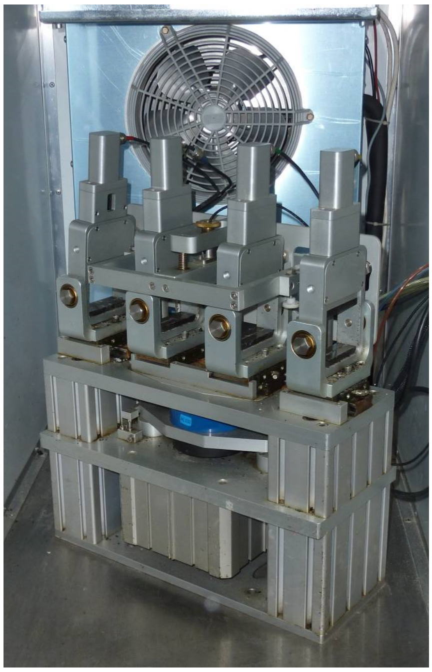


Fig. 2: Measuring device 4PB-PR.

3.2 Measurement of Complex Modulus by the 4PB-PR Method

Measurement of complex modulus was performed according to [6]. This standard prescribes that a beam specimen with smooth surface and entire edges is clamped by clips in the test equipment in four points (4PB-PR). All clamps should allow free rotation and shift in the longitudinal direction. The outer clamps should be firm, to defend vertical movements. Inner clamps deduce vertical cyclic loading. The evaluation of the test results is based on Euler-Bernoulli theory.

4PB-PR is a simple test, which can be characterized by a simple mechanistic theory, whereas gained results are important values applicable to the pavement design purposes. Nevertheless such conclusion cannot be fully applied for several reasons which are further summarized. The respective testing is influenced by factors, which introduce to the testing methodology errors or deviations which influence the overall result. These conditions are not attended by the testing methodology in any manner. It is the only question if it is possible to avoid them.

Firstly, the testing beam has to be locked in by the clamps. Such clamping locally constitutes stress and deformation which is introduced to the beam material. This extra stress and deformation can represent fatigue damage or local non-linear effects not included in the calculation of complex modulus.

Secondly, it is not possible to design 4PB-PR apparatus, which would accept friction, allow at the same time free displacement and will not deteriorate during the life-time. Such deformations related to the apparatus might influence the test results.

Thirdly, shear forces act on the test beam in the area between the outer and inner clamps. These forces are causing additional testing beam deformation, whereas such deformations are not taken into account in the used test methodology. Forces related to the shearing strain in this area equal F/2, i.e. the half of applied total loading.

Fourthly, clamps limit the possibility of displacement in cross-section. This leads according to our experience to ineligible deformations of the testing beam in the areas close to the clamps and this is not in accordance with the deflection theory.

Fifth factor influencing the test results is the specimen shape factor, which can be defined as a function of shape and test specimen dimensions. Similarly weight factor should be considered as well. This can be defined as a function of beam weight and apparatus weight, which by its inertial force influences acting force. The magnitude of measurement error or inaccuracy is dependent on the shear modulus and Poisson's ratio related to the tested material. These two factors are included in the calculation of complex modulus [10].

Dynamic complex modulus was determined by the 4PB-PR test method (Fig. 2) in the temperature range from -20 to +27 ° C and frequency from 0.1 to 40 Hz by the controlled deformation of 50 microstrain. These frequencies correspond to the real pavement loading caused by vehicles passing with different speed in the aforesaid temperature range.

Testing slabs were produced by segment compactor and after their curing as specified further the slabs were cut to required shape of beam specimens according to [6]. Respective curing of specimens (based on the CoRePaSol project recommendations) was as follows:

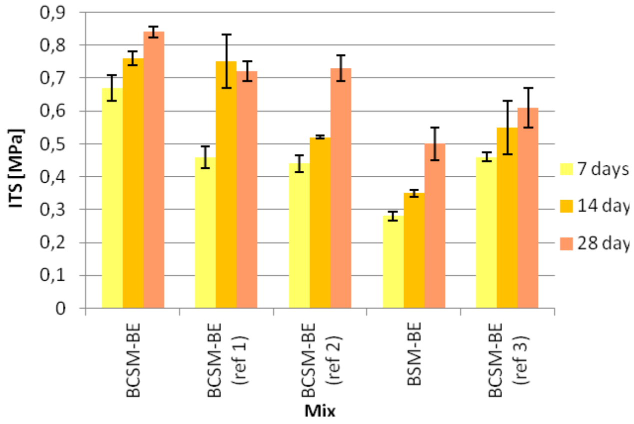
- Cold recycled mixes stabilized by cement and bituminous emulsion or foamed bitumen were firstly stored two days at 90-100 % relative humidity and temperature of (20±2) °C. Further the test slabs were stored in dust free area of the laboratory at 40-70 % relative humidity for additional 26 days at the same temperature.
- Cold recycled mixes stabilized by bituminous emulsion or foamed bitumen were stored for 1 day at 90-100 % humidity and temperature of (20±2) °C. Further the test slabs were conditioned at (50±2) °C in a climatic chamber for additional 4 days. The test specimens were then stored at 40-70 % humidity and the temperature of (20±2) °C for 14 days after this accelerated curing.

4 Results for Stiffness and ITS Testing of Cold Recycled Mixes

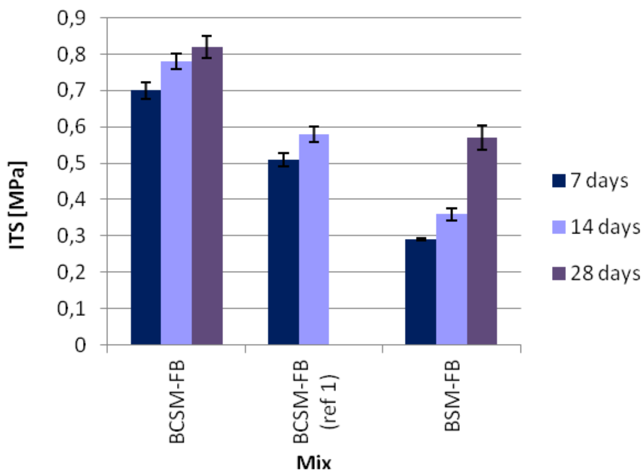
Fig. 3 and Fig. 4 show successive increase in both determined characteristics during the first 28 days of test specimens curing. At the same time the extent of characteristic increment in relation to the added hydraulic binder is illustrated.

In general it is possible to state, that time-dependent increase of stiffness modulus does very well correspond with indirect tensile strength values. From the point of view of both characteristics it is nevertheless possible to observe a difference if mixes with diverse content of cement are compared.

As can be induced from the Tab. 2 increase of both characteristics is always faster for mixes with higher cement content. The table summarizes selected values of ITS and stiffness for mixes with same bituminous binder content and 0 %, 1 % and 3 % cement. If comparing the assessed curing period between 7 and 28 days it can be stated that for mixes with higher content of hydraulic binders faster increase in strength properties is visible within the first 7 days. For the rest of the evaluated curing period the strength increase is rather slow. On the contrary for mixes containing only bituminous binder a very slow strength enhancement can be observed and even between the 14 and 28 days of curing there is still significant increase of the strength values.

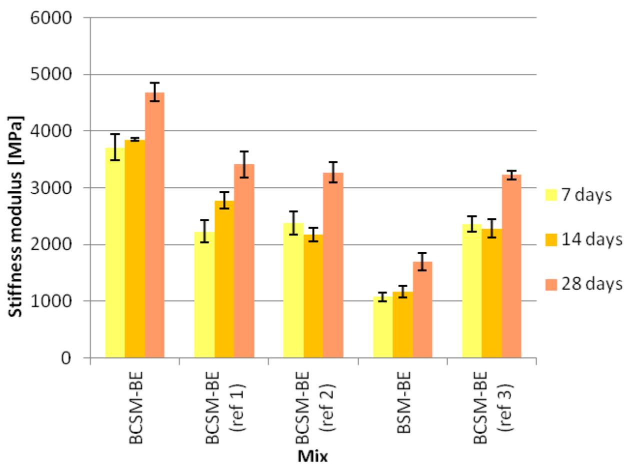


(a) mixes with bituminous emulsion

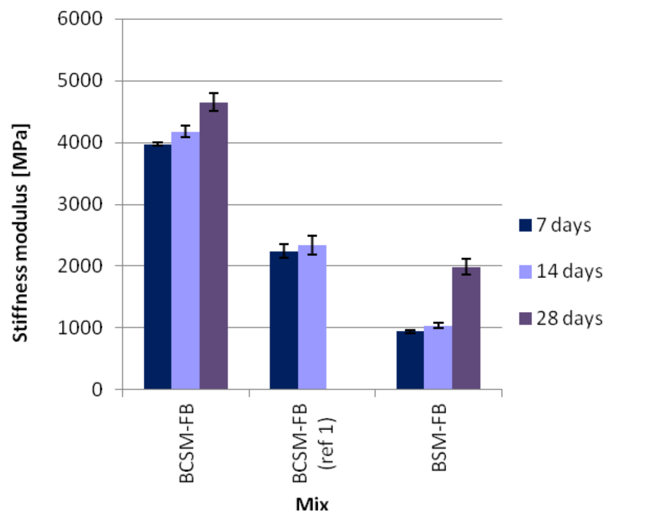


(b) mixes with foamed bitumen

Fig. 3: Indirect tensile strength of all tested cold recycled mixes.



(a) mixes with bituminous emulsion



(b) mixes with foamed bitumen

Fig. 4: Stiffness modulus of all tested cold recycled mixes.

From the presented stiffness and ITS results following conclusions can be made. There is an important difference in values determined after 7 days specimens curing for cold recycled mixes with cement and without cement. This difference then gradually decreases as can be seen in Tab. 3. Further, it is possible to show, that the use of cement has more positive influence on stiffness values than on indirect tensile strength values. Such finding does very well correlate with values gained also for other assessments done within the project CoRePaSol.

5 Results for 4PB-PR Testing of Cold Recycled Mixes

Results of dynamic resilient modulus determined by dynamic loading using 4PB-PR test method are summarized in the Tab. 4. The shown values are always determined as an average from last 10 measured values (complex dynamic modulus, loss angle) at different temperatures and for different frequencies. These results were used for decomposing the complex modulus to its real and imaginary part. These decomposed values are necessary, because they are used as input data in IRIS Rheo-Hub software. This calculation software (or any similar available on the market) allows successively to calculate and adjust the so called master curves.

The master curves were designed by using time-temperature superposition principle with relevant shift in horizontal and vertical plane. Shift factors were used to determine the temperature dependence of rheological behavior and extension of time-frequency range at given reference temperature.

Tab. 2: Time influence on ITS and stiffness modulus values.

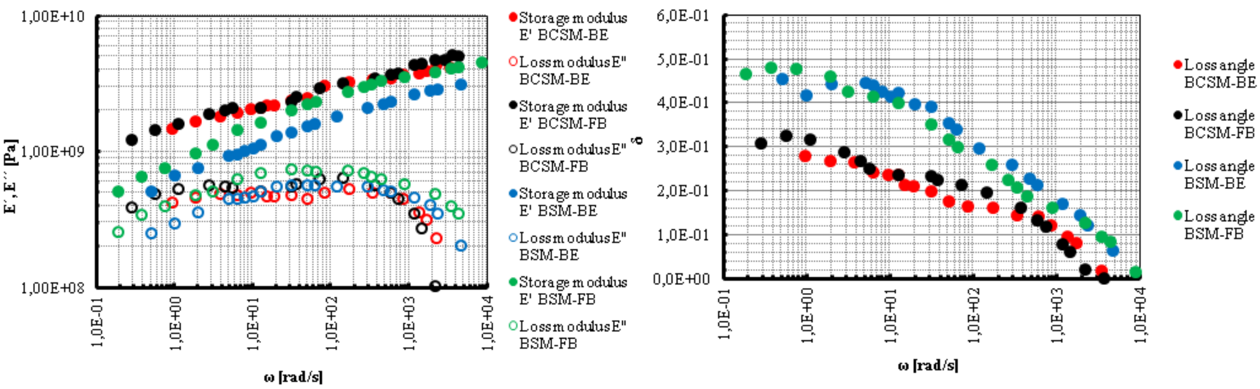
Indirect tensile strength [MPa]						
		BSM-BE		BSCM-BE (ref2)		BSCM-BE
7 days	0.28	100 %	0.44	100 %	0.67	100 %
14 days	0.35	25 %	0.52	18 %	0.76	13 %
28 days	0.50	79 %	0.73	66 %	0.84	25 %
		BSM-FB		BCSM-FB (ref 1)		BCSM-FB
7 days	0.29	100 %	0.51	100 %	0.70	100 %
14 days	0.36	24 %	0.58	14 %	0.78	11 %
28 days	0.57	97 %	-	-	0.82	17 %
Stiffness modulus [MPa]						
		BSM-BE		BSCM-BE (ref2)		BSCM-BE
7 days	1076	100 %	2380	100 %	3717	100 %
14 days	1164	8 %	2177	-9 %	3852	4 %
28 days	1695	58 %	3274	38 %	4687	26 %
		BSM-FB		BCSM-FB (ref 1)		BCSM-FB
7 days	941	100 %	2240	100 %	3971	100 %
14 days	1036	10 %	2331	4 %	4175	5 %
28 days	1988	111 %	-	-	4652	17 %

Tab. 3: Influence of cement content on ITS and stiffness.

Indirect tensile strength [MPa]						
	7 days		14 days		28 days	
BSM-BE	0.28	100 %	0.35	100 %	0.50	100 %
BSCM-BE (ref 2)	0.44	57 %	0.52	49 %	0.73	46 %
BSCM-BE	0.67	139 %	0.76	117 %	0.84	68 %
BSM-FB	0.29	100 %	0.36	100 %	0.57	100 %
BCSM-FB (ref 1)	0.51	76 %	0.58	61 %	-	-
BCSM-FB	0.70	141 %	0.78	117 %	0.82	44 %
Stiffness modulus [MPa]						
BSM-BE	1076	100 %	1164	100 %	1695	100 %
BSCM-BE (ref 2)	2380	121 %	2177	87 %	3274	93 %
BSCM-BE	3717	245 %	3852	231 %	4687	177 %
BSM-FB	941	100 %	1036	100 %	1988	100 %
BCSM-FB (ref 1)	2240	138 %	2331	125 %	-	-
BCSM-FB	3971	322 %	4175	303 %	4652	134 %

For the description of the shift factor in horizontal plane Williams-Landel-Ferry equation with C1 and C2 parameters was used. For the description of the shift in vertical plane a polynomial equation is preferred [2, 11]. The parameters of the vertical and horizontal shift are listed in the Tab. 5. All calculations were made in the IRIS Rheo-Hub software tool. The reference temperature for master curve determination was set at 20 °C.

As generally known, temperature and time have dramatic influence on viscoelastic response of bituminous binders and asphalt mixes. This is the reason why viscoelastic properties are usually determined in wide spectrum of temperatures and/or stress levels. Behavior of an asphalt mix or bitumen depends on the temperature as well as frequency and duration of repeated loading. If these variables are in a range, where the material behavior is defined as viscoelastic and it is possible to characterize the material as temperature-rheologically "simple", then it is possible to express the effect of time (frequency) and temperature by time-temperature superposition. Material characteristics given as a time-dependent function or frequency-dependent function (such as results of dynamic testing, or material spectrum measured at different temperatures), can be shifted along axes for creating various master curves [2]. The master curves of the researched cold recycled asphalt mixes with different binders (bitumen, cement, or their combination) are given in Fig. 5.



(a) storage modulus and loss modulus (b) loss angle

Fig. 5: Master curves of cold recycled mixes.

When analyzing the master curves from the right to the left, it is evident, that in the field of the lowest tested temperatures and highest frequencies, the material has tendency to behave almost elastically. The transition from viscoelastic to elastic behavior is evident from the last determined values in the decreasing imaginary part of the master curves. The real part of the master curve is increasing proportionally to the raising frequency. At the same time the first derivation is decreasing and is reaching almost zero level for the last couple of values. This phenomenon correlates very well with the transition of the material properties to the elastic area, where equilibrium modulus is reached [2, 12]. From the loss angle change it is clear, that cold recycled asphalt mixes are thermo-mechanically sensitive materials [3]. Comparing assessed mixes in terms of used binders, the influence of cement is evident in enhanced elastic properties at high temperatures and low frequencies. At the same time thermal susceptibility is decreased if cement is used. This fact is evident from comparing the elastic modulus curves, where the slope of elastic curve is more gradual for mixes with cement (less difference between the lowest and the highest determined modulus).

The difference between foamed bitumen and bituminous emulsion is apparent especially in the elastic modulus master curve. The variation might be partly caused also by different residual binder content. The binder itself will not play a role since for foamed bitumen and the bituminous emulsion 70/100 bitumen was used. The applicability of the test for cold recycled mix assessment seems however to be complex, because the correct preparation of unpaired beam specimens came repeatedly up with certain problems. The clear reason is the brittle character of these stabilized materials. This leads to low reproducibility of the results as well.

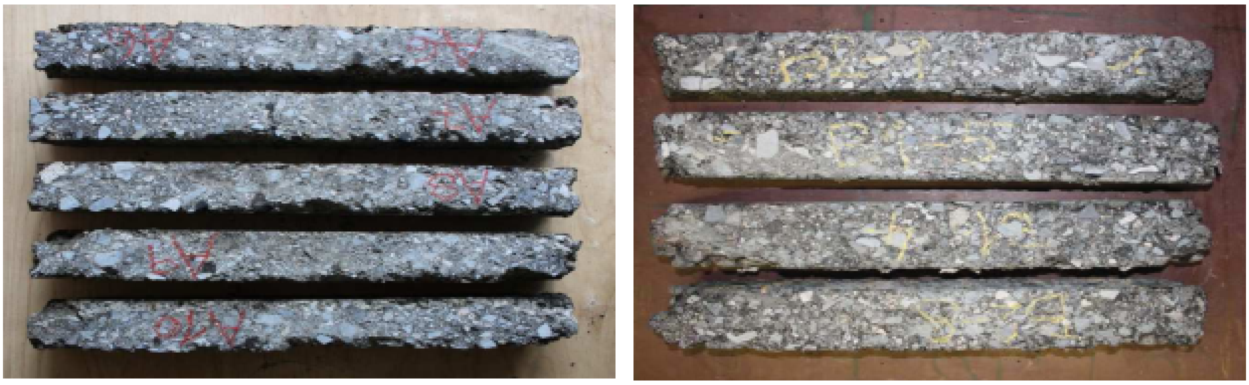
First problem related to cold recycled asphalt mixes and 4PB-PR testing appeared already during demoulding of the test slabs. Because the material is more brittle, it is necessary to secure sufficient separation between the steel bottom plate of the mould and the compacted material. Even if the separation is made properly, sometimes the whole slab can be broken during demoulding. Another problem occurred when beam specimens were cut from the slabs to get the necessary test specimens. During the cutting the material has tendency to brake off edges of the beams (figures 6 and 7). This finding is not unique. The same was found out in the past within several studies and master student projects at the CTU in Prague, as well as during fatigue experiments on cold

Tab. 4: Dynamic complex modulus and phase angle values (4PB-PR test method).

