CEDR Transnational Road Research Programme Call 2012: Recycling: Road construction in a post-fossil fuel society

funded by Denmark, Finland, Germany, Ireland, Netherlands, Norway



Conférence Européenne des Directeurs des Routes Conference of European Directors of Roads



Report on harmonised mix design procedure: Recommendations for mix design procedure, mixing, curing and applicable test methods

Deliverable D1.2 December 2015

Coordinator: Czech Technical University in Prague (CTU) Partner 1: University of Kassel (UKassel) Partner 2: University College Dublin (UCD) Partner 3: Laboratório Nacional de Engenharia Civil, I.P. (LNEC) Partner 4: Wirtgen GmbH

CoRePaSol Characterization of Advanced Cold recycled Bitumen Stabilized Pavement Solutions

Report on harmonised mix design procedure: Recommendations for mix design procedure, mixing, curing and applicable test methods

Deliverable D1.2

Due date of deliverable: 31.12.2014 Actual submission date: 15.12.2015

Start date of project: 01.01.2013

End date of project: 31.12.2015

Author(s) of this deliverable:

Fátima Batista, LNEC, Portugal Jan Valentin, CTU, Czech Republic Konrad Mollenhauer, University of Kassel, Germany Ciaran McNally, UCD, Ireland Michael Engels, Wirtgen GmbH, Germany Jan Suda, CTU, Czech Republic Zuzana Čížková, CTU, Czech Republic Diana Simnofske, University of Kassel, Germany

Version: 15.12.2015

CONTENT

E	Executive summaryi							
1	Introd	uction	4					
2	Synthe	Synthesis of mix design approaches for cold recycled bitumen stabilised materials						
	2.1 C	old-recycling techniques	7					
	2.2 M	aterials used and typical compositions	7					
	2.3 La	aboratory test procedures applied for mix design	13					
	2.3.1	Introduction	13					
	2.3.2	Compaction procedures	14					
2.3.3 2.3.4		Test specimens dimensions	21					
		Laboratory accelerated curing procedures	22					
	2.3.5	Laboratory test procedures for determination of mix composition	25					
	2.4 Q	uality control procedures	28					
	2.5 C	onclusions from review on specifications and international literature	29					
3	Experi	mental studies	31					
4	Test s	pecimens characteristics prepared using different compaction methods	34					
	4.1 E	xperimental study A1	34					
	4.1.1	Selected materials	34					
	4.1.2	Description of the study	37					
	4.1.3	Results	38					
	4.1.4	Main conclusions	43					
	4.2 E	xperimental study A2	44					
	4.2.1	Selected materials	44					
	4.2.2	Description of the study	45					
	4.2.3	Results	45					
	4.2.4	Main conclusions	47					
	4.3 E	xperimental study A3	48					
	4.3.1	Selected materials	48					
	4.3.2	Description of the study	50					
	4.3.3	Results	51					
	4.3.4	Main conclusions	52					
5	Cold r	ecycled mixture characteristics and performance, considering the effect of						
	different curing procedures							
	5.1 In	troduction	53					
	5.2 E	xperimental study B1	56					
	5.2.1	Selected materials	56					
	5.2.2	Description of the study	56					
	5.2.3	Main results	57					
	5.2.4	Main conclusions	61					
	5.3 E	xperimental study B2	61					
	5.3.1	Selected materials	61					
	5.3.2	Description of the study	61					
	5.3.3	Main results	62					
	5.3.4	Main conclusions	68					
	5.4 E	xperimental study B3	68					
	5.4.1	Selected materials	68					
	5.4.2	Description of the study	68					
	5.4.3	Results	69					
	5.4.4	Main conclusions	71					
	5.5 E	xperimental study B4	71					



	5.5.1	Selected materials	'	71
	5.5.2	Description of the study		71
	5.5.3	Results		72
	5.5.4	Main conclusions		74
	56 Exp	erimental study B5		74
	561	Selected materials		75
	562	Description of the study	•	75
	563	Description of the study		76
	5.0.5	Main conclusions		76
	5.0.4	ridin conclusions	••	70
	5.7 EXP	Calastad matariala	••	11
	5.7.1	Selected materials	••	11
	5.7.2	Description of the study	••	77
	5.7.3	Results	••	78
	5.7.4	Main conclusions	••	79
	5.8 Exp	erimental study B7	•••	79
	5.8.1	Selected materials		79
	5.8.2	Description of the study	8	80
	5.8.3	Testing	8	82
	5.8.4	Results	8	84
	5.8.5	Main conclusions	8	85
	5.9 Exp	erimental study B8	;	86
	5.9.1	Selected materials	8	86
	5.9.2	Description of the study	{	86
	5.9.3	Results	. 1	89
	594	Main conclusions		94
	5 10 Exp	erimental study R9		94
	5 10 1	Selected materials	· • ·	97
	5 10 2	Description of the study	· • ·	05
	5.10.2	Main resulte		05
	5.10.5	Main penelupiana		90
~	5.10.4	Main conclusions	· - }	90
ю	Ellector	binder type on cold recycled material performance related properties		97
	6.1 Use	of different bituminous binders for foamed bitumen production and the		~-
	influ	ence on cold recycled mixture properties	9	97
	6.1.1			97
	6.1.2	Experimental study - scope and found impacts	10	00
	6.1.3	Mix design	1	03
	6.1.4	Results and Conclusions	1	05
	6.2 Use	of alternative hydraulic binders: fly-ash	1	06
	6.2.1	Introduction	1	06
	6.2.2	Used materials	1	80
	6.2.3	Cold recycled mix assessment methodology	1	11
	6.2.4	Results and Discussion	1	14
	6.2.5	Conclusions	1	17
7	Sensitivi	ty studies on the influence of inhomogeneous materials properties of		
	reclaime	d asphalt	1	19
	7.1 Intro	duction	1	19
	72 Exp	erimental work and laboratory tests	1	19
	721	Mix design	1	10
	722	Mix variations	1 1	20
	722	Sample preparation and curing procedure	1	20
	7 0 A	Laboratory tosts	1	20 21
	1.2.4 7.2 F	Void contant	1	ר ב ר ב
	1.2.0	VUIL CUITETII	1	21
	1.2.0	mairect tensile strength	E	22