Testing temp.	Frequency	BCSM-BE		BCSM-FB		BSM-BE		BSM-FB	
[°C]	[Hz]	E* [MPa]	δ [-]	E* [MPa]	δ [-]	E* [MPa]	δ [-]	E* [MPa]	Δ [-]
0	50	5 930	0.00	6 459	0.00	6 211	0.00	7 543	0.00
	30	5 482	0.00	6 162	0.03	5 619	0.52	7 509	0.00
	20	5 100	1.05	5 782	0.59	5 137	3.74	7 050	0.92
	10	4 745	4.65	5 482	3.45	4 761	7.00	6 570	4.79
	8	4 589	5.45	5 385	4.53	4 651	8.25	6 453	5.54
	5	4 472	7.00	5 149	5.80	4 396	9.82	6 095	7.25
	2	4 195	8.29	4 941	7.57	3 972	12.33	5 599	9.20
	1	4 012	9.24	4 702	8.39	3 627	13.83	5 249	10.82
	0.5	3 721	9.37	4 461	9.09	3 309	14.23	4 833	11.52
10	50	4 620	0.03	5 669	0.10	4 495	5.80	5 222	0.15
	30	4 210	3.03	5 203	1.22	3 960	5.78	4 723	5.43
	20	3 729	4.08	4 582	3.87	3 503	8.95	4 156	7.35
	10	3 482	7.13	4 152	6.76	3 164	12.24	3 651	11.97
	8	3 403	8.12	4 066	7.63	3 053	13.05	3 565	12.96
	5	3 243	9.38	3 851	9.29	2 855	14.88	3 261	14.90
	2	3 314	11.32	3 551	11.23	2 515	16.98	2 794	17.20
	1	2 844	11.56	3 247	12.28	2 298	18.21	2 438	19.10
	0.5	2 614	11.87	2 842	12.82	1 981	18.81	2 037	20.91
20	50	3 870	0.00	2 852	12.69	2 829	6.44	3 892	5.20
	30	3 441	4.20	2 411	14.71	2 605	11.95	3 416	9.93
	20	2 955	4.85	2 039	26.11	1 983	14.86	2 827	13.29
	10	2 631	8.92	1 946	14.57	1 693	19.41	2 432	17.10
	8	2 550	10.10	1 955	15.97	1 637	20.28	2 343	18.06
	5	2 413	11.42	2 421	13.33	1 500	22.37	2 157	20.18
	2	2 178	13.47	2 174	13.58	1 239	24.15	1 780	22.87
	1	2 001	13.95	2 181	16.28	1 062	25.18	1 565	23.78
	0.5	1 704	12.89	2 078	16.60	809	23.67	1 241	24.40
30	50	3 053	6.66	3 168	3.61	1 925	17.01	2 398	8.68
	30	2 701	9.26	2 732	6.87	1 566	20.79	2 107	16.65
	20	2 115	8.88	2 207	9.27	1 120	22.72	1 537	19.33
	10	1 872	12.08	1 967	14.35	910	23.81	1 260	23.35
	8	1 852	12.23	1 889	15.31	879	24.43	1 211	24.67
	5	1 769	13.55	1 784	16.51	820	25.55	1 085	26.39
	2	1 554	15.26	1 524	18.19	654	25.36	859	27.31
	1	1 455	15.31	1 367	18.58	581	23.97	735	27.58
	0.5	1 271	15.96	1 150	17.67	452	26.01	568	26.74

Tab. 5: Parameters of horizontal and vertical shift within the time-temperature superposition.

Mix		BCSM-BE	BCSM-FB	BSM-BE	BSM-FB
$_{\mathrm{ref}}$	[°C]	20			
T_{min}	[°C]	0			
T_{max}	[°C]	30			
Fit of horizontal shift aT [K]	C1	100	100	100	100
	C2	1458.3	1215.5	1249.9	1016.7
Fit of vertical shift bT [-]	a_0	1.07	1.01	9.89E-01	9.94E-01
	a_1	1.1E-02	1.00E-02	2.52E-02	6.5E-03
	a_2	-2.4E-05	3.50E-05	2.96E-04	-5.7E-04



(a) mix BSCM-BE (b) mix BSCM-FB

Fig. 6: Damaged beam specimens before testing – mixes with cement.

(a) mix BSM-BE (b) mix BSM-FB

Fig. 7: Damaged beam specimens before testing – mixes without cement.

Tab. 6: Stiffness modulus determined by IT-CY method.

Mix	Stiffness modulus (IT-CY) @ 15 °C [MPa]
BCSM-BE	4 287
BCSM-FB	4 652
BSM-BE	1 745
BSM-FB	2 254

recycled asphalt mixes made e.g. at the University College in Dublin.

To compare the methodology of stiffness modulus testing by IT-CY and the dynamic modulus gained by 4PB-PR test, selected values of IT-CY stiffness modulus are shown in the Tab. 6. The comparison itself is a challenging and ambiguous because of different method of test specimen manufacturing (segment compactor for 4PB-PR vs. hydraulic press for IT-CY) as well as the principles of loading. However, the results show some trend or as the case may be it is possible to make some assumptions on the gained findings. Stiffness of cold recycled mixes with cement correlate more with the results of complex dynamic modulus at higher frequencies of loading (30-50 Hz), while the mixes with only bituminous binders have stiffness according to IT-CY more similar to lower frequencies (2-5 Hz) of 4PB-PR testing. The given comparison is just approximate due to the fact, that complex dynamic modulus were determined at temperatures of 10 °C and 20 °C, while the stiffness test is according to the standard procedure in the Czech Republic done at 15 °C.

6 Conclusion

Results presented mainly for determination of stiffness on cold recycled mixes represent only a small part of a more complex study done within the CoRePaSol project, where also other optional designs of this type of stabilized material were tested and compared, e.g. with various bituminous binders for foam production, with different types of reclaimed materials including recyclable concrete or unbound base layer material. Additionally stiffness values are available even for cold recycled mix options with multiple recycled asphalt, which was laboratory aged, re-crushed and re-used in a new cold recycled mix.

In general it can be stated that stiffness modulus determination by using IT-CY test method is a suitable procedure for cold recycled mix assessment and can be always done in parallel to indirect tensile strength test. This destructive test is so far used more often when characterizing cold recycled mixes. Stiffness determination is required in smaller number of countries using cold recycling techniques. It is therefore possible to recommend use both test methods – IT-CY and ITS test. Results from both these tests complement each other very well. Additionally stiffness determination has so far better shown the curing time dependency as well. On the other hand, if comparing ITS and stiffness for mix design with increasing cement content, stiffness modulus seems to be less sensitive to cement content in the mix, if comparing results to cold recycled mixes with same RAP but only bituminous binder content. This is valid mainly for results gained after more than 14 days curing when the difference becomes very small.

On the other hand, complex modulus was assessed only by 4PB-PR test procedure. It was known that, in the past, doubts were raised about suitability of the test especially because of test specimen preparation. It was proven by the research study within CoRePaSol project that the test would provide more data and most probably could better explain the behavior of the tested material. Nevertheless, reproducibility of the test seems to be very low mainly influenced by the quality of gained test specimens. Solving initial problems of demoulding $30 \times 40 \times 5$ cm test slabs it was then usually very problematic to properly cut test beams. Gained test specimens showed always some damages like loss of aggregate particles or impaired edges, which immediately influence the test results. Despite these facts, it was possible to get data for a selected temperature and frequency interval and even plot master curves. Nevertheless, the variation of measured values is much higher than in case of IT-CY stiffness modulus test.

Simple comparison of IT-CY and 4PB-PR was done to evaluate whether there is any correlation between IT-CY stiffness and complex modules at some frequency, ideally concurrent for all tested cold recycled mix designs. As was discussed in the article, such correlation was not found, only presumptions can be made for cold recycled mixes with or without cement.

From the practical point of view and expectations of the National Road Authorities as defined within the CoRePaSol project, if the 4PB-PR test would be required for cold recycled mix characterization, it could be performed only by a limited number of laboratories requiring suitable test apparatus and expecting more time demanding procedure for test specimen preparation.

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Impact of Mechanical Chemically Activated Rubber on Strain and Flow Behaviour of CRmB Binders

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Abstract: One of the progressive trends which have been developed intensively in the last few years in road structures engineering are technical possibilities for optimal utilization of recycled rubber coming from old tires in asphalt mixtures and bituminous binders. In this connection there exist already several techniques, but at the same time, the knowledge about alterations in strain behavior of bitumen if modified by crumb rubber is still insufficient. Furthermore, the problem of heterogeneity of final composite binder is well known and not qualified completely. Presently known and used milling techniques lead only to a partial solution. Experimentally, the focus is therefore oriented on possible use of so called mechanical chemically activated micro-milled rubber gained by the disintegration process. Application of this treated rubber material should allow sufficient stability of resulting bituminous binder. The paper summarizes experimentally made modifications with use of milled and micro-milled waste rubber together with application of further chemical additives, like polyphosphoric acid, which improve the stability of binder composite structure. Alteration in strain behavior and in dynamic viscosity values are assessed by means of rheological testing of bituminous binders which have not been aged o degraded. In this study, oscillatory measurements of strain characteristics (complex shear modulus in terms of temperature or frequency sweeps) have been done in temperature range 20-70 °C and in a wider frequency interval, simulating various traffic loading effects. Measurements are executed strictly in linear area of viscoelastic behavior of this material. Simultaneously, multi-creep stress recovery test with determination of characteristics describing compliance were performed. Last but not least, flow curves describing the level of workability of designed modified bituminous binders are compared and analyzed by application of rotational viscosimetry. Recommendations for further use of bitumen modification by pulverized rubber are given including potentials in increased strain resistance.

Keywords: Crumb Rubber Modified Bitumen; Dynamic Shear Modulus; MSCR Test; Dynamic Viscosity; Activated Rubber.

1 Introduction

As stated in different studies elaborated worldwide, it is assumed that the yearly waste production of old tires reaches more than 110-115 mil. tires of different types and composition. This might represent more than 7,000 kT of recyclable rubber most of it coming from EU countries and North America [6]. In many countries, different regulations and legal standards have been set for waste management of old tires. In the developed countries, it has for several years forbidden putting of the old tires on landfills and other solutions are forced. If following the European waste management strategies, the most preferred solution is recycling and reuse, in the second step then is energy use. The areas which are for several decades understood as potential fields for crumb rubber utilization are asphalt pavements and suitable bitumen modifications.

Generally, two methods are known for crumb-rubber use in asphalt mixes: (1) dry process during which crumb rubber is added directly to the asphalt mixture as a flexible modifier and to substitute part of the finer aggregates and (2) wet process where the bitumen is modified. Materials described in this paper follow the second process. Nevertheless one of the crucial issues related to this product is the homogeneity of the final crumb rubber modified bitumen (CRmB). Of course it is possible to produce CRmB directly at an asphalt mixing plant and there are various solutions of continuous blending. The question always prevails if this is the most suitable solution guaranteeing high-performance ready-to-use binder. If CRmB is produced industrially in a refinery, quality control is better and final products properties are declared. In case of EU, the producer is further responsible for necessary steps related to European directive on Registration, Evaluation, Authorization and Restriction of Chemicals (REACH). The target is to get a binder which can be transported for longer distances and can be stored for several days. Homogeneous binder is required, which is usually not easy to achieve because of very strong chemical sulfur bonds in the rubber. Several approaches can be found worldwide, e.g. based on polyphosphoric acid or macrocyclic polymers, to improve the stability between bitumen and dissolved rubber particles. Usually the additive itself is not the overall solution and the composite material crumb rubber-bitumen-additive works only for limited rubber content.

Based on this knowledge, this paper focuses on two issues. (1) using a special type of disintegration technique for producing pulverized rubber with particles <0.8 mm including impact assessment of such rubber on bitumen performance and (2) analyzing several types of additives which might help to produce a storage stable product.

2 High Speed Milling (Grinding)

Different industrial field may presently exist without mechanical crushing or pulverization of materials, starting from the exploitation of natural resources, power generation and metallurgy to the paper industry or the production of building materials. The majority of industrial products as known today could not exist without grinding [5], which is understood as a process of refining the grain size and increasing the specific surface of material, but also as a process of opening particle grains [8].

One of the trends which has been going through intensive development in the recent time is high energy milling (HEM). High speed milling (HSM) is understood as a sub-type of HEM which is characterized by large amounts of energy transferred per material unit treated in the pulverizing process. The term of HEM or HSM still lacks precise definition in the literature. It shares all its basic aforementioned characteristics, i.e. refinement of the grain size, increase in the specific surface, opening of grains, etc., with milling in its traditional sense. But, unlike classical milling, certain phenomena occur in HEM/HSM. It is these effects into which some part of consumed energy is transformed and used, mainly: (1) mechanical-chemical activation; (2) production of higher rates of micro- and/or nano-particles; (3) higher efficiency of using consumed energy for the creation of new surfaces.

Mechanical-chemical activation is studied by a branch called mechanical chemistry, which may be considered as an interdisciplinary field of science. With some simplification, it deals with the initiation and enhancement of the efficiency of chemical and physical processes through mechanical effects [4].

3 Assessed Crumb Rubber Modified Binder Variants

The assessment of the different experimental bituminous binders modified by crumb rubber (CRmB) involved an application of pulverized rubber of three grading (granularity) levels. At the same time, the effects of several catalysts were checked; these meant special organic solutions on an anhydrous basis, the varied chemical composition and pH levels. Catalyst K2 is neutral, i.e. with pH=7, catalysts K3 and K4 have a slightly acidic nature with pH=5. All three catalysts are based on a combination of methane $(CH2)_n$ and SO_2 compounds bond in a complex hydrocarbon chain. Simultaneously, an original Czech additive, Polyol was applied; it is a by-product of an innovative chemical recycling method for polyurethane. Last but not least, an additive commercially known as Vestenamer was used which is sufficiently well established from the ROAD+ technology. This additive is a mix of linear and macro-cyclical polymers, chemically termed trans-polyoctenamer (TOR). This was applied together with crumb rubber at a content not exceeding 5 % and then mixed in the bitumen. Variations of experimentally tested crumb rubber modified binders further discussed in this paper are summarized in Tab. 1. The choice for the basic bituminous binder was the regular neat bitumen 50/70 with a defined interval for the softening point as 46-54 °C and the penetration interval of 50-70 dmm.

Tab. 1: Summary of assessed crumb rubber modified bituminous binders.

Bitumen variant	Additives	Pulverized rubber	Bitumen composition
CR-L7_2	-	15 %; 0,8 - 1,0 mm	50/70 + 15 % CR
CR-L7_2_K2 @150	Catalyst K2	15 %; 0,8 - 1,0 mm	50/70 + 15%CR + 5%K2@150 °C
CR-L7_2_K2 @170	Catalyst K2 @170 °C	15 %; 0,8 - 1,0 mm	50/70 + 15%CR + 5 % K2 @170 °C
CR-L7_2_K3 @150	Catalyst K3	15 %; 0,8 - 1,0 mm	50/70 + 15%CR + 5%K3@150 °C
CR-L7_2_K3_P	Catalyst K3 + Polyol	15 %; 0,8 - 1,0 mm	50/70 + 15%CR + 5%K3 + 1%Polyol
CR-L7_2_K4 @150	Catalyst K4	15 %; 0,8 - 1,0 mm	50/70 + 15%CR + 5%K4@150 °C
CR-L7_2_K4 @170	Catalyst K4	15 %; 0,8 - 1,0 mm	50/70 + 15%CR + 5%K4@170 °C
CR-L7_2_K4_P	Catalyst K4 + Polyol	15 %; 0,8 - 1,0 mm	50/70 + 15%CR + 5%K4 + 1%Polyol
CR-L7_2_V	Vestenamer	15 %; 0,8 - 1,0 mm	50/70 + 15%CR + Vestenamer
CR-L8_2_K4 @150	Catalyst K4	15 %; 0,5 - 0,8 mm	50/70 + 15%CR + 5%K4@150 °C
CR-L9_2_K4 @150	Catalyst K4	15 %; 0,1 - 0,3 mm	50/70 + 15%CR + 5%K4@150 °C
CRmB_1	Catalyst K4 + Polyol	15 %; 0,1 - 0,3 mm	50/70 + 15%CR + 5%K4 + 1%Polyol
CRmB_2	Catalyst K4	15 %; 0,1 - 0,3 mm	50/70 + 15%CR + 5%K4
CRmB_3	Catalyst K3	15 %; 0,1 - 0,3 mm	50/70 + 15%CR + 5%K3
CRmB_4	PPA	15 %; 0,1 - 0,3 mm	50/70 + 15%CR + 1%PPA

4 Selected Test Methods

Standard empirical procedures and performance based tests were selected to evaluate the impact of the pulverized rubber and used additives or catalysts. Assessed empirical characteristics are:

- softening point determination, ring and ball method (EN 1427);
- determination of needle penetration under 25 °C (EN 1426);
- determination of elastic recovery under 25 °C (EN 13397);
- storage stability test; 72±1 h and temperature of 180 °C (EN 13399).

Performance based (functional) characteristics:

- determination of the complex shear modulus G* at 60 °C and 40 °C;
- frequency sweep for G* and δ with determination of the master curve for the reference temperature of 20 °C;
- dynamic viscosity determination (EN 13302);
- multiple stress creep recovery test [1,2].

This paper focuses on the analysis of performance based characteristics.

The dynamic viscosity assessment is based on the degree of resistance to the stress caused under the selected angular velocity. Dynamic viscosity is a value of significance for the description of bitumen workability. From this perspective, samples of bituminous binders were compared as a standard under the temperatures of 135 °C and 150 °C with a measurement or conversion for shear rate 6.8 s-1. Flow diagrams were assessed as well. A defined torsion stress was applied to the sample to obtain the relative resistance to spindle revolution. The measurement was taken under various test temperatures. The condition is important primarily with modified bituminous binders or in cases where bituminous binders are improved or modified by various additives. In accordance with the findings and recommendations of the U.S. SHRP program, measurements for distilled binders should be taken for the temperature of 135 °C which is considered a suitable representative for the determination of workability level of the sample in question [3, 10]. The standard stipulates a rotational spindle viscometer as the measuring apparatus and ranges for the shear rate (1-104 s-1) and dynamic viscosity (10-2 – 103 Pa.s) under temperatures ranging from 40 °C to 200 °C. The temperature regulation equipment must be capable of regulation with the precision of ± 0.5 °C.

The assessment of complex shear modulus G* and phase angle δ of bituminous binders using the dynamic shear rheometer (DSR) is governed by technical standard EN 14770. Simultaneously with the measurement of dynamic shear, the viscosity and elastic behavior of the binder can be examined under varying temperatures and frequencies which, together, cover a broad spectrum of possible conditions to which the bituminous binder might be exposed. The determination of G* and δ in the oscillation test is usually carried out for a temperature range of 20-100 °C. A specific stress frequency or a pre-defined frequency spectrum is selected. To obtain relevant results, the linear area of visco-elastic behavior must be defined, i.e. in the regime where the test is conducted with controlled stress; the constant shear stress for the test must be specified. In this study, the previous findings of the CTU in Prague were used. The shear stress of τ=2,000 Pa is considered as safe and appropriate.

Additionally using the time-temperature superposition principle, values obtained from measurements under various temperatures and frequencies may be summarized (transposed) in a single characteristic known as master curve for the selected reference temperatures which, in the case of the results presented below, amounted to 20 °C.

5 Results and Discussion

5.1 Dynamic Viscosity

The dynamic viscosity test with a focus on 20 rpm (the reference velocity considered by the American standards) shows a positive impact of catalyst K4 under both 150 °C and 135 °C. It is obvious that particularly catalyst K3 significantly increases the dynamic viscosity value mainly under 135 °C. The overall course of the viscosity (flow curve) in the thermal range of 100 °C to 150 °C demonstrates the best results for the option with catalyst K4 within the framework of CRmB binder comparison. Comparing the effect of rubber particle size, binders with rubber of 0.1-0.3 mm and 0.8-1.0 mm grading scored better. Even in this case, slightly illogically, the medium grading applied stands out. The overall course of viscosity in the range of 100 °C to 150 °C demonstrates poorer results for the option with 0.5-0.8 mm fraction applied.

If dynamic viscosity is assessed from the viewpoint effect of selected additives, the combination of catalyst K4 with Polyol scores better and also has good results in the entire thermal range. In contrast to the reference binder modified solely by pulverized rubber, the version with catalyst K3 and Polyol recorded the worst course of dynamic viscosity out of all of the versions compared. Nevertheless, TOR also has a negative effect on dynamic viscosity as has been proven within the experimental measurements at CTU also for other options of application of pulverized rubber and this additive.

From the data in Fig. 1 comparing binders CRmB_1 to CRmB_4, it is obvious that the CRmB_4 sample demonstrates higher dynamic viscosity values primarily under the higher temperature. With 135 °C, the highest viscosity was detected for the binder with catalyst K4. This does not quite correspond with the flow curves indicated below where, in case of selected shear rates, the dynamic viscosity value of binder CRmB_4 is always higher. Contrastingly, the sample marked CRmB_3 seems to be the most suitable and catalyst K3 has the most positive effect on dynamic viscosity.

From the point of view of comparing the dynamic viscosity determination, the course over the entire thermal range is important. For the selected shear rate of 1.0 s^{-1} the difference in dynamic viscosity values of the

Tab. 2: Dynamic viscosity of assessed CRmB binders for two temperature levels.

Bitumen variant	Dynamic viscosity @ 6.8 s^{-1} (20 rpm) [Pa.s]	
	135 °C	150 °C
50/70 REF	0.59	0.30
CRmB_1	4.09	1.31
CRmB_2	5.54	1.15
CRmB_3	3.58	1.24
CRmB_4	3.58	2.42
CR-L7_2	4.09	1.54
CR-L7_2_K2 @150	5.54	1.28
CR-L7_2_K3 @150	7.10	1.74
CR-L7_2_K4 @150	3.05	1.00
CR-L8_2_K4 @150	13.75	4.50
CR-L9_2_K4 @150	2.06	1.03
CR-L7_2_K3_P	6.86	1.84
CR-L7_2_K4_P	2.90	1.21
CR-L7_2_V	5.30	3.40

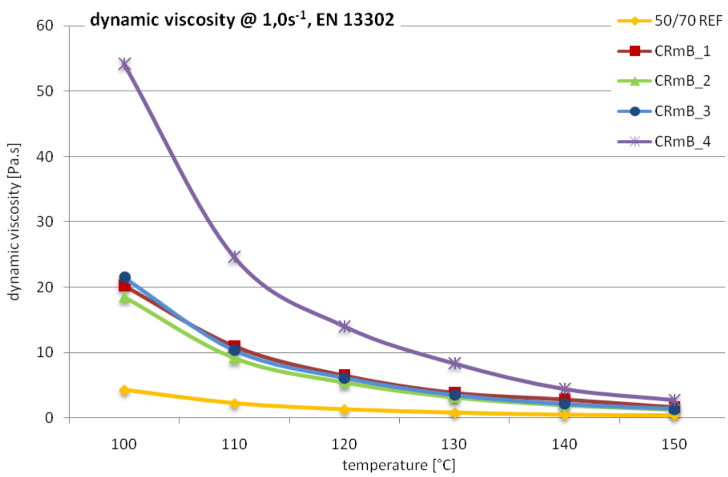

Fig. 1: Viscosity curves for CRmB with pulverized rubber of 0.1-0.3 mm size; shear rate 1.0 s^{-1}.

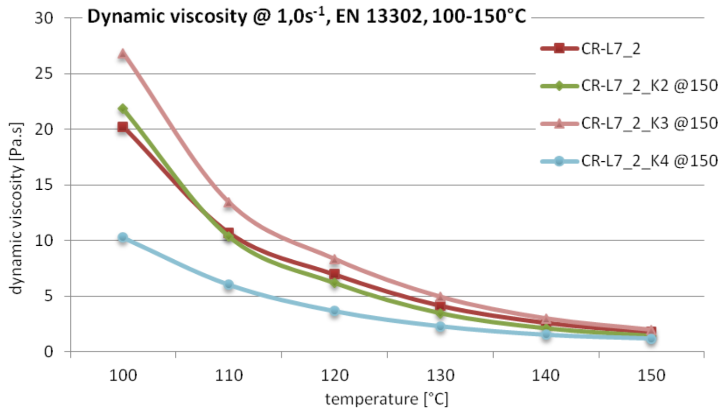

Fig. 2: Viscosity curves for CRmB binders with different catalysts; shear rate 1.0 s^{-1}.

reference binder, CRmB_4 and the remaining versions assessed is quite obvious. This confirms the effect of the phosphoric acid as repetitively detected in the past and it results in higher dynamic viscosity values [7]. CRmB_4 achieves significantly higher viscosity; in contrast to that, the remaining samples have very similar courses as well as individual values. This is not quite supported by the course of the flow curve of binder CRmB_1 under lower shear rate 0.1 s^{-1}. On the differences in dynamic viscosity in the interval of 100-120 °C, the binder CRmB_4 remains within the range of 2-10 fold increase of the characteristic in relation to the reference asphalt 50/70 in case of the higher shear rate.

Comparing assessed catalysts used for improved CRmB stability, particularly a significant difference between the influence of catalysts K3 and K4 is obvious. At 130 °C, the viscosity difference is double-fold, under 100 °C the flow curve values differ even more. From the point of view of the above stated, the influence of catalyst K4 is either similar to low-viscosity additives or helps improve dissolution of the rubber particles in the CRmB binder composite. From the perspective of the course of dynamic viscosity, catalyst K2 appears neutral when compared to the no-catalyst CRmB version which showed no effect whatsoever.

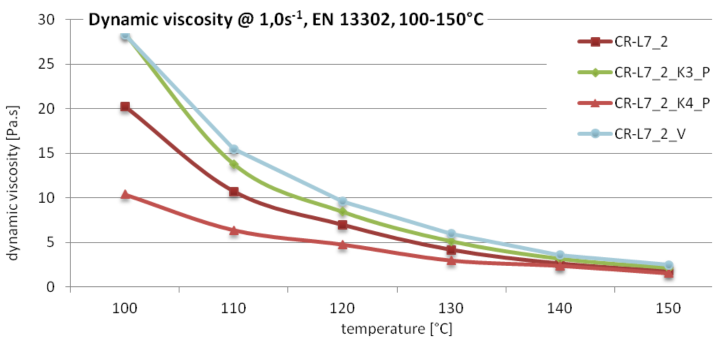

Fig. 3: Viscosity curves for CRmB binders with selected additives; shear rate 1.0 s^{-1}.

The flow curves, if comparing chemical additives show basically identical courses for CRmB binder with catalyst 3 + Polyol and the binder with Vestenamer in relation for the shear slope selected. The two versions have approx. 1.5 times higher values, particularly in the interval of 100-120 °C. The course of the curve for the binder containing catalyst 4 and Polyol is interesting; even under lower temperatures included in the assessment, the dynamic viscosity achieved is quite low when compared to the remaining samples. This can probably be attributed to the influence of the chemical additives applied; in comparison with the CRmB versions from the preceding group, Polyol probably has no influence.

5.2 Dynamic Shear Modulus

Test methods which determine the complex shear modulus G* and phase angle δ using dynamic shear rheometer (DSR) allow performance assessment under medium and higher operation temperatures. The essence of the basic test is a parallel oscillating plate device allowing heating or cooling of the bitumen sample between the plates. Constant gap is always utilized (plates of Ø25.0 mm and gap of 1.0 mm or plates of Ø8.0 mm and gap of 2.0 mm). In the case of the results described below and the transformation of the data measured into a master curve, the measurements were taken for the temperature range of 60-20 °C and frequency range of 0.1-10 Hz for each temperature measured, using the test procedure called the frequency sweep. The data obtained was subsequently used, applying the superposition of time and temperature principle, to generate the master curve for the complex shear modulus and phase angle depending on the frequency as an independent variable. The reference temperature to which the data for all temperatures was related and converted was set to 20 °C.

The transformation of the data measured into the master curve practically provides an illustration of the relationship between stress and deformation by means of the complex shear modulus curve, given for a very broad frequency interval. It primarily describes the resistance to permanent deformation and defines the fatigue behavior of the material. In the first step four experimental CRmB binders were compared to the reference binder and, at the same time, the impact of chemical additives were assessed. The data obtained out the difference between the reference sample, CRmB_4 binder and the remaining three versions of rubber-modified bitumen which have practically identical courses of the master curves. It can be noted that from the point of view of the complex shear modulus, it is not determining whether catalyst 3 or 4 will be preferred; it is even obvious that the effect of Polyol equals zero from the perspective of this characteristic. In the case of the phase shift, it is quite obvious that the application of rubber in the bituminous binder improves the elastic properties within the entire frequency interval, i.e. it increases the elastic component of the bituminous binder. Options CRmB_4 seems the most resistant against fatigue characteristics and permanent deformation; it clearly demonstrates the additional benefit of phosphoric acid which probably causes a change of the chemical structures not only in the bituminous binder but also in the used rubber granulate. It achieves higher values of the complex shear modulus (primarily in the interval of 10-6 to 10-1 Hz) but, in the frequency range of 10-6 to 10-3 Hz, it also demonstrates a higher proportion of the elastic component than the other samples tested. Moreover, in the case of the complex shear modulus, it is obvious that the combination of ground rubber and organic acid reduces the thermal sensitivity of bituminous binders. In the case of the remaining three CRmB binders, this is slightly worse. However, it is obvious that this characteristic will be by far the worst for the reference binder.

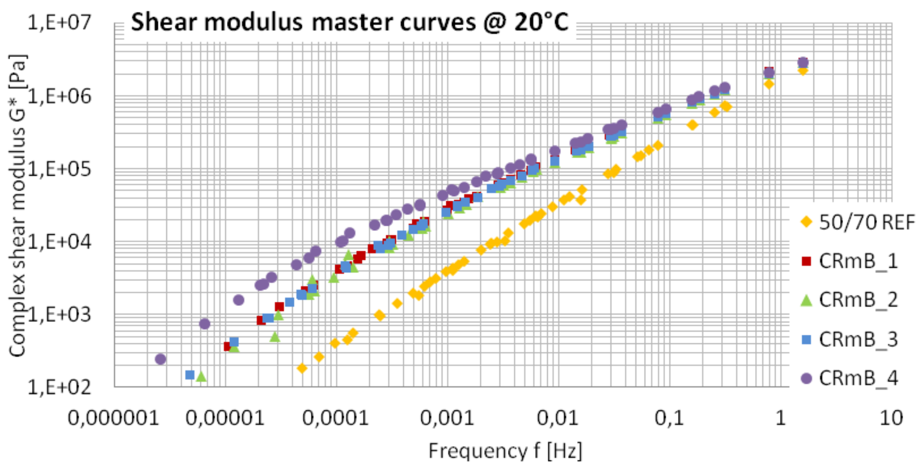

Fig. 4: Master curves for CRmB binders with pulverized rubber of 0.1-0.3 mm size; ref. temperature 20 °C.

The G* values under the key temperatures (40 °C and 60 °C) and frequency of 1.59 Hz are greatly affected primarily when catalyst K3 was applied, where CRmB binder reached noticeably higher values of G* than the bituminous binders versions containing the remaining two catalysts or CRmB with no catalyst applied. In this context, it has to be noted that the three assessed catalysts achieve almost identical results.

The assessment of the master curves for the CRmB binders examined with the determination of the influence of the pulverised rubber grading shows that the best values of the complex shear modulus by far are achieved by CRmB with rubber of 0.5-0.8 mm grading; for small frequencies, the differences are adequate to 1-2 decimal orders. At the same time, it is evident that this version of CRmB scores lowest on thermal susceptibility. Within the framework of the assessment of the remaining two versions, the binders are similar with minimum impact of the grading of the pulverised rubber applied. With respect to the finest and coarsest granulate, it must be assessed why the medium-granularity achieved such different results. In this context, the question is whether this result could have been caused by the effects of the relevant catalyst on the rubber granulate.

Tab. 3: Complex shear modulus values for two temperature levels; f = 1.59 Hz, tau=2,000 Pa.

Bitumen variant	60 °C	40 °C
	Complex shear modulus G* [Pa]	
CR-L7_2	3 309	66 742
CR-L7_2_K2@150	N/A	73 377
CR-L7_2_K3@150	22 926	131 374
CR-L7_2_K4@150	1 480	48 300
CR-L8_2_K4@150	81 200	555 000
CR-L9_2_K4@150	3 400	75 000
CR-L7_2_K3_P	12 292	99 943
CR-L7_2_K4_P	N/A	21 600
CR-L7_2_V	10 512	146 283

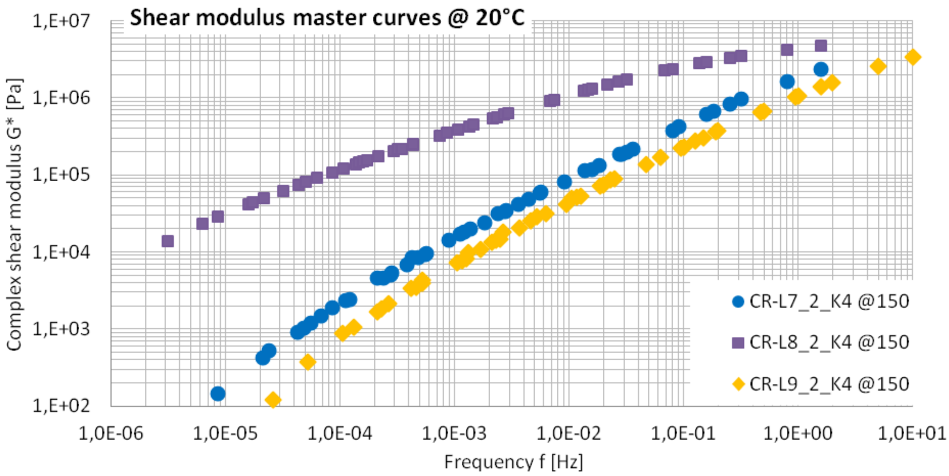

Fig. 5: Master curves for CRmB binders with selected pulverized rubber variants; ref. temperature 20 °C.

The findings of G* assessment for selected temperatures and frequencies are also obvious in the case of the master curves prepared. The CRmB option with catalyst K3 records higher stiffness within the entire frequency interval. The values of all binders assessed subsequently even out in the highest frequency interval, i.e. under 20 °C and the 1-10 Hz spectrum. Based on this CRmB, the binder with catalyst K3 will have the lowest thermal susceptibility value. In contrast to that, the course of the CRmB binder with catalyst K2 values in the smallest frequency interval (from 10-4 Hz) is interesting; the G* values decrease significantly.

If selected chemical additives are compared, the G* master curve has an analogous course for CRmB binders with catalyst K3 + Polyol and with Vestenamer alone. In both cases, higher modulus values are achieved; the binders should demonstrate improved resistance against deformation. This is reflected also in the courses of the phase angle master curve according to which the elastic component prevails in binders within the 10-6 to 10-1 interval. This is a quite important finding for the area of higher temperatures in particular. In the case of the last two binders, it is obvious that CRmB with catalyst K4 + Polyol has the least favorable course from the point of view of both G* and δ; it is likely to demonstrate the highest thermal susceptibility figures.

6 Conclusion

The comparisons conducted and divided on the basis of the possible influences of multiple CRmB binder options reveal certain tendencies in some cases. This means particularly the effect of crumb rubber granularity, where it was rather surprisingly demonstrated that particularly 0.8-1.0 mm is suitable for use with respect to

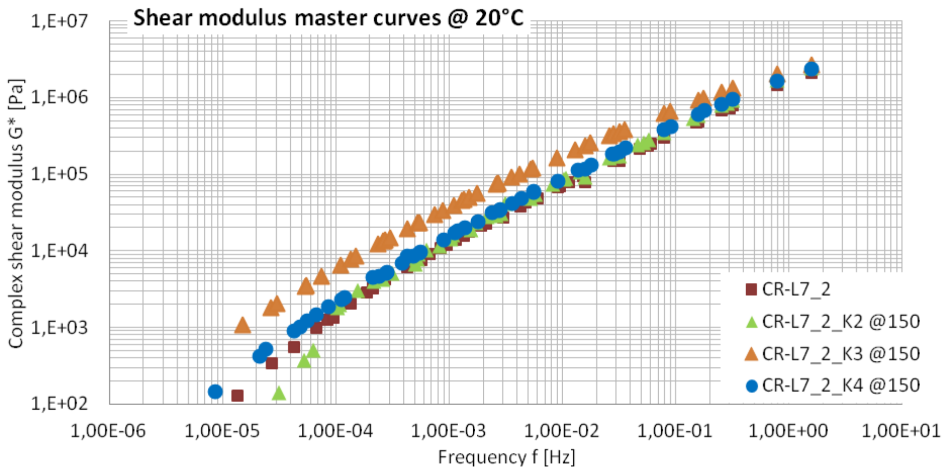

Fig. 6: Master curves for CRmB binders with different catalysts; ref. temperature 20 °C.

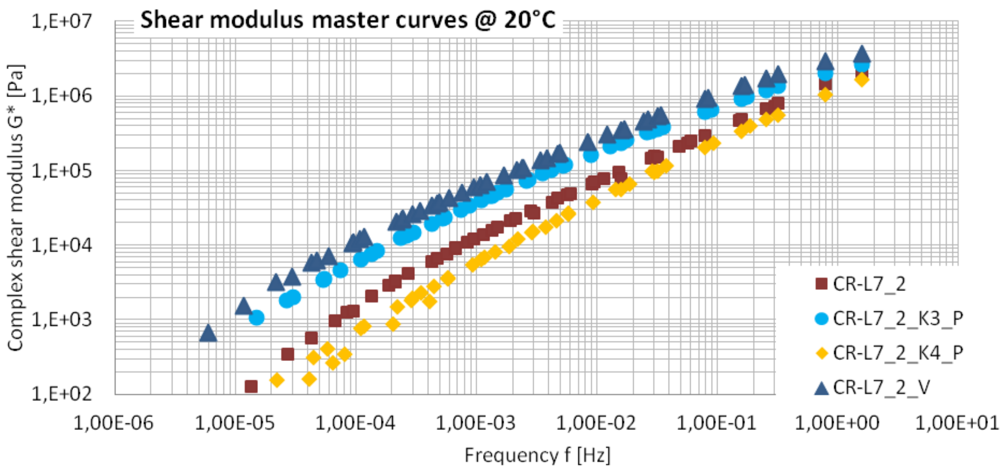

Fig. 7: Master curves for CRmB binders with different chemical additives; ref. temperature 20 °C.

dynamic viscosity. Contrastingly, rheological properties are best affected by the 0.5-0.8 mm granularity, to a lesser degree even by the finer grading of 0.3-0.5 mm. However, in this case the results might be affected by the proportion of the particle size and the gap between oscillation plates in the test apparatus. Due to that, for instance the German technical regulations concerning CRmB binders stipulate a 2 mm gap even for the geometry of PP25 to eliminate any possible influence of not dissolved rubber particles.

The choice of a suitable catalyst is not absolutely clear. Catalyst K4 has a positive effect on viscosity and basic properties of bituminous binders; in contrast to that, catalyst K3 modifies elastic recovery and complex shear modulus. Catalyst K2 appears to be the most appropriate alternative to reduce the difference in softening points after the storage stability test. The effects of additives on selected properties demonstrate that the empirical properties and the course of viscosity are improved primarily by a combination of catalyst K4 with Polyol. The storage stability and the complex shear modulus master curve are positively affected particularly by Vestenamer which, nevertheless, results in increased dynamic viscosity in the CRmB versions assessed according to the measurements taken and thus it has negative impact on workability. Again, the storage stability has been most distinctively affected by catalyst K3, this time in combination with Polyol.

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Rheological and Environmental Evaluation of Reclaimed Asphalt Incorporating a Wax Additive

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Abstract: This study evaluates the rheological properties and environmental impact of reclaimed asphalt binder with wax additive named RH-WMA. The virgin binder was blended with a high percentage of reclaimed asphalt binder obtained from three road sources. The rheological properties were assessed from the viscosity test results and rheological master curve. The results showed that the addition of RH-WMA enables reduction in production temperature. The rheological master curve indicates that the source of reclaimed asphalt affects the rheological behavior. The addition of reclaimed asphalt binder showed improvements in rheological properties at high temperature. However, the addition of RH-WMA improved the low temperature performance of reclaimed binder. The environmental evaluation suggests that RH-WMA could potentially reduce the fuel requirement and Greenhouse Gas emissions. The integration of reclaimed asphalt and wax warm mix additive can be an alternative material for sustainable road construction.

Keywords: Reclaimed Asphalt Binder; Wax Additive; Viscosity; Rheology; Greenhouse Gas Emissions.

1 Introduction

Waste materials produced from construction and maintenance works continuously threatened the environment and public health. Related industries have tried to minimize the environmental effects by developing new technology to recycle the waste material. In the asphalt industry, recycling of secondary material such as aggregate and binder contributes to the decrement of the environmental load, public health and construction cost.

Past research reported that utilizing recycle asphalt for pavement construction could potentially reduce the global warming, energy consumption, water consumption, life cycle cost and hazardous waste by 20, 16, 11, 21 and 11 %, respectively [1]. A hybrid life cycle assessment on asphalt mixtures showed a significant reduction in energy consumption and greenhouse gas emissions with the increasing amount of reclaimed asphalt (RA) pavement added into the mixture [2]. However, increased stiffness of binder due to the increment of RA content requires very high production temperature. The use of warm mix asphalt (WMA) technology to reduce the production temperature has been recently researched particularly with a high amount of RA. WMA additive can reduce the production temperatures between 10 and 38 °C lower than traditional HMA temperatures [3]. This will in turn reduce fuel usage as well as emissions directly related to the fuel usage. The discharge of gaseous pollutants (CO, NOx, and SO_2) and greenhouse gasses (CO_2) will be lowered. In addition, it will result in a reduction of hazardous air pollutants release into the atmosphere and provide a safer working environment for the workers. Based on the World Bank estimation, for each 10 °C drop in the mixture manufacturing temperature, it reduces fuel oil consumption by 1 liter and CO_2 emission by 1 kg per ton [4]. Consequently, utilizing RA and WMA additive will support the effort to develop sustainable pavement production.

The extent of viscosity reduction depends on the types of WMA additive. Wax type of additive showed significant viscosity reduction when incorporated into RA due to the melting point of the wax ([5,6]. The reduction in viscosity decreases the aging of RA binder because the mixture can be produced at a lower temperature. Wax additives such as Sasobit have been commercially used in WMA and incorporated to various percentages

Tab. 1: Properties of virgin PG64 binder.