	7.2.	7 Water susceptibility	123
	7.2.	8 California bearing ratio (CBR)	123
7	.3	Discussion and Conclusions	124
8	Gen	neral conclusions and recommendations	125
8	.1	Cold recycling materials	125
8	.2	Mixing	126
8	.3	Compaction	126
8	.4	Curing	127
8	.5	Test methods	129
9	Hari	monized advanced mix design procedure proposal	131
10	Ack	nowledgement	133
11	Refe	erences	134



Executive summary

There is a broad consensus that one factor hampering a widespread use of cold recycling techniques is the lack of a suitable and harmonised mix design procedure across Europe. Because of different existing laboratory compaction methods, the influence of miscellaneous accelerated laboratory curing procedures, the suitability of test procedures for moisture susceptibility and assessments of other performance related characteristics as well as the evaluation of different mix design test methods and requirements the common understanding and comparability is difficult. The solution might be a European-wide unified design approach. The overall focus should additionally respect the availability of defined test procedures and mix design approaches to regular laboratories which are providing their services to road administrators and/or contractors.

In order to address these issues, the first work package of CoRePaSol project (WP1 - "Advanced mix design of cold recycled bitumen stabilized material") has focussed on the assessment of available mix design procedures, test methods and specifications (standards) for use on cold recycling in pavement rehabilitation and construction. As a result, it was found that the applied mix designs vary considerably depending on general pavement design, climatic conditions and the existing local/regional practice. Therefore, the available materials considered as cold recycled materials (CRM) were described in more detail according to their binder composition, as follows:

- Bitumen-stabilised material (BSM).....1 % 3 % bitumen; ≤ 1 % cement
- Bitumen-cement-stabilised material (BCSM).....1 % 3 % bitumen; 1 % 3 % cement
- Cold asphalt mix (CAM)≥ 3 % bitumen; no cement

Of course this basic split in four key typical options of cold recycling is not comprehensive. In case of bitumen there will be always additional subdivision in bituminous emulsion and foamed bitumen, whereas both can contain additional additives (foaming improvers, rejuvenators etc.), or even fluxing agents. In case of hydraulic binders cement is commonly the most used binder in Europe. But similarly lime, hydraulic road binder or even fly-ash can be used separately or as a mixed binder. Cold asphalt mixes represent then a specific type of asphalt mixes which are so far broadly used only in some parts of France. These mixes can primarily contain 100 % of virgin aggregates, nevertheless combination with reclaimed asphalt material is a more sustainable alternative, which is also simpler especially if compared to hot mix asphalt. Finally the content of binders in these types of mixes (cold mixes or cold recycled mixes) can have a large variety from partially coated mixes (semibound) up to fully coated mixes (similar to HMA just produced and paved cold).

Regarding the various climatic conditions within Europe following recommendations regarding the applicability of various cold recycling materials could be made:

- Bitumen stabilised materials (cement content ≤ 1 %):
 - applicable with foamed bitumen for flexible pavements in cold climate;



- applicable with bitumen emulsion in dry or moderate climate;
- not applicable with bitumen emulsion in moist climate.
- Bitumen-cement stabilised materials (cement content > 1 %):
 - applicable for moist climate;
 - adequate for high early-life strength or required increased bearing capacity.

Based on these material approaches, a six-step mix design principle could be derived and recommended as best practice. Within these steps the variety of test methods and parameters shall be reduced in order to allow future comparisons of gained experience:

Step 1: Analysis of reclaimed road materials for suitability as mix granulates: aggregate grading (before and after bituminous binder extraction), determination of bitumen content, moisture content of the material as available in the road or at a stockpile.

Regarding the grading requirements of the mix granulate following threshold values shall be applied:

- content of fines (< 0,063 mm): 4 10 %
- content of fine aggregates (< 2 mm): 15 % 40 %

Step 2: Choice of suitable binders (bituminous emulsion / foamed bitumen, mineral or hydraulic binder type) and optimisation of foamed bitumen properties.

Step 3: Evaluation of optimum compaction water content and reference bulk density. The application of modified Proctor test is the most applied procedure internationally. However, shortcomings from an impact compaction procedure are reported (mainly when relatively high amounts of binder are applied). Therefore, an alternative compaction method might be more feasible for conducting tests for evaluating optimum compaction water content and reference density. Some additional experiments are necessary to define a suitable compaction procedure based on controlled compaction energy. Finally it is recommended not to overestimate the role of water content since it is only one of several factors influencing good or bad mix design.

Step 4: Mix preparation and specimen compaction

After mixing the cold recycled material in laboratory specimens need to be compacted. It has been found that it is not possible to define for Europe just one laboratory compaction method. Adequate compaction procedures identified within CoRePaSol project are:

- gyratory compaction according to EN 12697-31 (which needs however to be adapted to cold mixtures: e.g. perforated moulds/plates, no need for heating materials);
- static compaction with double-plunger and a compaction stress of 5.0-7.5 MPa, depending on the type of equipment/method of applying the static load (e.g. loading rate) and best national technical practice. In this case a new compaction standard needs to be drafted and prescribed for Europe.



Step 5: Curing of specimens

For simulating site-development of strength, suitable laboratory curing procedures are required. From the laboratory comparisons following curing methods are recommended for specimens, which are demoulded 1 day after compaction:

- for BSM (cement content \leq 1 %): curing of unsealed specimen at 50 °C for 3 d ays,
- for BCSM and SCRM (cement content > 1 %): curing of unsealed specimens at room conditions for 14 days, whereas it is up to each country if additionally strength properties after 28 days are required or not.

Step 6: Mechanical tests

Firstly bulk density of test specimens has to be determined for each cold recycled mix. It is recommended to determine the bulk density close before the indirect tensile strength test is done. Because of typical air voids content usually in a broad range of 8-16 %-vol., the test procedure calculating bulk density from test specimen dimensions has to be used. Then in order to assess the mechanical properties, the indirect tense strength as well as moisture/water sensitivity shall be assessed.