Properties	Value
Viscosity at 135^{o}C [Pa.s]	0.432
G*/sin δ at 64^{o}C [kPa]	1.529
Failure temperature [^{o}C]	67
Penetration [dmm]	87
Softening point [^{o}C]	46
Flash point [^{o}C]	331
Ductility	$>$100
Specific heat capacity [J/kg/°C]	920

of RA. Mixture performance incorporating Sasobit and RA showed that the WMA additive produced a more uniform mixture [7]. Sengoz [8] compared the strength of reclaimed asphalt with WMA (RA-WMA) produced from different types of WMA additive and concluded that wax additive exhibits the highest strength. On the other hand, mixtures performance containing RA also affects the composition in RA. According to Al-Qadi [9], the source of RA, type of aggregate, type of binder, additives, the level of damage and methods of storing; all affect mixture performance. Another research reported that the rheological properties of recovered RA blends at intermediate to high temperature performance influenced by the RA source, content, aging and test temperature affects the rheological properties [10]. This indicates that to utilize the RA for road construction and maintenance, it needs proper evaluation.

Based on the reported benefits of utilizing RA and WMA, and factors that influence the mixture performance, it is necessary to investigate the rheological properties of RA with wax additive. Environmental evaluation will provide an indication of the sustainability of the materials. This paper presents the rheological and environmental evaluation of RA incorporating a wax additive named RH-WMA.

2 Materials and Methods

2.1 Materials

A PG64 asphalt binder supplied by a local company in Malaysia was used as a base binder. Performance grading, as suggested by the Strategic Highway Research Program (SHRP), was used to evaluate the asphalt binder properties. Tab. 1 shows the basic rheological properties of the virgin base binder.

A wax WMA additive named RH-WMA, developed in China, was used as an additive. RH-WMA is made of polyethylene wax based additive and produced from cross-linked polyethylene. The optimum amount of RH-WMA added to the virgin binder was 3 %.

The RA was obtained by milling process from three local roads at difference state in Malaysia. These roads have been trafficked about 5-7 years. In this paper, the roads named as R1, R2 and R3. The RA aggregate and binder was separated using solvents, and the extracted binder were further recovered via rotary evaporator. The amount of RA binder incorporated with virgin binder was 50 %. Tab. 4 shows the rheological properties of the recovered binder.

2.2 Methods

2.2.1 Sample Preparation

The RAP binder was blended manually with PG64 virgin binder at 140 °C for 5 minutes under controlled temperature. For blending the RAP binder with RH, the temperature increases to 160 °C with 10 minutes blending duration. The blending temperatures of RH-WMA were based on the manufacturer recommendation, while the blending temperature of RA binder is based on previous research [11].

Tab. 2: Mixture properties of AC14 based on PWD specification, retrieved from [14];.

Tab. 3: Mixture properties of AC14 based on PWD specification, retrieved from [14];.

Properties of Mixture	Value	Prescribed ranges by PWD
Optimum binder content [%]	5.0	4-6
Air void [%]	3.90	3-5
VFA [%]	75.60	70-80
VMA [%]	15.37	-
Density	2.38	-
Stability [kN]	20.64	More than 8.00

2.2.2 Viscosity Test

Viscosity test was performed to measure the flow of binder that enables sufficient fluidity during asphalt production in the mixing plants. The rheological properties of the reclaimed asphalt binder at very high temperature were evaluated using rotational viscometer. The viscosity test was carried out at temperatures ranging from 120 to 170°C at 10°C increment using spindle number 21 in accordance with the Asphalt Institute procedure [12].

2.2.3 Frequency Sweeps

Evaluation of rheological properties of the binder from low to high temperature was conducted using the Dynamic Shear Rheometer (DSR). The test was performed in an oscillatory type testing mode via 8 mm diameter plates with a 2 mm testing gap and 25 mm with 1 mm thickness. The appropriate size of the plate was determined based on the stiffness of the specimen. For determining the linear viscoelastic region, amplitude sweeps were carried out at 10 and 40°C on 8 mm and 25 mm plate, respectively. From the amplitude sweep test, the strain of 0.1 % was employed for performing the frequency sweeps from 0.1 to 10 Hz at temperatures ranging from 5 to 65°C.

2.2.4 Aggregate Gradation and Mix Design

Granite and RA aggregate were graded and blended in accordance with the Malaysian Public Works Department (PWD) specifications for AC14 Marshall mix design [13]. The mix specification was adopted from previous research by Hamzah et al. [14] and shown in Tab. **??**. The granite aggregate specific heat capacity at ambient temperature was 790J/kg/°C.

3 Results and Discussion

3.1 Effects of Reclaimed Asphalt Binder on Viscosity

The relationship between viscosity and temperature of RA binder are illustrated in Figs. 1 and 2. In general, the viscosities of the RA binders are different. As indicated in Tab. 4, the R3 is the stiffest binder followed by R1 and R2. This trend is an agreement with the viscosity result. The stiffest binder has a higher viscosity and therefore, requires a higher production or mixing temperature. The addition of RH-WMA decrease the viscosity and mixing temperature (Fig. 2). For example, the viscosity of R3 reduces from 3.14 to 1.89 Pa.s. Addition of RH-WMA decreases the mixing temperature by about 39.8 %. At 120 °C, the addition of RH-WMA reduces the viscosity of R1, R2 and R3 by 31.3, 35.6 and 39.8 %. There is less reduction in viscosity as the temperature increase. The decrement of viscosity is influenced by the source and stiffness of RA binder. The corresponding mixing temperature based on the viscosity is presented in Tab. 4. In this study, the mixing temperature obtained from the graph of viscosity for RA binder with RH-WMA viscosity are further reduced to 20 °C.

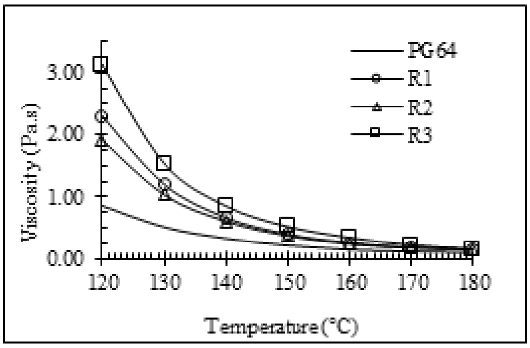


Fig. 1: Viscosity of base binder and reclaimed asphalt binder.

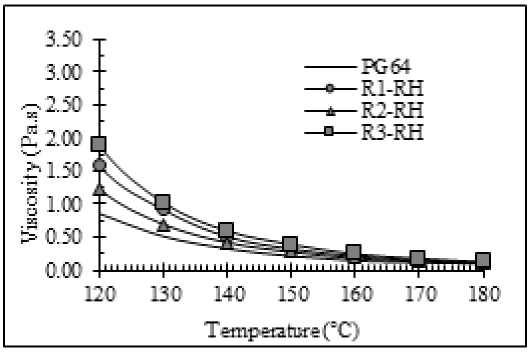


Fig. 2: Viscosity of base binder and reclaimed asphalt binder with RH-WMA.

Tab. 4: Rheological properties and mixing temperature of reclaimed asphalt binder blends.

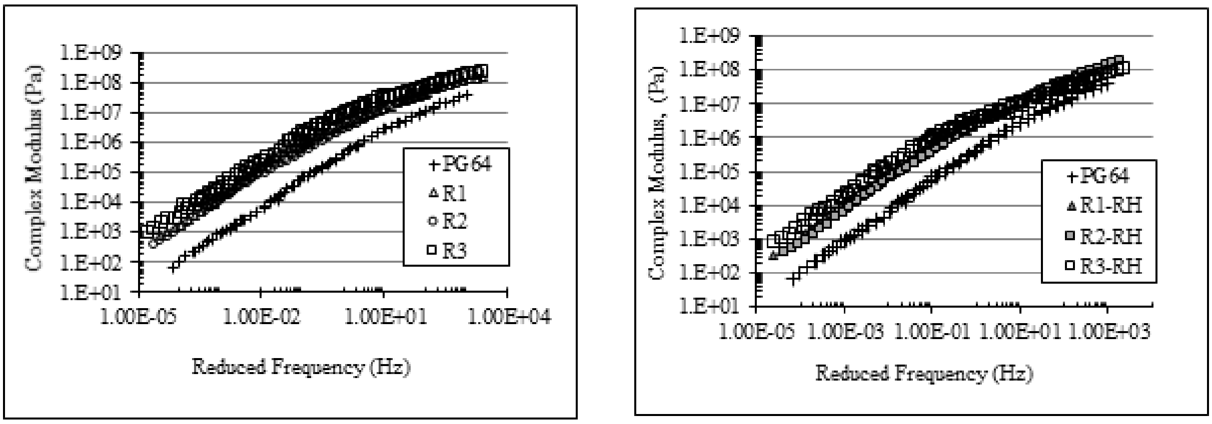
Binder Designation	Penetration at 25 °C [dmm]	Viscosity at 135 °C [Pa.s]	Mixing temperature [°C]
R1	24	0.912	170
R2	32	0.761	168
R3	22	1.112	176
R1-RH	38	0.686	146
R2-RH	43	0.541	142
R3-RH	30	0.762	152

3.2 Rheological Master Curve

A master curve was produced at a reference temperature of 25 °C to describe the rheological behaviour of the binders over a wider range of frequencies. Figs. 3 and 4 present the rheological master curve of complex modulus and phase angle. As shown in Fig. 3, the complex modulus increases with frequency. At lower frequency, there are notable differences among the RA, and it get closer to each other as the frequency increase. Lower frequency represents the binder behaviour at high temperature. At high temperature, the stiffest binder (R3) can better resist rutting compared to R1 and R2. The binders performance at a higher frequency or low temperature are almost similar as indicated by the overlapping curve. The addition of RH-WMA slightly reduced the G* and shown in Fig. 3b. Similar trends of the curve are observed at low frequencies. However, when the frequency increases the curves starts to overlap at G* above 2.E+07. This shows that the source of RA behaves differently due to variations in the RA material composition. The master curve of phase angle shown in Fig. 4 describes the elastic behaviour of the RA binder. The binder from R3 exhibits the lowest phase angle, followed by R1 and R2. The addition of RH-WMA slightly increases the phase angle. The overlap of phase angle master curve of R1-RH and R2-RH illustrates that these binders may have similar elastic behaviour. On the other hand, the differences among the RA binder are more significant in the phase angle master curve plots. The phase angle is more sensitive to the material composition and molecular interaction. Based on the phase angle, none of the binders exceeded the threshold for thermal cracking that is 1.E+09. Therefore, the use of 50 % RA are acceptable based on the rheological master curve.

3.3 Environmental Evaluation Based on GHG Emissions

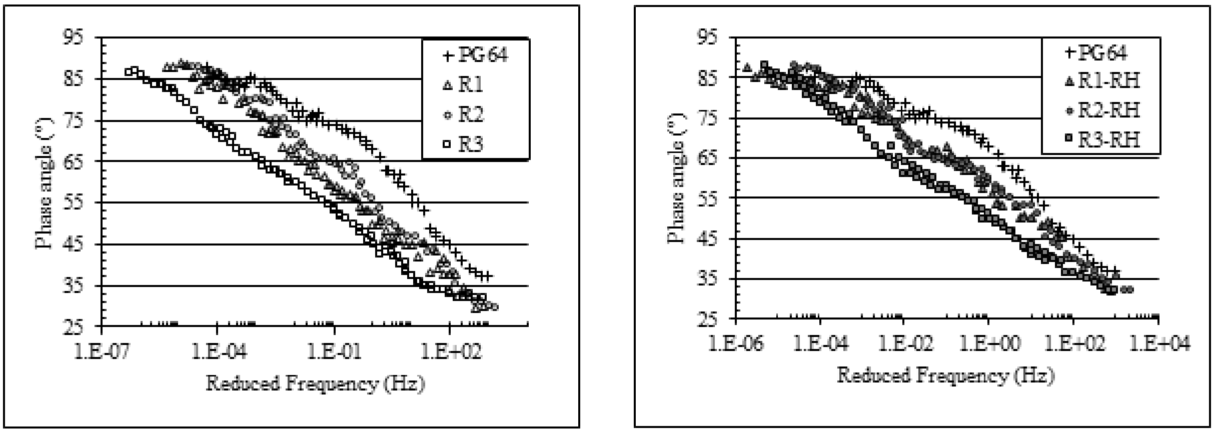
Environmental evaluation is necessary to help the policymaker or engineers to estimate the environmental load for different RA source. The environmental evaluation is measured based on the fuel requirement and Greenhouse Gas(GHG) emissions by adopting the approach proposed by previous researcher [13]. The analysis is carried out by calculating the amount of fuel to heat up the aggregate and binder from ambient temperature up to mixing temperature as specified in Tab. 4. The average ambient temperature of 33 °C is selected to represent the aggregate temperature at the quarry. This environmental evaluation is an indirect measurement



(a) reclaimed asphalt binder

(b) reclaimed asphalt binder with RH-WMA

Fig. 3: Master curve of complex modulus at 25 °C reference temperature.



(a) reclaimed asphalt binder

(b) reclaimed asphalt binder with RH-WMA

Fig. 4: Master curve of phase angle at 25 °C reference temperature.

and calculated using the formula shown in Eq. (**??**).

$$Q = \sum_{i=n}^{j=n+1} mc\Delta\theta \tag{1}$$

where, Q is the sum of required heat energy[J], m is the mass of materials[kg], c is the specific heat capacity coefficient [J/kg/°C], $\Delta\theta$ is the difference between the ambient and mixing temperature [°C], and i and $j$indicate the different types of material. The mix density and other volumetric properties of the mixtures were calculated based on the information given in Tab. **??**. Environmental analysis conducted based on the total material required for a construction of 10-km dual carriageway road with 3 lanes per direction and 5 cm thick wearing course. The type of fuel used in the calculation is diesel. For calculation, the required heat energy is converted to the required fuel and GHG emissions using conversion factor [15, 16]. The results of the analysis are summarised in Tab. 5. From the analysis, addition of 50 % RA increases the fuel requirement and GHG emissions regardless of RA source. There is more increment of fuel requirement and GHG emissions for the stiffer binders. Nevertheless, the addition of RH-WMA reduces the fuel requirement and GHG emissions for R1, R2 and R3 by 11.9, 15.3 and 6.8 % respectively, in comparison with the virgin base binder. The amount of fuel requirement and GHG emissions decrement for each sources of RA due to the addition of RH-WMA for R1, R2 and R3 are 18.9, 20.7 and 18.1 % respectively. Incorporation of RH-WMA reduces the environmental load. Therefore, the integration of RA and RH-WMA can be considered as an alternative for sustainable road construction.

TJ: terra joule; Q_T: required energy to heat the aggregate and asphalt binder; [a]: type of fuel is diesel and conversion factor is based, on DTI [15]; [b]: the direct GHG emission calculated based on the scope 1 in the GHG protocol in DEFRA [16].

Tab. 5: Analysis of fuel requirement and GHG emissions.

Mixture	Q_T	Fuel requirement[a] [ton]	GHG emissions[b]	Increase/decrease in fuel requirement [%]	Increase/decrease in GHG emission [%]
PG64	3.07	67	214425	-	-
R1	3.34	73	232898	+8.6	+8.6
R2	3.28	72	229187	+6.9	+6.9
R3	3.50	76	244078	+13.9	+13.9
R1-RH	2.71	59	188910	-11.9	-11.9
R2-RH	2.60	57	181697	-15.3	-15.3
R3-RH	2.86	62	199794	-6.8	-6.8

4 Conclusion

Rheological properties and environmental impact of binder containing 50 % reclaimed asphalt from three sources of roads and incorporated with RH-WMA were evaluated. The rheological properties evaluation based on viscosity and DSR conclude that the source of reclaimed asphalt binder affects the viscosity. Meanwhile, the addition of RH-WMA reduces the viscosity and production temperature. The frequencies dependency of the reclaimed asphalt binder was assessed by the rheological master curve of complex modulus and phase angle. The binder modification showed improvement in high temperature rheological properties. The addition of RH-WMA slightly enhances the low temperature performance of reclaimed asphalt binders. The master curve plots showed variation amongst the modified binder due to the different source of reclaimed asphalt. Environmental evaluation based on the fuel requirement and GHG emissions indicate that the environmental load increases due to the addition of reclaimed asphalt. Nevertheless, the addition of RH-WMA shows a potential reduction of fuel requirement and GHG emissions. Therefore, the incorporation of reclaimed asphalt and RH-WMA can be an alternative for sustainable pavement construction.

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Effect of F-T Wax on Aging Characteristics of Warm Mix Asphalt Binders

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Abstract: The use of rheology modifying additives in asphalt mixes for reducing the mix production temperature and/or increasing the reliability of the compaction process in order to obtain durable pavements, is state of the art. F-T wax decreases the viscosity of bitumen and imparts lubricity above its melting temperature range, resulting in improved workability and compactibility of the asphalt mixes. The wax is thoroughly mixed with the bitumen which poses the question of influencing physico-chemical properties of the binder, such as aging properties. In order to evaluate the potential effects on aging, three bitumen samples were prepared in the laboratory: a bitumen 50/70 as control sample and the 50/70 mixed with two F-T waxes of different chemical composition. The binders were evaluated in the fresh state as well as short-term aged (by RTFOT) and long-term aged (by PAV) states respectively. Besides effects on mechanical/rheological properties, the main focus was the detection of ageing effects on the chemical and colloidal composition of the binders. For the rheological characterization, complex shear modulus tests were conducted in a temperature range of 20 °C to 90 °C. The compositions of the binders in terms of major chemical groups were evaluated by IR spectroscopy and by performing SARA-analytics using the TLC/FID technique. The proportions of saturates, aromatics, resins and asphaltenes were measured. The compositions of the asphaltenes were further evaluated by separation into three fractions applying a dissolution/precipitation procedure.

The aging did not change the softening points of F-T wax modified bitumen, which are mainly influenced by the melting temperatures of the waxes, but resulted in the expected increase of the binder stiffness. The latter was observed through an increase of complex modulus and decrease in phase angle. This change in physical behavior caused by aging was a result of changed chemical composition: The percentage of aromatic compounds decreased whereas the fractions of resins and asphaltenes increased. Also the chemical composition of the asphaltenes changed towards the fraction of low solubility. The aging processes of the bitumen were only slightly influenced by the added F-T waxes, whilst one of the two wax types even yielded some minor advantages. These differences between the relative performances of the two wax types can be ascribed to chemical differences between them.

The observed influences of the F-T waxes on the chemical composition are minor compared to the variety found for bitumens from different crude origins. The results show that the chemical composition as well as the aging properties are predominantly affected by the bitumen characteristics, whereas effects of F-T wax do not compromise the aging properties of the binder.

Keywords: FT-Wax; Bitumen Ageing; TLC/FID; Infrared Spectroscopy; SARA Analysis; Chemical Characteristics.

1 Introduction

By adding Fischer-Tropsch-wax (FT-wax) to bitumen, its thermo-rheological properties can be modified, resulting in reduced viscosities at asphalt mixing- and compaction temperatures. This allows for the reduction of mixing- and compaction temperatures and therefore improves the materials CO_2-footpint and/or prolongs the time available for compaction of the asphalt course in order to improve the reliability of high-quality asphalt road laying.

However, the actual material performance of asphalt pavements changes due to environmental impacts, e.g. weathering and ageing. The latter occurs as short-term ageing during asphalt mix production by distillative and oxidative effects at elevated temperatures and as long-term ageing during service life because of oxidation. Both short-term as well as long-term ageing affect the rheological properties of the bitumen as well as the durability of the asphalt course.

The physical characteristics of a bituminous binder is directly associated with its chemical composition. However, the chemical nature of bitumen is a very complex system of a multitude of different hydrocarbon molecules [3]. A simple way for describing the chemical characteristics of bitumen by colloidal models is the fractionation into chemical families of different polarity. The model of a colloidal system allows the explanation of the thermo-rheological properties of bitumen and is based on the theory that colloids with high polarity (asphaltenes) are peptized to micelles in an oily phase with lower polarity, called maltenes [8].

Based on this bitumen model the thermo-rheological properties can be explained by two different types of materials: Sol and Gel. Sol material can be interpreted as Newton fluent, in which asphaltenes are fully dispersed in the maltene phase which results in non-elastic behavior [7]. Gel material shows non-Newton behavior caused by interaction between asphaltenes micelles. Most bitumen types show properties between these two extremes [6].

Furthermore bitumen ageing will affect the colloidal system and provoke a change from SOL type to GEL type [7].

2 Experimental

2.1 Materials and Aging Procedure

The investigation was performed using 50/70 penetration bitumen and two Fischer-Tropsch (F-T) waxes. Wax 1 is an unhydrotreated F-T wax, i.e. contains besides hydrocarbons also smaller amounts of alcohols and olefins and is characterized by a congealing point of 101.0 °C. Wax 2 is hydrotreated, i.e. consists only of hydrocarbons and has a slightly higher congealing point of 102.5 °C.

The bitumen (further referred to as 50/70) was modified by the addition of 3 wt.% of F-T wax 1 and F-T wax 2 respectively to the heated (160 °C) bitumen and homogenized by stirring for 10 minutes. The resulting wax-modified bitumen samples are labelled as F-T 1 and F-T 2 in this study.

Simulated aging was carried out in two steps applying the standardized procedures for short term aging by RTFOT (Rotating Thin Film Oven Test, EN 12607-1) and long term aging by PAV (Pressure Aging Vessel, EN 14769).

2.2 Softening Point Ring an Ball

The softening points ring and ball were measured according to the standard EN 1427.

2.3 DSR Complex Modulus Test

The rheological properties of fresh and aged binders were characterized by Dynamic Shear Rheology, using plate-plate tests according to EN 14770. For temperatures between 20 °C and 90 °C as well as frequencies between 0.1 to 10 Hz the shear moduli and phase angels were measured.

2.4 SARA- Analysis by TLC/FID

Four chemical fractions of different polarity (Saturates, Aromatics, Resins and Asphaltenes - SARA components), were determined by thin layer chromatography with flame ionization detection (TLC/FID) according to IP 469. For the analysis 0.1 g of the bitumen sample was dissolved in 5 ml of dichloromethane, resulting in a 2 wt. % solution. 1 μl of this solution is chromatographically separated on a silica adsorbent (Chromarod) using three different solvents successively, see Tab. 1. The chromatogram on the Chromarod was scanned using FID-technique and the proportions of the four bitumen SARA-fractions were determined quantitatively by area determination under the specific peaks. For each sample, five chromatograms were evaluated. The resulting chromatograms for the unaged binder samples are plotted in Fig. 1. The four visible peaks represent the four SARA-fractions. For the F-T-modified bitumen samples, there are two asphaltene peaks detectable. The first

Tab. 1: Fraction depending on order of solvents.

Solvents applied for TLC-separation	Fraction
Heptane	Saturates
Toluene/Heptane (80:20)	Aromatics
Dichlormethane/Methanol (95:5)	Resins
Not eluated	Asphaltenes

peak at the beginning of the Chromarod identifies a portion of non-soluble (considering the solvents applied for chromatographic separation) bitumen compounds in the binder for the temperature, in which the chromatography was conducted (20±3 °C). In order to evaluate this difference to unmodified binder, the asphaltenes are separately described by a 1^{st} and a 2^{nd} asphaltene peak area.

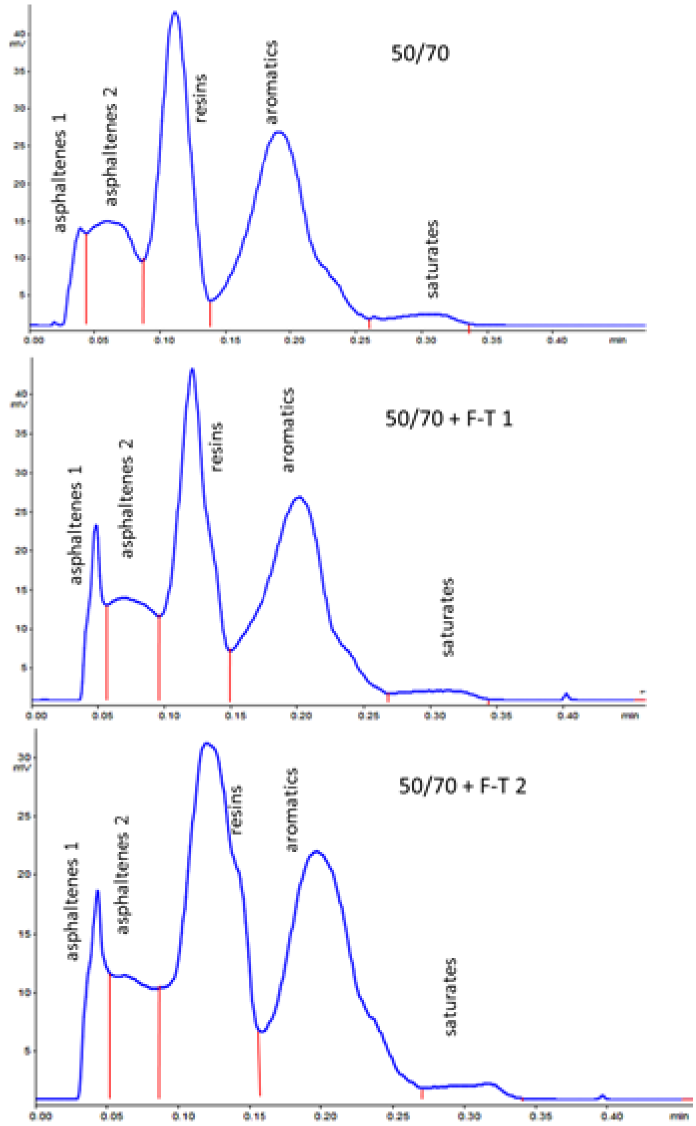

Fig. 1: Chromatogram for SARA components of 50/70 (unmodified and modified).

2.5 Compositions of Asphaltenes

The compositions of asphaltenes in term of low, medium and high solubility are measured by a dissolution/precipitation procedure with three different solvent combinations of iso-octane and cyclohexane as described

Tab. 2: Fractions depending on order of solvents.

Asphaltene solubility	Solvent	Solubility parameter δ $[MJ/m^3]^{1/2}$
High solubility	Iso-octane	14,0
Medium solubility	Iso-octane/cyclohexane (4:1)	14,8
Low solubility	Iso-octane/cyclohexane (1:1)	15,7

by Zenke [10], compare Tab. 2. Each solvent applied can be described with a solubility parameter, compare Fig. 2 [10]. This allows the further distinguishing of the asphaltenes.

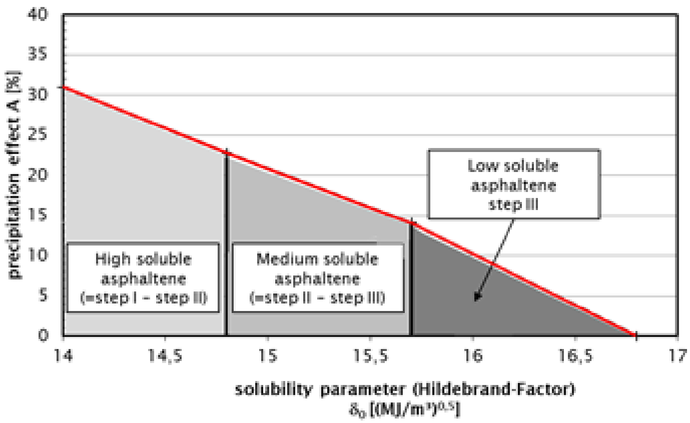

Fig. 2: Explanation to three asphaltene fractions in bitumen.

Asphaltenes are distinguished according to the method of identification as well as the applied type of solvent [9]. Whereas for the asphaltene composition experiment, asphaltenes are defined as the insoluble fraction in iso-octane, in SARA-analytics dichloromethane is applied for defining the asphaltene content. Therefore, it is taken into account that the three compositions of asphaltenes are not comparable to the asphaltene content measured by TLC/FID.

2.6 Infrared Spectroscopy

FTIR spectra of the binders were determined by two different procedures. Firstly, the transmission technique was applied by measuring the IR-spectra of the dissolved bitumen samples as 5 wt. % solution in CCl_4 and in 0.5 mm layer thickness.

Secondly the attenuated total reflection (ATR) procedure was applied directly on the pure, unsolved bitumen samples.

For the assessment of the ageing characteristics of the bitumen samples, the peaks in the spectra at a wavenumber of 1700 cm^{-1} and 1030 cm^{-1} were analyzed, representing carbonyl (C=O) groups and sulfoxides in the samples. These groups are formed due to oxidative ageing of the bitumen [1,3].

3 Results

3.1 Experimental Campaign

The results discussed in the following section were obtained during an experimental campaign conducted in the author's laboratories. The bitumen samples were prepared by SASOL's wax laboratories and shipped to UNI KASSEL laboratories in metal cans. The bitumen samples were re-heated once and separated into the required sub-samples which were used for preparing the needed specimens. By this, additional thermal loads were reduced to a minimum of impact.

Tab. 3: Results of softening point ring and ball tests.

Aging stage	Softening point ring and ball [°C]		
	50/70	F-T 1	F-T 2
Unaged	51.8	89.0	92.5
Short-term	57.6	88.0	90.0
Long-term	67.2	91.0	91.5

3.2 Results of Softening Point Ring and Ball Tests

The measured softening points are given in Tab. 3. The addition of F-T wax 1 increases the softening point of the bitumen 50/70 by 37.2 °C, F-T wax 2 even by 40.7 °C.

The ageing caused an increase of the softening point of the neat bitumen by 5.8 °C due to RTFOT and additionally by 9.6 °C due to PAV to a final value of 67.2 °C. In contrast, the softening points of the F-T wax modified binders stayed nearly constant at about 90 °C despite the applied aging procedures.

3.3 Results of DSR Complex Modulus Tests

In Tab. 4 and Tab. 5 the shear moduli and phase angles obtained at temperatures of 30 °C, 60 °C and 90 °C at a frequency of 1.59 Hz are summarized.

The complex shear moduli measured at the frequencies 0.1 Hz, 1.59 Hz and 10 Hz are plotted versus the temperature in Fig. 3. The corresponding phase angles are plotted in Fig. 4.

The addition of either wax product increases the shear modulus of the bitumen in the unaged stage considerably. This increase is most obvious for the temperature of 60 °C for which the shear modulus is increase by a factor of about 17. The difference at 90 °C (factor of roughly 10) and also at 30 °C (factor of less than 4) is lower than this. Here the viscosity-changing effect of wax addition can be clearly demonstrated. Especially for the temperatures where asphalt mixtures show sensitivity regarding rutting, the shear modulus is increased considerably, whereas the viscosity effect at both lower and higher temperatures are smaller.

For all bitumen samples a stiffening effect due to aging can be observed. For the straight bitumen 50/70, short-term aging by RTFOT results in57 % higher shear modulus at 30 °C testing temperature, in 117 % increase at 60 °C and 88 % increase at 90 °C. After PAV long-term aging, the shear modulus increases further. The increase is highest at the highest test temperature (90 °C) and results in a shear modulus of more than five times higher compared to the unaged samples.

For the wax-modified samples, the aging simulation has less effect on shear modulus results. At a test temperature of 30 °C, the shear modulus is increased only slightly whereas at 60 °C and 90 °C the value is even reduced after RTFOT. Long-term aging results in a shear modulus which is slightly higher compared to the values obtained for the unaged samples.

For the phase angle results as indicated in TABLE V. and plotted in Fig. 4, the addition of wax modifier to bitumen results in a decrease of the phase angle for all test temperatures which can be interpreted as a shift towards more elastic and less viscous material performance. For the unaged samples, the wax modification effect on the phase angles depends on the loading frequency. At high frequency of 10 Hz the increase in temperature results in an increase of phase angle as also observed for the unmodified bitumen (Fig. 4, bottom).

However, at a low frequency of 0.1 Hz the phase angles of the two unaged wax modified bitumen decreases with increasing temperature. For the medium frequency of 1.59 Hz, the phase angle indicates only small changes with increasing temperature. An explanation for this effect can be found in the structural properties of the wax-modified samples. At high frequency, the bitumen predominates the overall rheological properties in of the wax-modified binder. With lower frequency, more time is available allowing internal flows of the bitumen besides or around the wax structure. Therefore, the wax predominates the type of rheological reaction which results in a phase angle decrease with decreasing bitumen viscosity provoked by increase of temperature. At temperatures between 70 C and 80 C, the phase angle plots indicate bends and changing temperature-dependency. This can indicate the beginning melting of the wax, compare [2].

The aging of the straight bitumen sample results in a significant decrease of the phase angle at all test temperatures. For the wax modified samples this short-term aging effect can only be observed for low temperatures

Tab. 4: Results of DSR tests: Complex shear modulus |G*| obtained atthe temperatures 30 °C, 60 °C and 90 °C and a frequency of 1.59 Hz.

T [°C]	Aging state	Shear modulus G* (1.59 Hz) [Pa]		
		50/70	F-T 1	F-T 2
30	Unaged	608 352	2 094 287	2 134 912
	Short-term	953 420	2 547 040	2 377 696
	Long-term	2 035 684	3 357 754	3 192 656
60	Unaged	4 898	87 304	87 802
	Short-term	10 641	86 655	82 871
	Long-term	38 524	122 268	116 134
90	Unaged	176	2 227	1 889
	Short-term	330	1 980	1 709
	Long-term	1 065	2 333	2 097

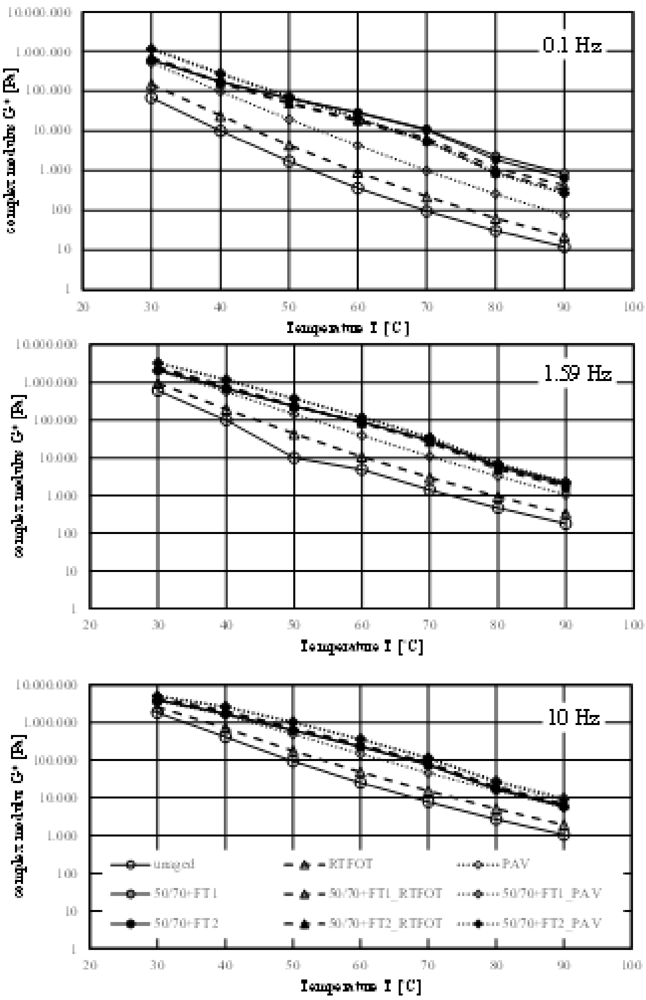

Fig. 3: Complex modulus versus temperature measured at 0.1 Hz (top), 1.59 Hz (middle) and 10 Hz (bottom).

Tab. 5: Results of DSR tests: Phase angle δ obtained at the temperatures 30 °C, 60 °C and 90 °C and a frequency of 1.59 Hz.

T [°C]	Aging state	Phase angle δ (1.59 Hz) [°]		
		50/70	F-T 1	F-T 2
30	Unaged	65,4	45,8	45,7
	Short-term	58,9	45,8	47,9
	Long-term	45,1	39,9	40,3
60	Unaged	81,9	45,0	44,9
	Short-term	76,9	59,0	54,1
	Long-term	68,0	56,2	55,7
90	Unaged	89,0	44,1	47,2
	Short-term	87,2	58,1	60,7
	Long-term	83,3	71,7	68,8

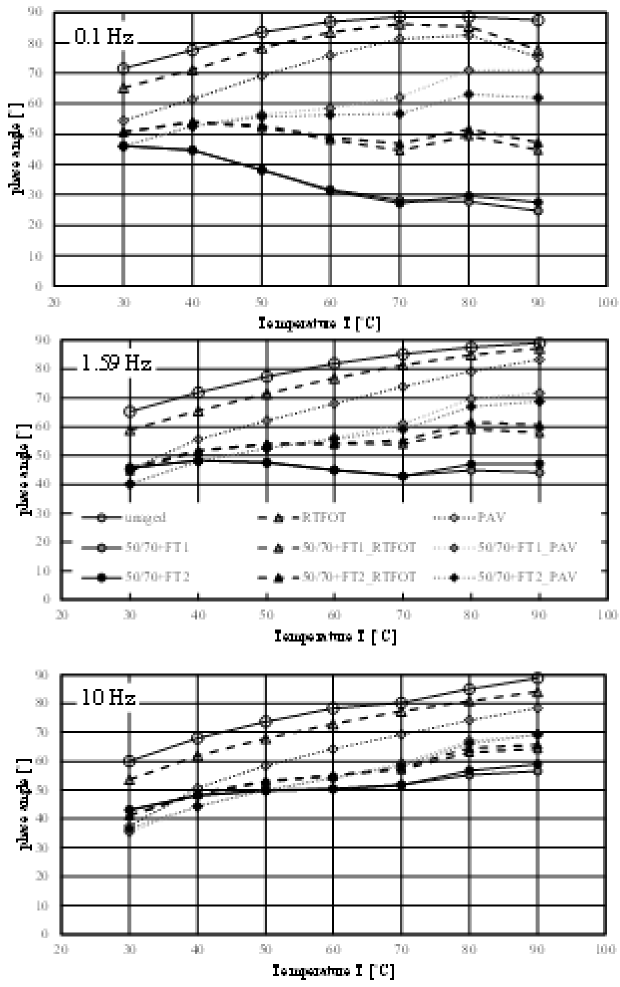

Fig. 4: Phase angle versus temperature measured at 0.1 Hz (top), 1.59 Hz (middle) and 10 Hz (bottom).

and high frequencies. At higher frequencies and especially at all temperatures at the lowest applied frequency of 0.1 Hz, the ageing results in an increase of phase angle. This indicates a shift of the material performance towards viscous properties. Therefore, the difference in the phase angles between unmodified and wax modified samples is reduced with proceeding ageing.

Comparing the phase angles of the bitumen samples modified with different F-T-waxes, it can be observed, that for unaged and RTFOT-aged conditions similar results are obtained. However, for the PAV-aged samples, F-T1 indicates a higher increase in phase angle compared to F-T2.

From the shear modulus G* and phase angle obtained at each temperature and frequency (0.1 Hz, 1.0 Hz, 1.59 Hz, 10 Hz), storage and loss moduli were calculated. The storage modulus indicates the elastic material properties, whereas the loss modulus can be interpreted as the ability of the material to dissipate energy into viscous motion. For the results obtained on the unmodified bitumen 50/70, the Cole-Cole plots are displayed in Fig. 5. The aging results in a shift of the Cole-Cole plots towards lower loss moduli and higher storage moduli.

The measured values can be fitted by a potential function as indicated in Eq. (**??**).

$$G'' = aG'b \tag{1}$$

where a and b are regression factors.

This rather simplified approach for the interpretation of Cole-Cole plots allows the visual comparison of the aging effect of wax-modified samples in the viscoelastic domain.

Therefore, Fig. 6 shows the aging effect in the Cole-Cole plot. For the samples of 50/70, the aging results as already described in a shift towards reduced viscous and increased elastic stiffness properties.

The diametric other effect can be observed for the wax-modified samples. Here, the aging results in a considerable upward shift of the curves in the Cole-Cole plot. It can be assumed that prolonged long-term aging results in similar visco-elastic properties of the straight and the wax modified bitumens. These trends can also be observed for the phase angles results.

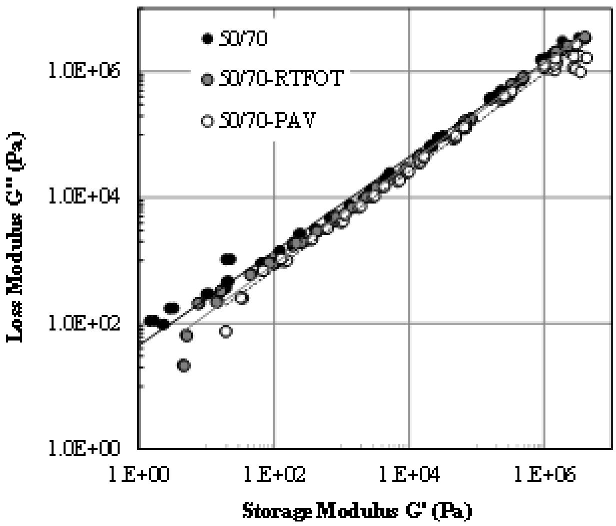

Fig. 5: Cole-Cole-plot of the DSR-temperature-frequency sweep tests obtained on the unaged, short- and long-term aged samples of the straight bitumen 50/70 – addition of fitting curves.