Depending on the type of mixtures and the specific requirements of the given road and its pavement (e.g. traffic level – intensities, type of traffic and its typical loading, etc.), other performance evaluation tests can be important (stiffness modulus, permanent deformation, etc.). These are described separately in other reports of CoRePaSol project related to activities done within the work package WP2.



1 Introduction

The main objective of CoRePaSol project is to develop and recommend a harmonized advanced mix design procedure for cold recycled bitumen stabilized materials, to be applied throughout Europe.

Because of different existing laboratory compaction methods, the influence of miscellaneous accelerated laboratory curing procedures, the suitability of test procedures for moisture susceptibility, selection of simple but best predicative test methods for strength properties and assessments of other performance related characteristics as well as the evaluation of different mix design test methods and requirements the common understanding and comparability is difficult. The solution might be a European-wide unified design approach. The overall focus should additionally respect the availability of defined test procedures and mix design approaches to regular road laboratories which are providing their services to road administrators and/or contractors.

In order to address these issues, the first work package of CoRePaSol project (WP1 - "Advanced mix design of cold recycled bitumen stabilized material") has focussed on the assessment of available mix design procedures, test methods and specifications (standards) for use on cold recycling in pavement rehabilitation and construction. WP1 was structured into five tasks, as follows:

- Task 1.1: Review on specifications and international literature on mix design, comparison of mix designs applied internationally.
- Task 1.2: Selection and characterisation of materials.
- Task 1.3: Comparison of compaction and curing methods.
- Task 1.4: Cold recycled mixture characteristics and performance.
- Task 1.5: Harmonized advanced mix design procedure.

In this context, one of the first activities of WP1 (Task 1.1) consisted on collecting world-wide information about mix design procedures with emphasis on European countries. Within this scope, questionnaires have been issued to gather information about common practice on cold-recycling, used laboratory test procedures and available standards and legislation. Through these questionnaires, information has been collected from several countries which the partners have been in contact with, given that, apart from their own national responses, each partner has tried to gather information from at least one additional country. In order to obtain a more widespread international overview, information has also been collected through the following initiatives: review of international existing literature on the field of cold recycling (e.g. from recent research projects or from other technical documents); internal workshop on "mix design", on April 2013.

Based on the collected information, deliverable D1.1 was issued. That report comprised a synthesis on the available mix design procedures, as well as a comparison of used test methods and specified requirements. Differences were analysed in terms of the following aspects:

• Type of cold recycling techniques generally used (e.g. type of application and materials used);



- Requirements on reclaimed pavement materials used in cold recycled bitumen stabilised materials;
- Characteristics of bituminous binders as well as of hydraulic binders (including cement) used in cold recycled bitumen stabilised materials;
- Typical compositions of cold recycled bitumen stabilised materials;
- Laboratory test procedures applied for mix design (e.g. type of test specimens, compaction methods, curing procedures, performance test methods);
- Used standards and references.

On Deliverable D1.1, besides the results obtained within task 1.1, first experimental studies carried out in the framework of tasks 1.2, 1.3 and 1.4 were also presented, thus providing already information about some compaction and curing methods comparative studies, as well as about cold recycled mix characteristics and performance behaviour.

Along with WP1, others WP of CoRePaSol project were developed, in which aspects such as performance related properties and its evolution with time were further addressed. Therefore, additional studies on the stiffness modulus and the moisture susceptibility of cold-bitumen stabilized materials are included in other separate reports, e.g. report D2.1.

The purpose of Deliverable D1.2 is to provide advanced recommendations for a harmonised mix design procedure, addressing the following key issues: mixtures and test specimens preparation, curing procedures, performance related test methods as well as mix design procedures.

Within this scope, various aspects of cold recycling, from characterization of milled materials to formulation of cold recycled mixes and their properties were addressed through experimental studies. Besides indirect tensile strength, other performance related characteristics, such as stiffness modulus of cold recycled mixes manufactured either with bituminous emulsion or with foamed bitumen were evaluated under various curing conditions. The impact of the addition of different quantities of cement was also assessed.

Assessment of performance based characteristics commonly present for hot mix asphalt according to EN 12697-parts and for hydraulically bound and unbound materials according to EN 13286-parts represent key part of functional mix design but are in use for cold recycled asphalt materials only in limited extent and only in some countries. They should be taken into consideration if cold recycled mix design is fully based on hydraulic binder. Therefore, in this project, experimental studies using those standards were also performed whenever it was considered suitable and feasible. When required, some adjustments to the standards were performed in order to comply with specific characteristics of cold recycled mixtures.

For planned and extensively analyzed experimental studies, various sets of laboratory designed cold recycled mixtures have been produced and tested, in order to cover all key technical options. Taking into account the results achieved in Task 1.1 (Deliverable D1.1), research studies had focused on the following two main types of cold recycled mixtures:

 Cold recycled mixes produced with bituminous binders (bituminous emulsion or foamed bitumen) and no or only small amounts of hydraulic binders (cement content not exceeding 1 %);



• Cold recycled mixes produced with combinations of bituminous binders (bituminous emulsion or foamed bitumen) and hydraulic binders (cement content higher than 1 %).

Additionally, some experiments were carried out on reclaimed material containing not only recycled asphalt pavement layers/mixtures, but also recycled unbound or hydraulically bound mixtures (cement-based materials).

In following chapters, first a synthesis of the main conclusions achieved in the framework of Task 1.1 of project CoRePaSol are given, subsequently chapters 3 - 7 are dedicated to present the extensive experimental studies that were undertaken within Tasks 1.2 to 1.4, and in last two chapters recommendations on fundamental technical aspects (mixing, compaction, curing, and laboratory test methods for cold recycled mixtures) are provided and a proposal on a harmonised mix design procedure is presented (Task 1.5).