3.4 Results of SARA- Analysis

The proportions of the bitumen SARA fractions calculated from the chromatograms obtained from TLC/FID tests are plotted in Fig. 6. When comparing the SARA fractions of the three unaged binders, the F-T wax modified binders indicate increased contents of resins and asphaltenes as well as decreased contents of aromatic and saturates fractions compared to the neat bitumen. Further the asphaltene peak shows significantly different peak shapes, compare Fig. 1. This results in higher area proportions for the 1st asphaltene peak as indicated in Fig. 6 in the increasing magnitude of this first column. Short term ageing results in an increase of asphaltenes and in a decrease of saturates for all bitumen samples. For unmodified binder and F-T 1 an increase of resins

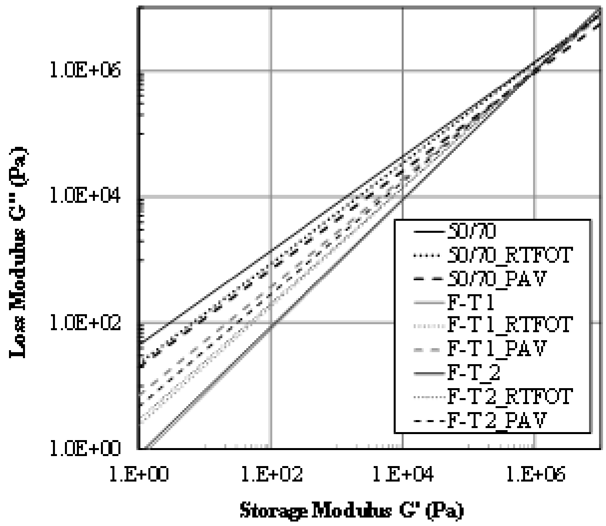

Fig. 6: Regressing curves of the straight and wax modified bitumen samples at varied aging stages in the Cole-Cole-plot.

and a decrease of aromatics were recognized over the ageing stages. For F-T 2 less ageing effects for resins and aromatics are identified. It is noticeable that the asphaltene content after PAV ageing decreases.

Table VI presents the average of the SARA components. These values are plotted in Fig. 7, in which also the scatter within the five repetitions are given.

All bitumen types show an increase of resins and a decrease of aromatics and asphaltene content after PAV ageing. The contents of saturates do not show significant changes.

Both wax modifiers have no influence on the SARA composition in unaged bitumen samples. After PAV ageing F-T 2 shows a lower increase of resins and a lower decrease of aromatics compared to 50/70 and F-T 1.

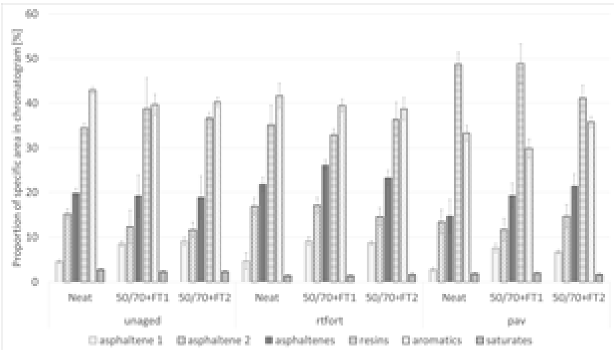

Fig. 7: SARA components of unaged and aged binders.

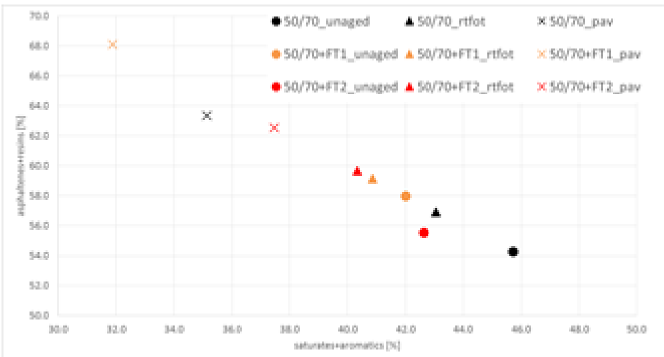

Fig. 8: Grouping of SARA fractions.

Tab. 6: Results of SARA analysis: asphaltenes 1, asphaltenes 2, asphaltenes (total), resins, aromatics and saturates.

sample	SARA components	unaged	rtfot	pav
50/70	Asphaltenes 1	4.60	4.69	2.80
	Asphaltenes 2	15.23	16.90	13.43
	Asphaltenes (total)	19.82	21.79	14.71
	Resins	34.46	35.12	48.63
	Aromatics	42.89	41.67	33.28
	Saturates	2.83	1.41	1.86
F-T 1	Asphaltenes 1	8.57	9.13	7.58
	Asphaltenes 2	12.29	17.08	11.70
	Asphaltenes (total)	19.23	26.21	19.28
	Resins	38.78	32.94	48.84
	Aromatics	39.64	39.46	29.87
	Saturates	2.36	1.39	2.01
F-T 2	Asphaltenes 1	9.05	8.72	6.73
	Asphaltenes 2	11.68	14.57	14.66
	Asphaltenes (total)	18.93	23.29	21.40
	Resins	36.65	36.38	41.13
	Aromatics	40.27	38.66	35.78
	Saturates	2.35	1.68	1.70

In Fig. 8 the fractions with highest polarity (asphaltenes + resins) are plotted versus the fractions with lowest polarity (aromatics+saturates). This graph visualises the effect of increasing polarity by increasing resin content and decreasing aromatic content during ageing. Here the effect of lower influence of PAV ageing for F-T 2 becomes more obvious.

3.5 Results of Asphaltene Composition Analysis

The results of the precipitation experiments for separating three types of asphaltenes, total asphaltene and maltene content of all unaged and aged binders are presented in Fig. 9.

All bitumen samples show in the unaged stage a maximum of medium soluble asphaltenes.

As expected the total maltene contents decrease and the total asphaltene contents increase after ageing. However, the increase of asphaltenes for F-T 2 is less severe compared to F-T1 as well as the neat bitumen.

With regards to the proportions of the three types of asphaltenes, a continuous increase of low soluble asphaltenes and a decrease of medium and high soluble asphaltenes can be recognized during ageing for the unmodified binders. The plots of solubility profiles as shown in Fig. 10 allow a further interpretation of the results. The vertical position of the lines indicate that the asphaltene composition is stronger affected by the ageing affects compared to the effects of wax modification. For wax modified binder F-T 1 only a small change in low soluble asphaltenes (solubility parameter of 15.7) content after RTFOT aging is recognized. After PAV ageing a significant increase of these asphaltenes is noticeable. The contents of medium and high soluble asphaltenes are equal. After PAV aging a significant increase of the low soluble asphaltenes and a decrease of medium and high soluble asphaltenes to the same range are noticeable.

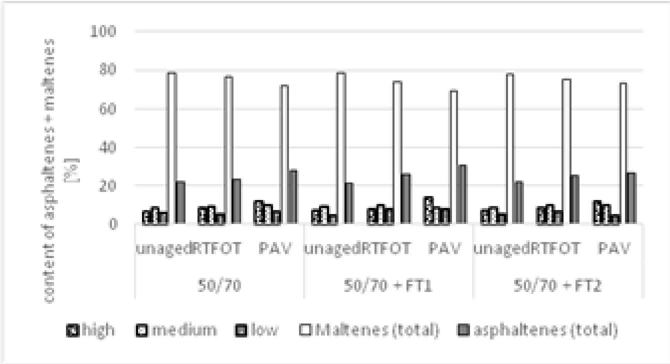

Fig. 9: Content of low, medium, high soluble asphaltenes, total asphaltenes and total maltene content.

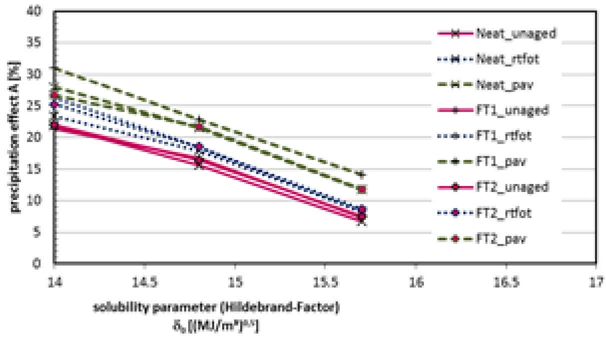

Fig. 10: Profile of solubility and precipitation limits.

3.6 Results of IR Spectroscopy

Infrared spectroscopy by transmission and ATR techniques (Fig. 11 and Fig. 12) were carried out to observe the oxidation progress. The IR spectra of the unaged and aged binders differed mainly in the wavenumber range around 1700 cm^{-1} and 1030 cm^{-1}. Absorptions in the range of 1700 cm^{-1} are caused by carbonyl groups (C=O) whereas the peaks at 1030 cm^{-1} indicate sulfoxides, which result from oxidation [4]. Fig. 11 shows a detail of the IR spectra obtained from the transmission experiments for the C=O peak range. The same range is plotted in Fig. 12 for the ATR experiments. With both techniques similar effects were identified: The unaged binders show high transmission values (Fig. 11) and low extinction results (Fig. 12). After RTFOT, the absorption in the C=O-range was slightly intensified. The long-term ageing by PAV results in significant peaks in the two spectrograms for the C=O.

In order to quantify the extent of oxidation, the area of the C=O peaks were integrated. In Fig. 13 the C=O peak areas as evaluated from the transmission and ATR experiments for the nine binder samples are shown. Whereas the transmission experiment identifies a clear increase of the C=O peak areas due to RTFOR ageing, this increase is in the ATR experiment only in the range of test scatter. However, both types of IR test setup identify significant increases of the C=O peak area for the PAV aged samples. The presence of F-T wax has no significant effect on the oxidation during short term aging but indicates a tendency of reducing the oxidation after long term aging, especially F-T wax 2.

Fig. 14 shows the results from ATR experiments for the wave number 1030 cm^{-1}, identifying the presence of sulfoxides. Similar effects can be observed as for the C=O ageing peak. Whereas RTFOT has a marginal ageing effect, significantly higher peaks can be observed after PAV. Again, the ageing effect in F-T 2 is less intense compared to F-T 1 and neat bitumen 50/70.

Furthermore, the FTIR experiments allow the identification of wax-modification in the bitumen samples. As described in [1] the IR-signal at a wave number of 720 cm^{-1} can be used “as an indication of amorphous and/or crystalline structures in the binder due to wax content” for F-T waxes (Fig. 15). It can be observed, that the ageing has no effect on the peak at wave number around 720 cm^{-1} and that the extinction is constant for all bituminous binders so that the difference can be identified as wax components.

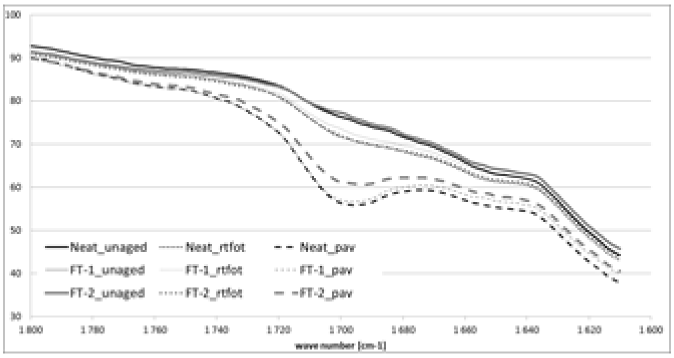

Fig. 11: FTIR spectra (detail in 1700cm^{-1} range) of unaged and aged binders in transmission technique.

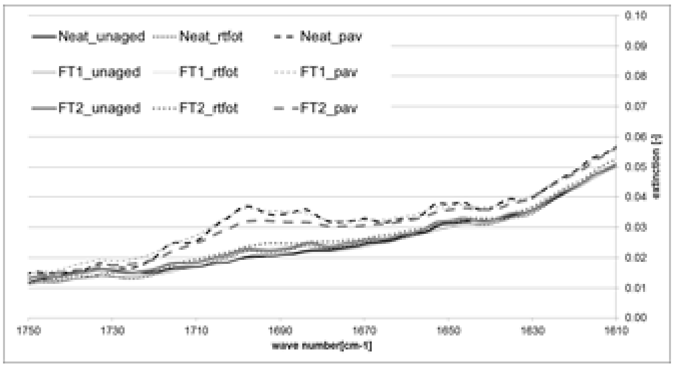

Fig. 12: FTIR spectra (detail in 1700cm^{-1} range) of unaged and aged binders in ATR technique.

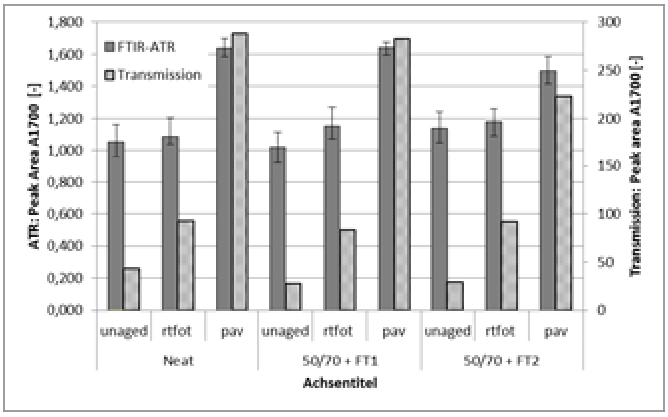

Fig. 13: Peak areas of FTIR spectra in the carbonyl range (1720 – 1670 cm^{-1}) at different aging stages.

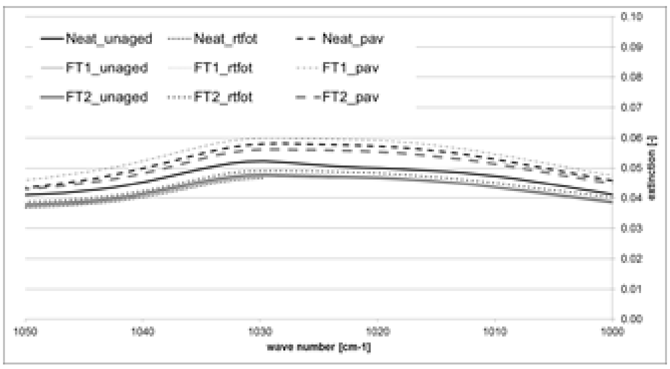

Fig. 14: FTIR spectra (detail in 1030 cm^{-1}) of unaged and aged binders in ATR technique.

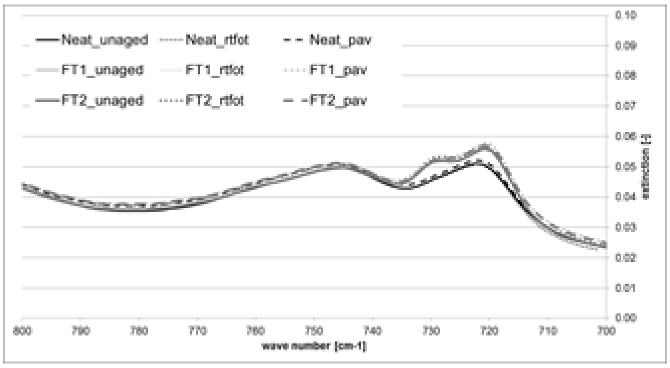

Fig. 15: Identifying wax modifiers in bitumen.

3.7 Interpretation of Test Results

As expected and described in [1], wax modification in bitumen increases the binder stiffness as observed in increasing softening point ring and ball and in increasing shear modulus in a temperature range from 30 °C to 90 °C for the unaged binders. The decreasing phase angle shows an increase of elasticity caused by wax modifiers especially in a temperature range from 50 °C to 70 °C. Within this temperature range, the stiffness of the wax network formed within the binder is higher than the stiffness of the neat bitumen and therefore the wax properties predominate the phase angle of the resulting binder. At lower temperatures the high bitumen viscosity as well as at higher temperatures the beginning of wax melting will result in increased phase angles. Loads above the linear viscoelastic range, which more realistically indicate the deformation resistance, were investigated by two research groups applying the MSCR technique [11, 12]. It was found that 2 % F-T wax significantly increased the percentage of recovery and significantly decreased the irrecoverable compliance of unaged bitumen.

Regarding the chemical properties of the unaged binder samples, the modification can be identified in the FTIR-analysis as well as by a significantly changed asphaltene peak shape in the TLC/FID. The high peak at the sample spotting point of the Chromarods indicates highly insoluble compounds in the wax-modified binders as the F-T wax is almost insoluble at the applied temperature. However, besides of these items, no significant differences were identified between the wax-modified and the neat binder. F-T 1 and F-T 2 have the same effect on the SARA fractions in the unaged binder independently of their individual chemical structure.

Fig. **??** compares various neat bitumen 50/70 samples from different sources in their components of asphaltenes. A, B, C constitute 50/70 of one supplier, D, E, F represent 50/70 of additional different suppliers. The figures show that all 50/70 do not have the same chemical compositions in spite of them being the same penetration grade. The differences regarding the colloidal composition of neat bitumens shows significantly higher variability compared to the effect of wax modification. It is obvious, that these analytical methods are not feasible for identifying differences in the mechanical properties for wax-modified binders.

Aging usually increases the stiffness of the bitumen (for example its shear modulus) due to a decreasing content of volatile bitumen compounds and the formation of larger molecules due to oxidation. However, ageing also affects the mechanical properties of modifiers in the binder. At the same time, modifiers will influence the chemical stability of the bitumen compounds. These effects can be observed in the results of mechanical and chemical measurements. Firstly, the ageing did not affect the softening point of the wax modified bitumen. The softening point is primarily controlled by the wax modifier and can, therefore, not be applied for identifying the ageing state of the bitumen compounds.

The evaluation of the mechanical properties within a large temperature range applying DSR allows a better identification of ageing effects. At lower test temperatures (30 °C), the wax modified binder samples indicated the expected increase of shear modulus after short-term ageing. Though, at the elevated temperatures of 60 °C and 90 °C the shear modulus of the wax modified samples even indicate a decrease after ageing. Again, at these high temperatures the shear modulus represents the resulting stiffness of the wax network in the sample, whereas the increasing viscosity of the bituminous compounds still result in lower stiffness compared to the wax modification and therefore the ageing effect is not visible. Only with continued (long-term) ageing, the proceeding stiffening of the bitumen will also at elevated testing temperatures result in a stiffening of the wax modified bitumen.

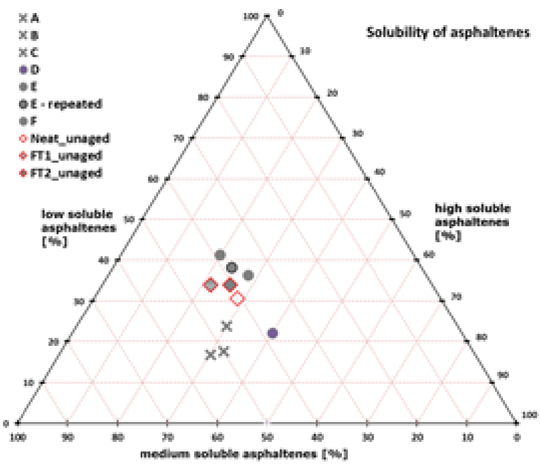

Fig. 16: Comparison between asphaltene components by [10] of different 50/70 bitumen types.

These effects can also be identified by the phase angle measurements. As indicated in Fig. **??** (1), increasing temperature will result in increasing phase angles identifying viscous properties. This temperature-dependent increase can also be observed for aged binders with lower phase angle values. However, for the wax-modified binders (2), three phases of temperature-dependency can be identified. Below the temperature of 40 °C, an increasing temperature results in increasing phase angles as the comparatively high viscosity of the bitumen predominates the overall rheological properties. Though, with further increasing temperatures up to 70 °C the temperature increase results in a phase angle decrease. This can be explained by the increasing importance of the structural wax network within the binder, with higher elasticity. Again, with increasing temperature up to a temperature of 90 °C, the wax network indicates reducing strength at temperatures near its melting point which results again in an increase of phase angle. Whereas the short-and long-term ageing have little effect on the shear modulus of the wax-modified binders, a shift in the temperature-depending phase angle can be observed, see Fig. 17 (3) and (4). Because of the increasing viscosity of the bitumen, the temperature identifying the change of predominating bitumen and wax properties is increasing to approximately 60 °C (after RTFOT) and 70 °C (after PAV).

When applying the Sol/Gel-model, the decreasing phase angle as a result of wax modification can be interpreted as increased Gel characteristics in this bitumen.Regarding the effects of ageing on the chemical composition of bitumen, the expected results are obtained for the unmodified bitumen (50/70). In the SARA analysis, decreasing contents of saturates and aromatics are observed, whereas resins and asphaltene contents increase. Only the decrease of asphaltenes for the PAV-aged samples is unreasonable. Here, the procedure for SARA detection may explain this value. Bitumen is dissolved in dichloromethane at the beginning of the TLC/FID and afterwards separated on chromarods using three different solvents successively. Non-solubles in dichloromethane will not be analyzed in the test as they do not show up in the applied samples. Reference [5] supports this assumption with the same experience describing “further oxidation of the asphaltenes to insoluble carboids. The carboids [. . .] do not dissolve in the solvent employed in spotting the sample on the Chromarod. Because the carboids are not transferred to the rods, the sum of the areas [. . .] is reduced.” Therefore, SARA analyses by TLC/FID demands for the separation of the asphaltenes prior to the experiment for detection of resins, aromatics and saturates.The increasing asphaltene content by ageing can be observed better by the results of asphaltene compounds analysis. Here a clear increase of asphaltene contents can be observed for the neat bitumen. The modification of the binder with F-T wax is influencing the ageing properties of the bitumen. Especially F-T wax 2 beneficially results in higher maltene contents (identified by asphaltene precipitation) as well as high contents of non-polar compounds of aromatics and saturates indicating less aged bitumen properties. This confirms the reduced ageing susceptibility observed in the DSR experiments.

Nonetheless, the following statements can be made about influence of F-T wax 2 on ageing with regard to the chemical characteristics:

- Lower increase of sulfoxides and carbonyl groups measured by infrared spectroscopy
- Lower content of resins and higher content of aromatic components within SARA analysis
- Higher content of maltene phase and lower content of asphaltenes (iC_8- asphaltenes) measured by the

Zenke test.

On the basis of lower iC_8-asphaltenes and higher maltenes after ageing the colloidal structure is more comparable to a Sol-type. As result of the chemical analyses it may be stated that F-T wax 2 has favorable properties compared to F-T wax 1.

With regards to rheological properties the following statements may be summed up from this study:

- Whereas the presence of wax considerably increases the shear modulus of the bitumen in unaged stage, it reduces its further increase due to ageing.
- For unaged wax-modified bitumen the wax will improve the binder's elasticity especially in the range of elevated service temperature. Due to ageing this beneficial effect is reduced but simultaneously counteracted by increasing binder viscosity.
- Similarly, the phase angle decrease due to ageing is superimposed by higher elasticity of the wax modifier.

The results of the phase angle measurements can be interpreted as follows: The wax modifiers change the bitumen characteristics in an unaged binder first into Gel bitumen and after ageing to Sol bitumen while the resulting stiffness is nearly constant.

With regard to rutting resistance assessed in the dynamic shear rheometer, the F-T modification shows the lowest phase angles at low frequencies. This will result in higher resistance against permanent deformation especially for slow moving or even static loads.

Caused by the lower content of iC_8- asphaltenes (degree of ageing) and higher elasticity F-T 2 has more positive ageing properties and better rheological properties compared to F-T 1 and the neat bitumen 50/70.

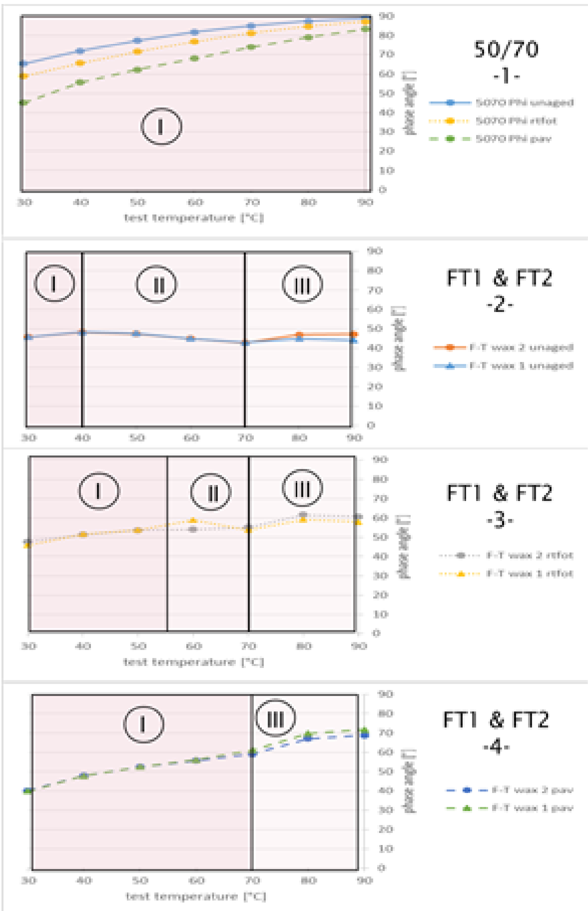

Fig. 17: Effect of F-T wax based on phase angle.

4 Conclusion

The following conclusions can be drawn from the results of the presented test campaign:

The evaluation of asphaltene contents shall be done by solution/precipitation procedures in order to include also non-soluble compounds especially of aged binders into the SARA evaluation. For conducting SARA evaluations, the asphaltene contents shall be evaluated by precipitation experiments. Afterwards the contents of saturates, aromatics and resins can be measured by TLC/FID of the residual maltene fractions.

Wax modification does not significantly affect the colloidal proportions of unaged bitumen. Differences between various neat bitumens of similar penetration grade are higher compared to differences originating from wax modification.

Wax modifiers change the chemical structures in bitumen and their physical properties. This results in reduced ageing susceptibility. There are no significant differences in the chemical and rheological behavior of F-T 1 and F-T 2, but in some aspects F-T 2 shows favorable ageing properties.

The stiffening effects of wax in bitumen are most pronounced in fresh bitumen. Aging results in a reduction of the elasticity-increasing effect of wax-modified bitumen. Especially at early age of the asphalt pavement, wax will reduce the rutting susceptibility of the asphalt mixture.

From DSR temperature-frequency sweeps the effects of ageing and of binder modification can be clearly evaluated. Especially the temperature-dependent phase angle provides valuable information regarding the rheological properties of modified binders.

The softening point ring and ball of wax-modified bitumen is predominated by the wax properties which hides any ageing effects of the bitumen. Therefore, this parameter is not feasible for estimating the properties of modified bitumen even after long-term ageing.

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The Effects of Magnetic Field on E. Coli Bacteria Accumulation and Disinfection in Water Advanced Treatment

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Abstract: As we know there are a lot of effects of magnetism on animals like Arctic navigation by birds and the water circles in vortex flows. It is also known that the people in north part of the Earth have bigger tendency to turn their head to the right and also there is more than 80 % probability that they choose the object on the right side if you give them an opportunity to choose an object between two choices on right side and on the left side. The performed research observes the influence of geographic poles while growing the E. Coli Bacteria in the medium placed at incubator. The growing of bacteria based on 0.5mcfarland standard was conducted in EMB (Eosin Methylene Blue). Another standard based on direct view by its accumulation in medium was observed too. Both of them were induced repeatedly for 6 – 7 times and the results showed that the bacteria while growing in the medium had tendency to accumulate on the right side (East Side) after bringing out of the incubator.

Keywords: E. Coli Bacteria; Magnetic Fields; Sheath; KAP; Disinfection.

1 Introduction of KAP (Karmania Aqua Purifier)

One of the systems used in disinfection of bacteria (in this case E. Coli) is a sheathing made in England and Switzerland that covers the pipe while disinfection in both large and small size. We use one of the small one in laboratory to cover around the pipe in circulation system. The accounts of bacteria before accounting is 650×10^4 and after 2.5 – 3 hour circulation it will decrease to 30×10^4 by imposing 300 Gs of magnetic field directly on bacteria.

We use our Modified model KAP (at first stage impose 60 – 70 Gs and then impose 300 Gs while we switch the faucet of both sides to maintain hydraulic detention for 1.5 hour). The time required at first stage (imposing the 60 – 70 Gs) while the faucet in the bucket with internal engine for water circulation is closed is 40 minutes and the time required for the second stage (imposing 300 Gs) is 50 minutes. Here is the result of the 2 forms based on 0.5mcfarland standard for accounting. The number of E. Coli Bacteria before imposing Magnetic field was 650×10^4

A model (created by modeling software like MATLAB) is a simple presentation of a complex system as we know in the cases of prediction and analyzing of the behavior of pollutants etc. Finally the environmental issues can be converted into the mathematic formula.

$$Q_\mathrm{b} = T \frac{1}{\mu D} \left(M_\mathrm{max} - M_\mathrm{min} \right) \tag{1}$$

Variables:
- Temperature (T)
- Volumetric flow rate (Q)
- Viscosity of liquid (μ)
- Differences in the intensity of magnetic field based on Gauss (M)
- Diameter of pipe (D)

Fig. 1: Process of the performed experiments.

2 Experimental Part

The presented figures depict some parts of the performed experiments.

3 Results

Tab. 1 summarizes the gained results concluding from the performed experiments.

4 Conclusion

The gained results showed that the bacteria growing in medium had tendency to accumulate in right side (East Side) after bringing out of the incubator. The number of E. Coli Bacteria before imposing magnetic field was 650×10^4DC/mL and the number of E. Coli Bacteria after the procedure was less than 30 DC/mL.

This research has been done in laboratory conditions and small diameter pipe was used. As I have already mentioned in large portions we use large diameter pipe with larger sheathes and much more magnetic intensity

Fig. 2: Process of the performed experiments (continued).

Tab. 1: The results of performed experiments.

Numbers of E. Coli Bacteria crossed from KAP (at first stage impose 60 – 70 Gs and then 300 Gs)	Numbers of E. Coli Bacteria crossed from magnetic sheath (300 Gs)	Time
[DC/mL] $\times 10^4$	[DC/mL] $\times 10^4$	[hr]
	= 550	0.5 hr, 1^{st}
< 40	= 400	0.5 hr, 2^{nd}
	= 300	0.5 hr, 3^{rd}
	= 150	0.5 hr, 4^{th}
	< 30	0.5 hr, 5^{th}

is imposed but it is more expensive than smaller diameter pipe. If we impose lower levels of magnetic field by applying detention and hydraulic pause in the system, we can reach the same results and it will be economically less expensive. In order to prevent from hydraulic hammer impact we should close the faucet gradually.

Acknowledgement

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Shrinkage of the Cement Pastes with Different Amount of Finely Ground Recycled Concrete

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Abstract: Complete recycling of the old concrete is very problematic mainly because of fine fraction (< 1 mm). Some studies focus on using recycled concrete powder as thermal activated binder or alternative raw material for manufacture of the Portland clinker. But those approaches are not sustainable and environmental-friendly. Use recycled concrete powder as cement replacement or filler seems to be a better way. This study deals with influence of finely ground recycled concrete (FGRC) on shrinkage of cement pastes. Finely ground recycled concrete was produced by LAVARIS Ltd. by using high speed mill. The old concrete was obtained from old railway sleepers. Four sets of cement paste with different amount (0, 33, 67, 100 wt. %) of FGRC were prepared. Increasing amount of FGRC negatively influenced shrinkage of the cement paste. It is mainly caused by increasing w/c ratio due to higher water demands of FGRC. Improvement could be achieved by using high amount of superplastizers.

Keywords: Recycled Concrete; Concrete Powder; Finely Ground; Substitute Binder; Cement Paste; Shrinkage.

1 Introduction

Recycling of the concrete waste is relatively well known in construction practice. Old concrete is usually crushed and separated into several fractions. Low quality concrete waste can be used for embankment or unbound roads [1]. Coarse fraction of concrete waste can be used as recycled aggregate into new concrete mixture [2]. Lack of recycled aggregates is the increased water absorption which negatively affects the workability, the mechanical properties and durability of the concrete. But those effects could be diminished by using proper admixtures and additives. The concrete with recycled aggregate can be comparable with concrete with natural aggregate [3]. A problem arises with processing very fine fraction having a grain size <1 mm (powder) generated by the recycling of old concrete. So far has not been found suitable application for this fine concrete waste.

There can be found several ways in current research works. Recycled concrete powder mainly consists of hardened cement paste and aggregate residues. Therefore first attempts try thermally reactivate binding properties of the concrete powder. It was found that the cement paste in a concrete powder can be dehydrated by proper heat treatment. Studies show that the hardened cement paste in concrete powder treated by a temperature of 500–800 °C is composed mainly of dehydrated C-S-H gel, CaO and partly from C-H. After mixing with water, there was a restoration of the hydration products of C-S-H gel, ettringite and $CaOH_2$ [4].

Similar way for using recycle concrete powder is partial replacement of raw mixture for Portland clinker production. Studies have shown that it is possible to utilize a recycled concrete powder as an alternative to conventional raw materials, primarily for the source of SiO_2 and in some cases, due to high content of CaO, for limestone also [5]. But the total amount of recycled material in mix should not exceed 30 % [6].

However both mentioned methods require high amount of energy and produce CO_2. On the other hand non-renewable natural resources are preserved. As environmental friendly and cheaper appears the possibility of using recycled concrete powder as filler for asphalt mixtures [7]. Very interesting is also the possibility to use the powder for production of geopolymeric binder [8]. Recent studies suggest that it is also possible to

Tab. 1: Composition of the tested samples.

Mixture	Cement (CEM I 42.5 R) [g]	FGRC [g]	Water/mixture mass ratio	Flow expansion test [mm]
A (REF., 0 wt. % FGRC)	1000	-	0.35	130
B (33 wt. % FGRC)	670	330	0.38	130
C (67 wt. % FGRC)	330	670	0.42	130
D (100 wt. % FGRC)	-	1000	0.45	130

use recycled concrete powder as a partial replacement of cement [9, 10]. Therefore this study is focused on the influence of cement replaced by recycled concrete powder on shrinkage of the cement paste with finely ground recycled concrete (FGRC).

2 Materials and Samples

The samples were made of Portland cement CEM I 42.5 produced in Radotín and finely ground recycled concrete. FRGC was produced by LAVARIS Ltd. by using high speed mill. The old concrete were obtained from old railway sleepers type PB2 and SB8. FGRC has fraction 0 – 65 μm and the specific surface area was equal to 412 m^2/kg. Four mixtures were prepared where cement was replaced by 0, 33, 66 and 100 wt. % of FGRC (Tab. 1). Due to different behavior of cement and FGRC when mixed with water, the water-mixture ratio was within the range between 0.35 (mixture A) and 0.417 (mixture D), depending on the amount of FGRC. Unifying parameter for these mixtures was workability defined by the flow expansion test. This solution provided a similar homogeneity for all mixtures and in the case of mixtures containing FGRC reduced the size and amount of technological pores in the hardened composite [11]. Each set contained 6 prismatic samples having dimensions of 40 $\times$ 40 $\times$ 160 mm. The samples were removed from casts after 2 days and shrinkage measurement were performed. The specimens for microstructure examination by optical and scanning electron microscopy were cured for 28 days in water at the temperature of 21 $\pm$ 2 °C.

3 Measurement Methods

The first step of measurement was the length measuring of each box in mold before the mixture was placed into. The measurement of samples was performed after 2 days when they were removed from casts. The resulting shrinkage of each sample was related to the length of 1 mm. The microstructure of the samples was examined using scanning electron microscope in BSE mode. A Philips XL30 ESEM-TMP FEI scanning electron microscope was used for identification the individual phases and their amount at 100$\times$ and 250$\times$ magnification. SEM was used in the BSE mode at low pressure (9–10 Pa) and at accelerating voltage set to 30 kV.

4 Results and Discussion

Diagram of shrinkage is presented in Fig. 1. It is obvious that increasing amount of FGRC negatively influences shrinkage of the cement past. This difference between shrinkage of the samples is probably caused by higher water content in mixtures due to higher water demands of FGRC. Higher shrinkage could be also caused by changes in hydration process of the samples with FGRC. This is supported by SEM images (Fig. 2) where the distribution of the various phases within the cement paste can be detected [12]. The aggregate contained in FGRC can be identified as dark grey spots in the sample B. The decrease of unhydrated cement grains (displayed as white spots) is obvious in case of samples containing FGRC compared with reference sample without FGRC. Given the ratio of cement and FGRC in the sample B, the amount of unhydrated cement grains should be about 33 % lower than in the sample A. However, the difference reached almost 60 %.

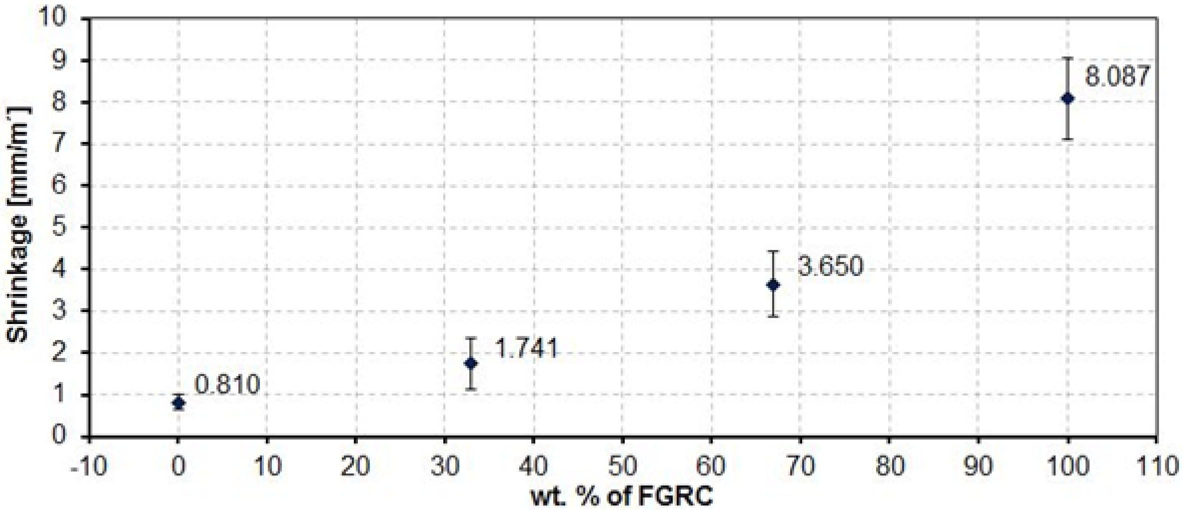
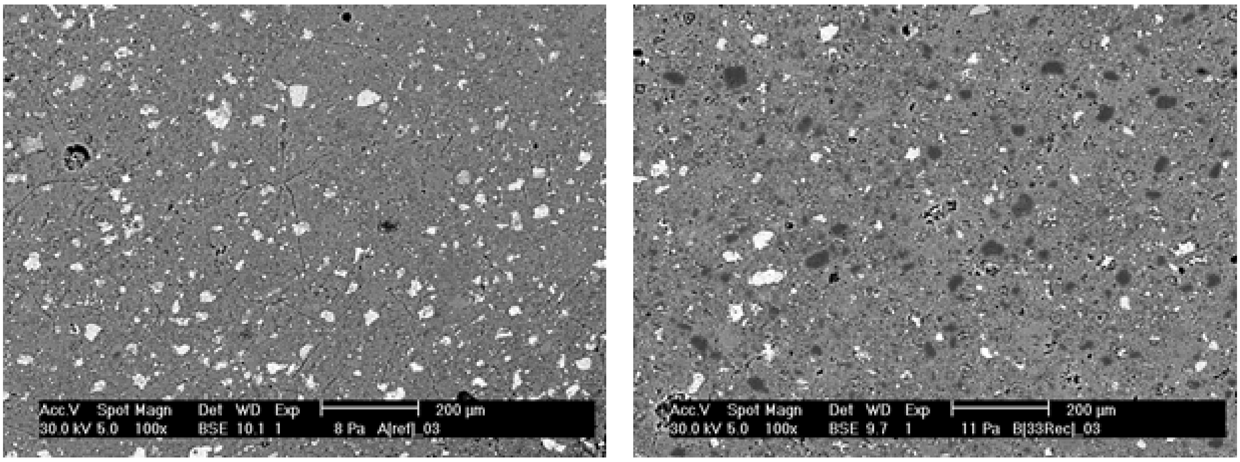


Fig. 1: Dependence of shrinkage of the samples on cement replacement by FGRC.



(a) sample A (REF., 0 wt. % FGRC)

(b) sample B (33 wt. % FGRC)

Fig. 2: SEM images of the samples with different amount of FGRC at 100 $\times$ magnification..

5 Conclusion

The presented results are a part of the larger research. It was found that FGRC can act as a filler and cement replacement. The presented work was focused on the influence of cement replacement by FGRC on shrinkage of the cement pastes. Based on the results it can be concluded that:

- increase in shrinkage directly depends on the amount of FGRC in a sample,
- the samples with 33 wt. % of FGRC have higher shrinkage compared with the reference samples without FGRC, but still not so high,
- higher shrinkage is probably caused by higher water demands of samples with FGRC compared with cement,
- difference in shrinkage could be reduced by using superplasticizers,
- the results in related research suggest that FGRC could be used as a partial cement replacement (if added below 33 wt. %) with minimal negative impact on composite properties.

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Effect of Micronized Recycled Materials on Mechanical Properties of Cement Mortar with Crushed Bricks

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Abstract: The article presents the result of mechanical tests on cement mortar with crushed bricks and micronized recycled materials. Mixtures with 2 different micronized materials (recycled concrete prefabricated at the factory and marble powder) were tested. Micronized materials were produced in the company Lavaris Ltd. (Czech Republic) by use of high speed grinding. The investigated parameters were compressive and flexural strength and bulk density of the samples. Flexural strength and compressive strength were determined for the 28 days old specimens. Testing was performed on beams of dimensions 40 × 40 × 160 mm. The mechanism behind the increase of mechanical strength and elastic stiffness is explained by means of microscopic analysis, which was carried out by electron microscope. Electron microscopy was performed using a ZEISS merlin at the UCEEB CTU in Prague The aim of this article was to determine the influence of type recycled material on the resulting mechanical properties.