2 Synthesis of mix design approaches for cold recycled bitumen stabilised materials

2.1 Cold-recycling techniques

From the previous research carried out within CoRePaSol project (Project Report for Deliverable D1.1) the following conclusions were drawn:

- Most of the considered countries perform mainly in-situ cold-recycling, generally in excess of 90 % (more flexible, time-saving, more cost-effective);
- In general, cold-recycling is commonly applied to a wide range of pavement layers, from sub-grade material to bituminous surface. Therefore, reclaimed asphalt (RA) used in cold recycling can include materials originating exclusively from the asphalt layers, or being a mixture of these with materials from unbound granular layers or even cement/lime stabilized materials;
- The large majority of cold recycled bitumen stabilised materials are applied in base or binder courses;
- With respect to bituminous binder, bituminous emulsions and foamed bitumen are used by all addressed countries, except by Finland, Norway and Sweden (not using emulsion) and Portugal and Spain (not using foamed bitumen);
- As secondary binders, cement and hydrated lime are commonly used for most countries. On the contrary, Finland and Sweden don't use any of these, but report the use of corrective materials (aggregates, i.e. gravel). At any case, usually there are efforts to use other mineral binders (fly-ash, dust filler - especially if containing calcium, slag). Besides, it is worth mentioning that other countries also use other corrective materials besides cement and lime (for instance, natural aggregates / filler to adjust the grading curve);
- Some countries reported the use of additives such as foaming agents, adhesion promoters and/or fly-ashes.

2.2 Materials used and typical compositions

Gathered information showed that generally there are requirements for particles size distribution of reclaimed asphalt material or for the final granular mix composition. Nevertheless, requirements on its quality are rather unusual and hardly possible, since this material will be always classified as heterogeneous (effect of milling different layers together, impact of local repairs during the pavement life-time, effect of possible overlays, *etc.*). From the perspective of securing more homogeneous material, the only way is to require selective cold milling, which can in general be done; nevertheless it is only applicable if one separate layer is recycled in situ or if the material would be used in a mobile plant for producing high quality cold recycled asphalt mix. In general selective cold milling always leads to increased construction costs, whereas it is expected that the milling costs are raised by 25-30 %. It is then the solely decision of the road administrator if selective milling creates to him sufficient added value for cold recycling technique.



Figure 1 presents recommended grading envelopes for cold bitumen stabilised mixtures according to Wirtgen Cold Recycling Manual (2012). Figure 1 shows further typical reclaimed asphalt grading curves as well.



Figure 1: Recommended grading envelops for bitumen stabilisation (Wirtgen, 2012)

Generally, requirements for the material to be cold recycled using bituminous binders (alone or in combination with hydraulic binders) consist on using a well-graded material that should fit grading envelops such as the ones presented in Figure 1. This aims to ensure sufficient cohesion of the mix, to meet voids content requirements and to obtain the required strength for the mixture.

Usually, additional imported material (e.g. crushed aggregates/filer) needs to be added to the reclaimed material, in order to achieve the desired grading curve, fitting a given envelope. As stated before, natural aggregates / filler are typically used as corrective materials with the purpose of adjusting the grading curve. Other corrective materials such as cement or lime are also added to the reclaimed asphalt, so that the final granular mixture presents the desired characteristics. That is because the addition of cement or lime can also aim at increasing early age stiffness of the recycled layers and to decrease both its water sensitivity and curing time, especially when the reclaimed material includes unbound granular layers (Mollenhauer *et al.*, 2011b).

Another solution could be to increase thickness of recycling in terms of incorporating also a portion of the bottom base layers (i.e. unbound granular material). This however can lead to the presence of cohesive materials (e.g. clay) in the reclaimed asphalt, which should be avoided. In this case, it is preferable to incorporate new materials. For in-situ recycling, that can be made by pre-spreading those materials as a layer on the surface before recycling (Wirtgen, 2012). This has, for instance, been done on one of the trial sections monitored within the CoRePaSol project in Finland.

With respect to the typically used binders, gathered information shows that:

• **Bituminous emulsion**: Slow setting bitumen emulsions produced with at least 60% of paving grade bitumen (C60B5 according to EN 13808:2013, which comprises both former C60B6 and former C60B7 in agreement with EN 13808:2005) are the most popular. In some countries, such as Norway, the use of medium setting bituminous



emulsions with modified binders (C60BP4 and C65BP4, which correspond to former C60BP5 and former C65BP5, respectively) is also allowed. In Czech Republic, bitumen emulsions produced with polymer modified bitumen (C60BP5 and C65BP5, which correspond, respectively, both to former C60BP6 and former C60BP7 and to former C65BP6 and former C65BP7) are also used for partial recycling if required by the mix design and the purpose of the cold recycled material in the final pavement (in-plant recycling).

- **Foamed bitumen**: A wide range of bituminous binders (e.g. 50/70, 70/100, 100/150 or 160/220 paving grade bituminous binders according to EN 12591:2009) can generally be used to produce foamed bitumen. Climatic conditions have a major influence on the selection of the bituminous binder grade. In practice, Southern European countries generally use harder grades and Northern countries softer bituminous binders.
- **Cement**: Majority of countries require Portland cement (CEM I according to EN 197-1:2011) or Portland-composite (e.g. slag or limestone) cement (CEM II) to be used in cold recycling. For high cement contents, the use of low resistance cement class (with less heat of hydration) may be recommended in order to minimize the occurrence of shrinkage. As such, it is usually recommended to use 32.5 MPa cement strength class. However, CEM I 32.5 is not marketed or even produced at the present in most countries, being then replaced by 42.5 MPa resistance strength Portland cements (CEM I 42.5) or, as an alternative, Portland-composite cements (CEM II 32.5).
- **Special hydraulic road binders**: Some countries, such as Czech Republic and Germany, have specifications for hydraulic road binders (HRB) other than cement (e.g. lime or slow hardening hydraulic binders) to be used in road paving construction. In this case, 32.5 MPa or lower resistance class is recommended.

Additionally it is possible to use also other alternative binders or mineral additives, such as slag or fly-ash. Other ongoing research activities are looking in the opportunities to activate dust particles gained during aggregate production or use pulverized recycled concrete (especially its fine particles with increased content of hydrated cement mortar). From the international technical audience, it is even known that tests and studies have been made to evaluate the potentials for using geopolymers as another alternative for this type of mixtures.