Keywords: Recycled Materials; Compressive Strength; Flexural Strength; Electron Microscopy; X-Ray Spectrometry.

1 Introduction

Mortar is one of the most popular building materials and it has been extensively used since the antiquity times. Romans people successfully built structures made of lime mortar with crushed brick before 1st century BC. Mortar consisting of lime and crushed bricks was called cocciopesto [1]. When designing historic mortars of cocciopesto type, it has been found that the presence of crushed bricks increased to mortar strength [2]. Mixtures of modern cocciopesto were designed with Portland cement as binder and micronized recycled materials as microfiller. Two variants of recycled material were used to increase the content of recycled material in mixtures.

First micronized recycled material was waste concrete prefabricated at the factory (in our case railway sleepers). In the work [3–6] micronized recycled concrete was used to compensate natural filler in the mortar. The result was that the replacement of the recycled material has reduced the workability of mixture, all of the modified mixture showed higher compressive and tensile strength of the bending and all mixtures showed the same adhesion, shrinkage and water absorption.

Second micronized recycled material was marble powder (in our case from west bank of the Jordan). The marble solid and sludge wastes were used in many applications such as the construction industry [7], paper industry [8] and cement manufacture [9]. Marble dust in mortar has resulted in increase of mechanical properties [10].

Tab. 1: Composition of the tested materials..

Set	**Mass [kg]**				
	CEM I	**Sand** (0-4 mm)	**Brick** (2-5 mm)	**Marble powder** (< 0.04 mm)	**Recycled concrete** (0-0.1 mm)
A	0.07	1.75	1.05	0	0.63
B	0.07	1.75	1.05	0.63	0
C	0	1.75	1.05	0	0.7

Tab. 2: Water ratio, workability and bulk density of the tested materials.

Set	Water ratio	Spillage (after 10 impulses) [mm]	Spillage (after 20 impulses) [mm]	Bulk density [kg/m^3]
A	0.512	105	120	1749
B	0.518	111.5	118.5	1724
C	0.568	109.5	118	1777

2 Tested Materials and Specimens

All of the tested mixtures were composed of natural sand (from Zálezlice with a fractions 2-4 mm) and crushed bricks (from Bratronice with a fraction 2-5 mm). Mixtures A and B contained Portland cement CEM I 42.5R (Radotín) and microfiller. Mixture C contained only microfiller without Portland cement. Recycled railway sleepers (microground by Company Lavaris) of a fraction 0-0.1 mm was used as microfiller for mixtures A and C. Marble powder (microground by Company Lavaris) of a fraction 0-0.04 mm was used as microfiller for mixtures B. The weights of individual components in the mixtures presents Tab. 2.

Amount of water was designed to maintain the same workability of fresh mixture. Consistency of the mixtures was determined using slump-flow test. Tab. 2 presents specifications for individual sets. Six specimens were made for each mixture and their dimensions were equal to 40 $\times$ 40 $\times$ 160 mm. Samples were demolded the day after production, and the samples were stored loosely in a laboratory environment at 22 $\pm$ 1 $^\circ$ C and relative humidity 50 $\pm$ 2 %.

3 Experimental Methods and Results

All samples were weighed and measured their dimensions, before was used destructive methods. These values were used to calculate bulk density of individual samples / materials, which can be used to compare the effects of individual materials on the monitored parameters (Tab. 2). The values of bulk density were similarly for each sample.

The flexural and compressive strength were determined using the Heckert device, model FP100 on the 28 days old specimens. The testing was displacement controlled at a constant rate of 0.1 mm/s in the case of three-point bending and 0.3 mm/s for the compressive test. The distance between supports for three-point bending test was equal to 100 mm. The uniaxial compressive test was performed on the broken halves of the specimens with effective dimensions of 40 $\times$ 40 $\times$ 80 mm.

Value of compressive strength of the mixture with micronized recycled concrete (A) was higher about 1 MPa than the compressive strength of the mixture with micronized marble powder (B) and the mixture without Portland cement (C) had similarly value of compressive strength as the mixture with a micronized marble powder.

In the next step, electron microanalysis (Fig. 2) was used for determine the effect of microfiller on the resulting mechanical properties of the samples. The recycled railway sleepers and marble powder samples were analysed chemically to determine the percentages of their different constituents. Energy-dispersive (EDS) and

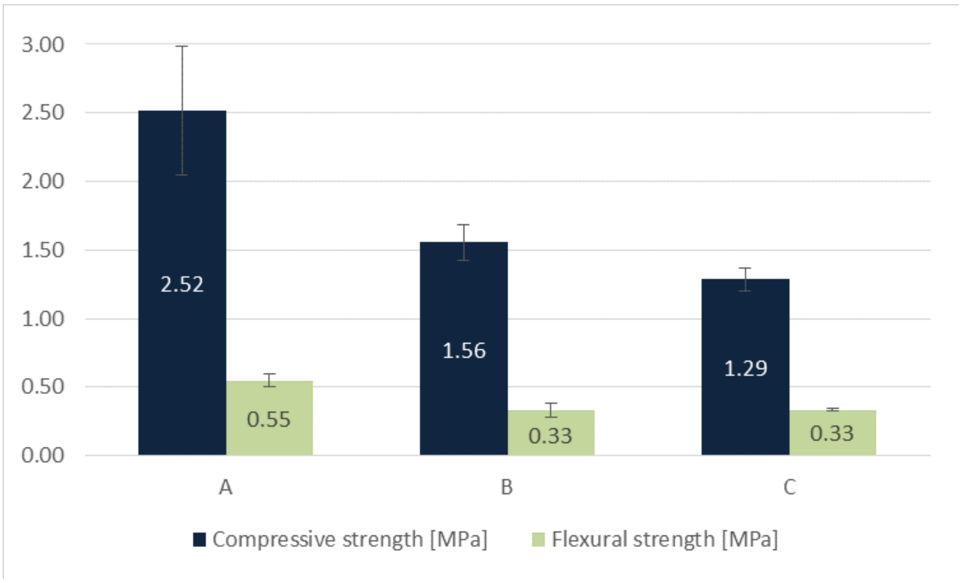
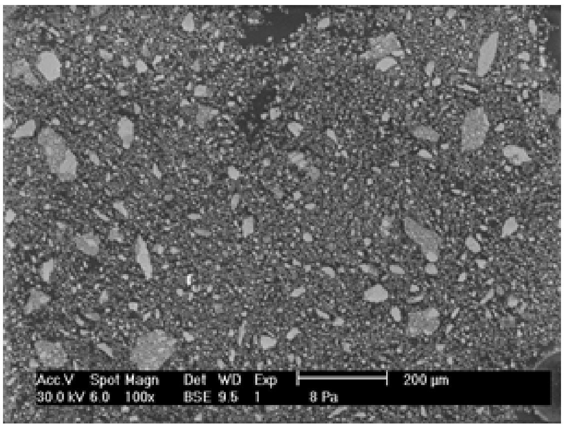
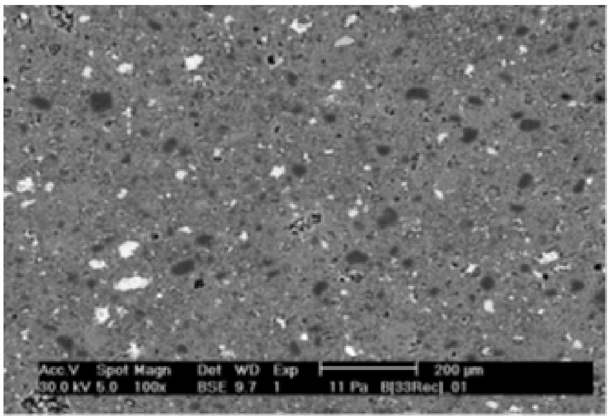


Fig. 1: Comparison of compressive and flexural strength (with standard deviation).

wavelength-dispersive (WDS) X-ray spectrometry was used. $CaCO_3$ content marble powder samples (Fig. 2a) was 99 % and $MgCO_3$ wasn't dominant with a content of less than 1 %. Marble powder samples was completely inert. The recycled railway sleepers samples (Fig. 2b) contained 10 % of clinker minerals, which joined to hydration of the cement.



(a) marble powder samples



(b) mixture with micronized recycled concrete (A)

Fig. 2: The image from Environmental scanning electron microscope, magnification 100×.

4 Conclusion

The tested materials seem very promising for the use as a microfiller and binder. Micronized recycled concrete seems most advantageous from the viewpoint of the mechanical properties of the tested materials. Micronized recycled railway sleepers (A, C) contained 10 % non-hydrated or milling activated clinker minerals which had influence on the development of mechanical properties. Marble powder samples (B) was completely inert and had only function of microfiller. The future work of our team will deal with micromechanical study of recycled railway sleepers and marble powder as presented e.g. in [11, 12].

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Effect of Microstructure on Mechanical Properties of Fly Ash-Based Stabilizer

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Abstract: The presented article deals with fly ash-based stabilizer used in railway trackbed and investigation of a development of its properties. Initially the compression strength, static deformation modulus, permeability, thermal conductivity, moisture and bulk density of the stabilizer were observed. The increase of the compression strength and static deformation modulus was detected on extracted samples during long-term observation in years 2005 to 2015. In particular the article is focused on principles and reasons, why the mechanical strength and stiffness of the stabilizer increased. An electron microscopy was employed to analyze the microstructure. Its results provide explanation of the long-term improvement of the stabilizer.

Keywords: Fly Ash-Based Stabilizer; Railway Trackbed; Compressive Strength; Microstructural Analysis; Electron Microscopy.

1 Introduction

At the Department of Railway Structures, Faculty of Civil Engineering, Czech Technical University in Prague several projects with the focus on the use secondary materials were realized between 2002 and 2011. In particular, the projects were focused on fly ash-based stabilizer produced by Chvaletice plant (CHCE). In 2005 there was established a 330 mm long trial section (Fig. 1) in the railway station Smiřice (the railway track between cities Pardubice – Liberec) for the long-term monitoring of the fly ash-based stabilizer. In the trial section the layer with thickness 250 mm was laid on the subgrade surface made from argillaceous limestone which is sensitive to an effect of rainwater. The experimental layer made of stabilizer was covered with crushed stone mixture with thickness 150 mm [1, 2]. The goal of the long-term monitoring was to determine whether the properties of the stabilizer change due to environment and railway traffic [3].

2 Tested Materials and Specimens

The composition of the fly ash-based stabilizer used for the layer in railway track-bed is in the Tab. 1. Based on [4] there was no assumption regarding the formation of ettringite, which negatively affects the size of swelling [5]. Parameters of the stabilizer used in railway station Smiřice are summarized in Tab. 2.

The mechanical properties were determined based on a set of laboratory tests performed on extracted specimens. The extraction of specimens was carried out using boreholes with diameter of 100 mm. For drilling the stabilizer layer the rig based on the diamond coring tool Hilti DD130 was chosen. The coring tool contained the diamond core bit, the water supply unit and the power generator. Using hand-guided drilling the hole was able to sample the stabilizer within the whole thickness of the layer (up to 280 mm) [3].

There was extracted a total of 111 specimens for determination as follows laboratory tests: compressive strength, bulk density (non-saturated), permeability and thermal conductivity. There are mean values of properties in 2005 and in 2014 complemented with static deformation modulus of whole stabilizer layer in Tab. 3. Measurement of the static deformation modulus was conducted in the trial section in Smiřice with static plate load test.

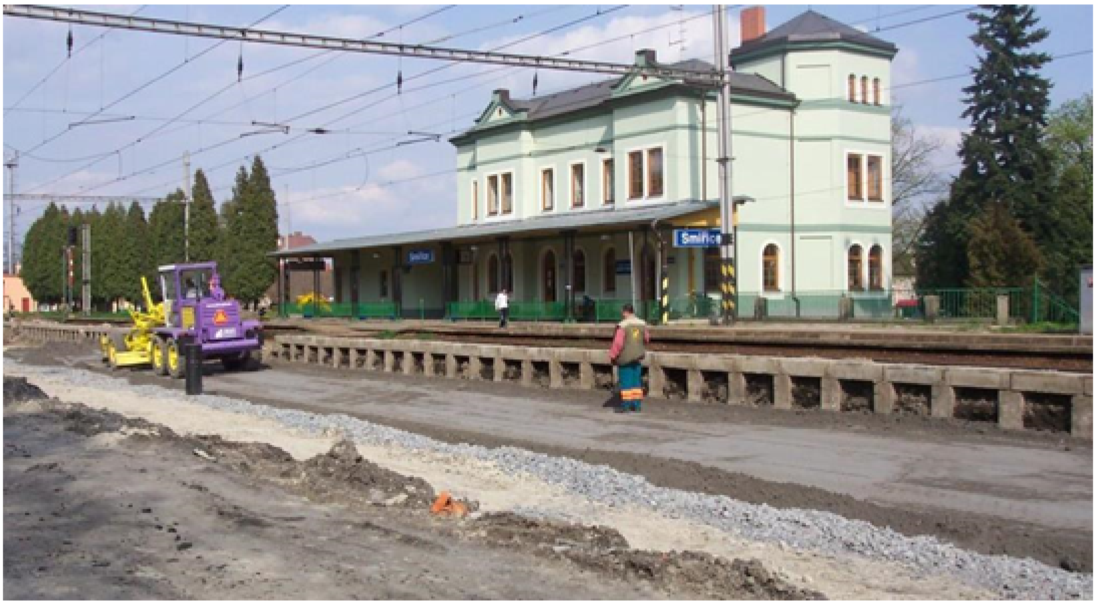


Fig. 1: Placing of fly ash-based stabilizer layer in railway station Smiřice in 2005.

Tab. 1: Fly ash-based stabilizer composition – mixture R4 [2].

Description of ingredients	Weight proportion [%]
Fly ash – lignite from ECH	52.0
Calcium oxide – lime CL 90 (origin: Kotouč Štramberk)	3.0
Gypsum – product of fume gas desulphurization of ECH	28.4
Water – process water	16.6

Tab. 2: Properties of fly ash-based stabilizer layer [2].

Property	Value
Bulk density [kg m^{-3}]	1236
Compressive strength (non-saturated specimens, 201 days after placing) [MPa]	3.5
Coefficient of thermal conductivity [W $m^{-1}K^{-1}$] (at 45.5 wt. % of moisture)	0.8
Permeability [m s^{-1}]	1.3×10^{-7}

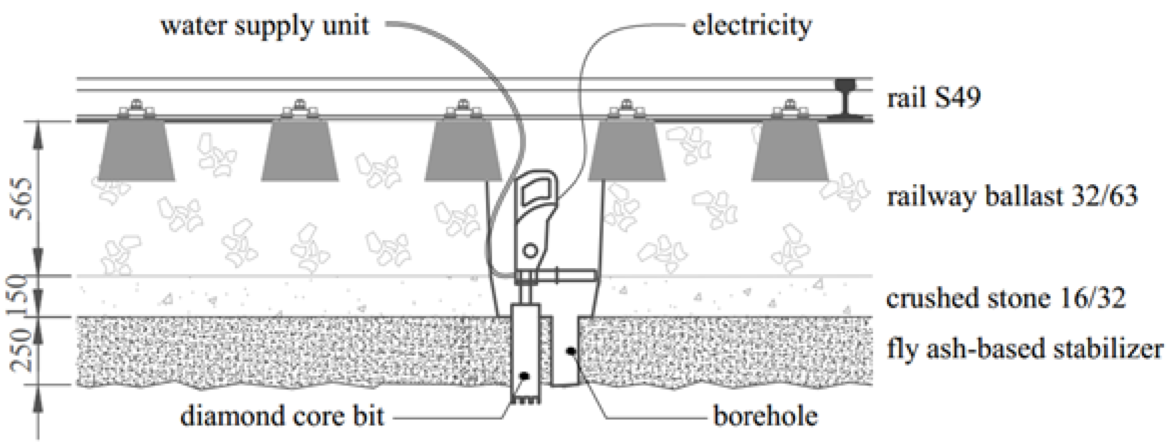


Fig. 2: Method of specimen extraction using core drilling in railway station Smiřice [3].

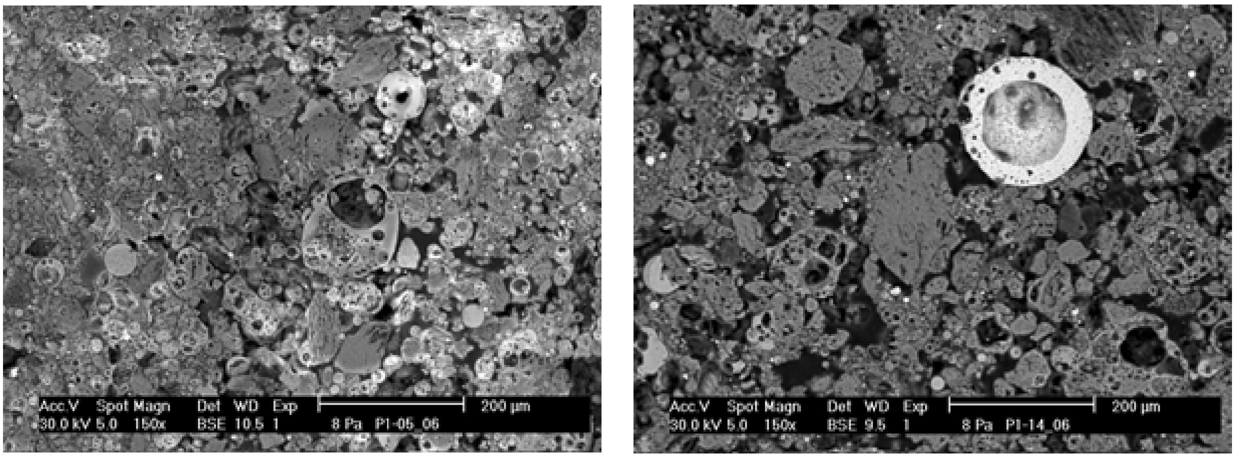
Tab. 3: Comparison of fly ash-based stabilizer properties in 2005 and in 2014 [2].

Parameter	Mean value	
	Autumn 2005	Autumn 2014
Bulk density [kg m^{-3}]	1233	1307
Compressive strength [MPa]	3.5	5.8
Static deformation modulus of the layer [MPa]	363	900

In general the behavior of the layer in the first three years may be characterized by the increase of compressive strength, which significantly increased together with the static deformation modulus. The improvement of the properties slowed after three years. Such behavior of the fly ash-based stabilizer is presumably caused with changes on the structural level of this material. For this reason the microscopic analysis of stabilizer specimens extracted between 2007 and 2014 was carried in the framework of project CESTI in 2015.

3 Experimental Methods and Results

In order to obtain detailed information about individual phases 4 extracted samples were investigated using electron (BSE) and optical microscopy. However, for since the BSE images provide more detailed description, only these are presented here. The samples extracted in 2005 and 2014 are presented here, the rest can be found in [2]. The investigated samples of the fly-ash stabilizer were polished, and microscope HR FEG MERLIN (by Zeiss) located at UCEEB institute in Buštěhrad, was employed for the study.



(a) sample extracted in 2005 (b) sample extracted in 2014

Fig. 3: Electron microscopy image of samples extracted in 2005 and 2014; magnification 150×.

The results demonstrate that the extraction methodology of the fly ash-based stabilizer layer by hand-guided core drilling was well chosen and allows to obtain a sufficient amount of specimens for following laboratory test. The main reason causing the increase of the stabilizer compressive strength depending on time is synthesis of the new binder co called CASH gel made of slug-ash particles in alkaline environment of CaO respectively $Ca(OH)_2$. CASH gel synthesis in track-bed given by specific environment is slow and the synthesis intensity is caused:

- directly with the "activator", i.e. CaO, resp. $Ca(OH)_2$,
- together with low temperature of the stabilizer in the railway track-bed,
- and in addition with low water saturation, resp. oscillating water content, which depends on atmospheric precipitation.

The metamorphic process, the recrystallization of the flue gass desulphurization gypsum (FGD gypsum) particles is as follows: from originally fine-grained aggregates varied in the shape gradually grow larger gypsum crystals of flake-shape to create an interconnected net of microcenters [3,5], yielding a compact structure. Such crystallization results in strengthening behavior of the stabilizer.

4 Conclusion

The studied track-bed stabilizing material exhibited the strength and stiffness increase over the period of a decade, however, to generalize the findings further studies must be carried out. The presented results are related only to the studied material based on fly-ash when deposited in specific conditions. Without a proper study it is not possible to apply the results on any fly-ash based material. The same applies for instance on recycled concrete based on micro-ground activated cement [**?**].

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