As regard binder contents, collected information shows a geographical distribution among European countries, with Central European countries using bituminous binders combined with relatively high contents of hydraulic binder ($\approx 3-5$ %), and Southern countries (e.g. Portugal and Spain) using only bituminous emulsion or emulsion combined with low content of hydraulic binders (≤ 1 %). Some Northern countries don't apply cement at all. The use of bituminous binders together with moderate/high cement contents is related to climatic conditions of lower temperatures and relatively high humidity, which are favourable for the use of hydraulic binders, combined with the opportunity of using cold recycling for increasing bearing capacity of base/binder layers. Of course during cement hardening the temperature should not reach values below 0°C to avoid freezing of the water and later cracking or frost heaves. The hydraulic binders are therefore required to bind the emulsion water after breaking of the bituminous emulsion. Especially in moist regions, the base layers otherwise don't have the possibility to dry and gain their strength. In Southern Europe the dryer and



warmer climate usually allows the drying of the cold-recycled layer. However this can take several months (Batista and Antunes, 2005). On the other hand, in northern countries, the flexible nature of the pavements with the ability to endure frost heave in winter times require flexible base courses without rigid properties introduced by hydraulic binders.

These differences in applied binder contents and in the traditions in many countries result in different types of cold recycled material approaches. One approach is to increase the bearing capacity by high contents of cement binder in the cold recycled layer, resulting in hydraulically dominant mix properties. By adding bituminous binder the flexibility of this material can be increased. By means of high binder contents, a complete covering of the reclaimed granulate material is reached, which enables the recycling of environmental hazardous (e.g. tar-containing) road materials. On the other hand, the addition of lower bitumen contents (residual binder content $\leq 3 \%$, i.e. low to moderate bitumen contents) hardly changes material nature and usually results in discontinuously bond materials which are referred to as "Bitumen Stabilised Materials (BSM)", which combines the performance of flexible bound and unbound as well as rigid pavements influenced by the binder contents.

The effect of variation of bitumen and cement content on the mechanical properties can be best explained by Figure 2 which allows the classification of BSM as well as other cold recycled materials (Collings *et al.*, 2009).



Figure 2: Conceptual behaviour of pavement materials (Collings et al., 2009)

These differences in mix design approaches also result in considerably varied experiences with cold recycled materials within Europe. During Direct-Mat project, a total of 21 case studies on cold recycling procedures were gathered (Mollenhauer *et al.*, 2011). The applied binder contents vary considerably throughout the collected case studies - though some general trends can be observed (Figure 3):



- a. In case studies from Sweden no cement and low bitumen contents (≤ 2 %) were applied;
- b. Case studies from Spain and Portugal are characterised by moderate bitumen content (2 3 %) and low cement content (≤ 1.5 %);
- c. In case studies from Germany, Poland and Slovenia moderate bitumen contents were applied (2 3.5 %) combined with moderate/high cement contents (2 4 %).

Both previous *a.* and *b.* cases can be seen as typical BSM.

These different experiences were taken into account when comparing the applied mix design approaches and when drawing up recommendations for a harmonized design procedure for this type of rehabilitation technique.



Figure 3: Binder contents of cold recycling case studies gathered during Direct-Mat project

Grilli et al. (2012) define four kinds of cold recycled materials:

- Cement-treated materials (CTM) without any bitumen;
- Bitumen stabilised materials (BSM) which contain bituminous emulsion or foamed bitumen as bituminous binder (residual binder content ≤ 3 %) and may contain some cement binder as active filler (≤ 1 %);
- Cement-bitumen-treated materials (CBTM) with residual bitumen contents ≤ 3 % and cement contents ≤ 2.5 %;
- Cold asphalt mixtures (CAM) with high bitumen contents (≥ 3 %) and possibly cement as active filler.

Within CoRePaSol project, similar classification of cold recycled mixes was proposed, considering:

Bitumen stabilised materials (BSM) with bituminous emulsion (BE) or foamed bitumen (FB) contents limited to maximum values (e.g. residual binder content < 3.5 % and cement binder as active filler (< 1.5 %), as similarly defined by Grilli *et al.* (2012);



 Bitumen-cement stabilised materials (BCSM) with bituminous emulsion (BE) or foamed bitumen (FB) contents limited to residual binder content < 3.5 % and cement binder content (≥ 1.5 %).

Figure 4 shows cold recycling concepts as defined by Grilli *et al.* (2012) in combination with the different binder contents applied in selected European countries according to data collected previously (Deliverable D1.1).

From Figure 4 it is further evident, that the binder contents applied in Europe show some deviation from the cold recycling concepts as defined by Grilli *et al.* (2012). Only the mix specifications as applied in Portugal, Spain, Sweden and Norway can clearly be classified as bitumen stabilised material (BSM) according to Grilli *et al.* (2012). For the other countries, higher contents of cement and/or of residual bitumen are applied, which cannot be always assigned exactly to cold recycling types defined by Grilli *et al.* (2012).



Figure 4: Different binder contents of European countries

These differences may result to some extent from historical reasons. For instance, in Germany cold recycling technique is used for recycling tar containing pavement layers. Therefore, higher bituminous binder content is provided to ensure a complete coating of these aggregates and therefore, a feasible immobilization of that hazardous material. On the contrary, in Portugal or Spain coal tar had barely been used in asphalt pavement applications, even during World War II, thus the use of higher bituminous binder content was



not enforced. Nevertheless, for some countries, if tar has not been used another potential application for immobilizing effect might be seen for asbestos.

It is therefore difficult to establish different categories for cold recycled materials, allowing their clear distinction according to the resulting mechanical material properties. These differences demand for varying mix design procedures as will be discussed in following sections.

2.3 Laboratory test procedures applied for mix design

2.3.1 Introduction

After establishing a suitable granular composition (reclaimed asphalt + corrective materials if required) and selecting bituminous binder to be used, mix design procedures comprise:

- Determination of water content for optimization of the mix workability and the layer compaction during construction;
- Determination of optimum residual bitumen content (or required bituminous emulsion/foamed bitumen content) in order to ensure the desired mix stability and strength during the pavement's service life.

Commonly for all internationally applied mix design specification, the optimal water content for enabling feasible workability is estimated according to laboratory Proctor test results (Batista *et al.*, 2012; Wirtgen, 2012). In contrast, there is no consensual method for detecting the optimum residual bitumen content.

Even though the selection of the bitumen content being usually based on the mechanical properties of the mixture, the correspondent test procedures differ considerably worldwide, namely with reference to compaction methods and specimens dimensions, curing procedures, testing conditions and required quality parameters. This prevents any simple comparison between measured values on apparently similar test specimens. Nevertheless, the analyses of gathered information allow for the following generic findings:

- **Compaction methods**: Several countries perform static compaction procedures to obtain cylindrical test specimens of different dimensions. Some countries are using gyratory compaction, which procedures are described in an existing European standard for hot bituminous mixtures. It is worth mentioning that although the type of compaction may be the same, there are still some differences from country to country, since the referred compaction methods are performed in different ways (e.g. applied load pressure, loading time, loading procedure, number of revolutions, *etc.*).
- Specimens' dimensions: There is a significant difference between dimensions of cylindrical test specimens among each country, not only in terms of the diameter (Ø) of test specimen (i.e. Ø ≈ 100 mm, Ø ≈ 120 mm and Ø ≈ 150 mm), but also on the ratio between its diameter and height (h/Ø = 1; h/Ø < 1; h/Ø > 1). This might even differ between bitumen and cement dominated mixture types, even within the mix design specifications of the same country.
- **Curing procedures**: There are again significant differences between curing procedures across countries, in terms of number of days (covering a range between 3



days and 28 days, with 7 days and 14 days being popular), conditioning temperature (from 5 °C to 50 °C, passing through temperatures around 20 °C) and conditioning relative humidity (some countries not having this requirement and others specifying values from 40 % to 95 %, or even 100 % in the case of sealed specimens). The specified curing times of 7, 14 and 28 days seem to be related with common curing times from cement-based materials (cement concrete).

Mechanical evaluation (testing conditions and quality characteristics): The existing practice focuses mainly on the effect of water on the mixtures (whether based on indirect tensile tests or compression tests), strength (either indirect tensile strength or compression strength) and stiffness (repeated indirect tensile tests). Even though the mechanical type of testing could be the same, the obtained results can vary significantly depending on the test conditions applied (temperature, loading rate) as well as regarding the specimen preparation procedures as compaction, curing and conditioning. In some sporadic cases, additional performance-based properties are evaluated, such as: a) Shear strength properties determined by triaxial test in South Africa, as supported by research studies (Collings & Jenkins, 2008); b) Fatigue resistance, that was investigated by two-point bending beam tests (2PBT) within SCORE project (which reported some problems during specimen preparation), by four-point bending beam tests (4PBT) - for which some problems are also reported and by indirect tension tests (ITT) within some other research studies (Chamot & Romero, 2009), by indirect tension to cylindrical specimens (IT-CY) tests within Portugal research studies (Batista, 2004) and by indirect tension tests within the UK research studies (Khweir et al., 2001; Fordyce & Khweir, 2002).

There are several possible explanations for the high variety of cold recycled mix design approaches around European countries, but historical reasons seem to play an important role on this. For instance, some countries are already adapting existing European standards for hot mixtures (such as the ones on water sensitivity) to cold mixtures testing, but others keep using other standards to perform tests.

2.3.2 Compaction procedures

In order to compact cold recycled materials in which hydrostatic pressure plays an important role, the majority of European countries apply either static compaction or gyratory compaction.

Static compaction is a relatively quick and simple method used in Czech Republic, France (together with gyratory compaction), Germany, Norway, Portugal, Spain and other European countries. The procedures used can however vary a lot, as follows:

 In Czech Republic cylindrical test specimens of 150 mm in diameter are compacted in accordance with technical specifications TP 208, by two pistons moving against each other. The height of the specimens used for further strength testing should be at least 125 mm, especially if used for compression strength assessment. The required slenderness ratio, as known more from soil or concrete testing, has to be at least 0.8.



To allow drainage of water, which is released during compaction, steel back plates at bottom and top of the compacted specimens have slots (see Figure 5).

The specimens are compacted by applying an axial pressure of 5.0 MPa (88.5 kN for specimens of 150 mm in diameter). During the compaction it is necessary to compensate repeatedly the axial force until reaching the state when the power is stabilized for 30 seconds at the value of approx. 88.5 kN for 150 mm diameter specimens. According to the experience gained during many field applications of cold recycling in the Czech Republic the final tension is usually reached after 6 to 8 cycles depending on the moisture content and character of the cold recycled mix (used binders, used RAP and its grading).



Figure 5: Drainage slots in the bottom plate of a mould

In France, the Duriez method has been used in the design of cold recycled mixes, in accordance with the French standard NF P 98-251-4. This method comprises the preparation of cylindrical test specimens of 80 mm or 120 mm diameter (Figure 6) by applying a static pressure for 5 minutes. Two different modes of loading may be applied depending on aggregate maximum size (< 14 mm or ≥ 14 mm) and mould dimensions. In its original version, compressive loads of 60 kN or of 120 kN are issued, but some research results and site experiments have concluded that moulding forces of 20 kN and 40 kN (respectively for D < 14 mm or D ≥ 14 mm) would better reproduce on site densities immediately after field compaction (Serfass *et al.*, 2009).





Figure 6: Mould and compression piston for cold mixtures with bitumen emulsion (D ≥ 14 mm), according to Duriez test method [Source: NF P 98-251-4:1992]

On the other hand, it should be noted that in France, besides the *Duriez* test for water sensitivity evaluation, compaction tests are usually performed using the French Gyratory Shear Compactor (GSC), which will be further addressed in following paragraphs.

According to the German procedure of cold recycled mix compaction (DIN 1048 standard), a static axial pressure of approx. 2.8 MPa (49 kN for specimens of 150 mm in diameter) is applied by a double piston. The plastic behaviour of the cold recycled mixture leads to a stress depression and therefore it is necessary to apply 5 to 7 loading cycles until reaching the final pressure of approximately 2.8 MPa and 2.6 MPa for maximum and average values, respectively (Figure 7). In this respect, the method is therefore similar to the procedure applied in Czech Republic. One advantage of the method is the applicability on the construction site by using of standard loading devices as also applied in bearing plate loading tests.

In this case, test specimens of approx. 150 mm in diameter and about 125 mm in height (h/ $\emptyset \approx$ 80-90 %) are usually prepared.





b) Load-time-curve at static pressure (M KRC)

- According to the Norwegian methodology, one possibility is to use a static pressure of 4.5 MPa applied for 120 seconds.
- Static compaction with double plunger is similarly used in Portugal and Spain. In these • Southern European countries, laboratory test specimens for emulsion cold mix designs are usually prepared using a static compaction method based on the Spanish specification NLT 161 (which was adopted from ASTM D1074). According to this standard, the moulding of bituminous test specimens comprises firstly an application of an initial compression of 1 MPa, in order to slightly settle the mixture into the mould. Then, the definitive compaction of the mixture takes place by applying a full double-plunger action, with an increasing load pressure (with a velocity as uniform as possible) in order to reach the entire moulding load of 21 MPa (170 kN for specimens of 101.6 mm in diameter) in 2-3 minutes, and maintaining this maximum load for further 2 minutes (Figure 8-a). However, some research studies have shown that the standard pressure led to higher densities than the ones achieved in the field (Batista, 2005; Martínez et al., 2007). Comparative studies on the same type of cold recycled mixes have led to the recommendation that compressive stress loads from 7-8 MPa (resulting in compressive loads of approx. 60 kN for 100 mm diameter specimens) should be applied in order to obtain densities of the same order as typically obtained in situ (Figure 8-b).

Both NLT 161 and ASTM D1074 present the same dimensions for standard test specimens (cylinders of 101.6 mm in diameter and in height, i.e. slenderness ratio of 1), allowing cylindrical specimens of dimensions other than 101.6 mm, providing that the relation between its diameter and height remains the same ($\emptyset \approx h$) and its diameter is not less than four times the nominal size of the largest aggregate particles.





Figure 8: Static compaction based on ASTM D 1074 / NLT 161 standards: a) using standard load pressure; b) using a lower compaction pressure

As stated before, countries like France, Ireland, Norway and Spain have already begun to use **gyratory compaction**, as it has become more popular in last two decades. Gyratory Shear Compactor (GSC) is used for testing of some mechanical properties and for determination of volumetric properties of the compacted specimens (e.g. voids content and bulk density). The principle of the gyratory compaction is based on the combination of a static compression on the sample and a shearing action resulting, respectively, from a constant axial force and from the motion of the axis of the sample (Figure 9).





Some of the procedures that comprise gyratory compaction are the following:

In France, the GSC (PCG - Presse à Cisaillement Giratoire) has been used (in accordance with the French standard NF P 98-252 or, more recently, the European standard EN 12697-31) for optimizing the granular composition and approximate the percentage of voids that will be obtained on site for conventional bituminous asphalt mixes (i.e. HMA - Hot Mix Asphalt). This is not the case of emulsion stabilized graded aggregate layers, since it is considered that GSC test does not realistically reproduce field densities for this type of materials (Serfass *et al.* 2009; Olard *et al.*; 2009). Nevertheless, according to Serfass *et al.* (2009) "GSC is still considered as an useful



tool for comparing and optimizing mixture formulas in terms of aggregate gradation, binder and moisture contents";

In Ireland, laboratory test specimens for cold recycled BSM design are usually prepared using gyratory compaction, which is conducted in accordance with EN 12697-31 standard. As referred before, the procedure uses the kneading motion in the compaction process, whereby a simultaneous static compression and shearing force, resulting from the rotation of the top surface of the mould, is used to compact the mixture. The static compaction pressure is set at 0.6 MPa with an angular velocity of 30 gyrations per minute and the gyratory angle set at 1.25°.

Due to the large nominal size of the aggregates in cold recycled mixtures, moulds with a diameter of 150 mm are used for the production of test specimens. Also due to the high fluids (i.e. water) content <u>slotted moulds are advised to be used</u> for the compaction of the cold asphalt mixes. The slotted moulds incorporate narrow (approximately 1 mm) slots running vertically up and down the mould (Figure 10).

For the cold asphalt mixtures the compaction moulds don't have to be pre-heated to an elevated temperature prior to the compaction, but instead should be maintained at ambient room temperature.



Figure 10: Gyratory compaction method used in Ireland: a) Coopers Technology gyratory compactor at UCD; b) 150 mm diameter slotted moulds

The concept of using slotted moulds in order to drainage fluids (mainly water) is similar to the perforated moulds with annular inlets not larger than 2 mm in diameter used by other countries, such as Czech Republic (see Figure 5). In France on the other hand the moulds are fitted by minimized annular inlets in the shell of the mould.

- Norwegian methodology prescribes compaction with 1° angle of gyrations at a static pressure of 600 kPa and an angular speed of 30 gyrations per minute. Usually one set of specimens is prepared – test specimens compacted at number of gyrations that gives 96 % of the density of 200 gyrations.
- Some Spanish research studies (Martinez *et al.*, 2007) refer that gyratory compaction (600 kPa and 1.25°, 30 rpm) is suitable to obtain cold recycled mixtures (with bituminous emulsion) test specimens with equivalent densities to the ones obtained by static compaction (60 kN on 100 mm diameter specimens).



In other Spanish research study (Guisado *et al.*, 2011), perforated moulds (Figure 11) were used to compact cold recycled mixtures, using bituminous emulsion as binder, by the gyratory compaction method (600 kPa, 1.12 mrad, 180 gyrations).



Figure 11: Mould used for gyratory compaction of cold recycled mixtures (Guisado et al., 2011)

Still in respect to gyratory compaction, it should be noted that there are more factors influencing cold recycled mixtures compaction than in the case of HMA. For instance, the compaction of cold recycled mixtures is strongly affected by its water content. In fact, the water drainage during compaction usually prevents an accurate determination of the mix composition. Other factors, such as the type and content of the used binder as well as the aggregate properties also play important roles concerning GSC tests. In addition, a good correlation between cold recycled mixtures densities of specimens produced in laboratory and *in situ* compacted layers is generally very difficult to find.

With regard to **impact compaction**, the focus is essentially concentrating on Marshall and Proctor compaction. The first decided as the only compaction method for cold recycled mixes in Poland, the latter one then used in Finland.

Despite of **Marshall compaction** being one of the most worldwide used methods for preparation of HMA specimens, it is not so popular for cold mixtures. There are similar standards specifying this type of impact compaction method (e.g. EN 12697-30, ASTM D6926 and AASHTO T245). According to this type of compaction, a given number of blows (typically 50 or 75), delivered by a compaction hammer, is applied on both faces of cylindrical specimens. In some countries, such as Portugal, Czech Republic or Germany the use of Marshall impact compaction for cold recycled materials is faced with some reservations. One reason for this is due to the presence of fluids (water + bituminous emulsion) in cold mixtures composition during compaction, thus requiring a compaction method that releases hydrostatic pressure, which is essentially achieved by water drainage. Some of the previous research studies showed that Marshall compaction could lead to loss of fines entrained in water "splashes", resulting in high variability of specimen's densities and in lower levels of compaction (Batista, 2004).

Conversely, the **modified Proctor compaction** is a common procedure to determine the optimum water content of bitumen stabilized materials (BSM). The main reason for this is



related to the fact that, during compaction, such materials act as unbound stabilized materials, presenting in some extend a similar behaviour to granular materials, where shear properties play a stronger role. In fact, this type of compaction is worldwide used for determining the optimal moisture content and the correspondent maximum dry density of soils and aggregates. This test generally consists of compacting unbound materials into a standardized cylindrical mould using a given compaction effort for different levels of moisture content. Usually the unbound material is compacted into the mould to a certain amount of equal layers (5 for the modified Proctor test), each receiving a number of blows from a standard weighted hammer at a specified height. Currently, the procedures and equipment details for the modified Proctor compaction test are specified in the European standard EN 13286-2 (which also comprises the original Proctor test, that is commonly known as the "standard Proctor compaction test"), as well as in other standards, such as ASTM D1557 and AASHTO T180.

In addition, Proctor compaction "modified" version is often used as a complementary compaction method for an initial estimation of optimal water content, which later on is adjusted on field (Batista *et al.*, 2012). Furthermore, this type of compaction is also generally accepted outside Europe as best practice test to achieve a reference density for BSM (Collings *et al.*, 2009; Wirtgen, 2012).

In light of the above, both static compaction with double plunger and gyratory compaction are considered to be suitable methods in Europe for cold recycled and stabilized materials, and were further addressed within CoRePaSol extended experimental study. Comparison tests between the referred compaction methods were carried out using the same materials and mix composition.

Since Proctor compaction is generally assumed as being a useful tool to complement laboratory design of cold recycled mixtures and for comparing the results with other applied methods mentioned before, a comparison of these compaction methods was also performed within the present project. On the other hand, it will be recommended not to use impact compaction for standard Marshall specimens since no reliable correlation was found with specimens compacted by the other methods.

2.3.3 Test specimens dimensions

An overview about the dimension of cylindrical test specimens used in cold recycled mix design is given in deliverable D1.1 (Chapter 2). The data table highlighted the existing differences across European countries both in terms of specimen diameter (\emptyset) and in terms of the slenderness ratio between its height and diameter (h/\emptyset), as summarized below:

- ø ≈ 100 mm, h ≈ 50-60 mm ⇒ h/ø = 0.50-0.60 (Norway);
- $\emptyset \approx 100 \text{ mm}, h \approx 100 \text{ mm} \Rightarrow h/\emptyset \approx 1.00 \text{ (Portugal & Spain);}$
- $\phi \approx 120 \text{ mm}, h \approx 135 \text{ mm} \Rightarrow h/\phi = 1.10-1.15$ (France for Duriez method);
- $\emptyset \approx 150 \text{ mm}, \text{ h} \approx 70-75 \text{ mm} \Rightarrow \text{h/}\emptyset = 0.45-0.50 \text{ (France & Ireland for gyratory);}$
- $\phi \approx 150 \text{ mm}, h \approx 125 \text{ mm} \Rightarrow h/\phi = 0.80-0.85$ (Czech Republic & Germany).



The minimum dimensions of the test specimens are usually related to the maximum particle size of the granular material (D) used in the mix, as follows:

- NLT 161 Spanish standard (used in Portugal and Spain for static compaction) establishes that the diameter shall not be less than four times the nominal diameter of the largest aggregate particle size, i.e. ø ≥ 4 x D; and refers that generally test specimens are cylinders of 101.6 mm diameter.
- Czech technical specifications TP208 prescribes only test specimens of 150 mm diameter and a thickness of 125 mm to reach a suitable slenderness ratio. Test specimens with smaller diameter are not allowed so far and at the same time thinner specimens were not required since stiffness assessment was not standardized for cold recycled mixes in the Czech Republic. It is worth mentioning that if stiffness determination will be introduced it will be probably necessary to limit the specimen thickness by maximum 65 mm, due to equipment restrictions.
- EN 12697-34 specifies that the maximum aggregate size of the mixtures for specimens of 101.6 mm in diameter and 63.5 mm in height shall not exceed 22.4 mm, i.e. ø ≥ 4.5 x D and h ≥ 2.8 x D.

Generally, asphalt laboratories are equipped for testing Marshall specimens, i.e. specimens of about 102 mm diameter and 64 mm height ($h/a \approx 60-70$ %). However, grading of reclaimed asphalt for new mixtures can frequently exceed 0/22 mm going up to 0/45 mm grading. In this case, a diameter of 150 mm is recommended. Further, the placement of the material in the mould plays an important role in preparing suitable specimens to be used in mechanical tests. Especially large aggregates at the surface of the specimens may affect the stress distribution and deformation of the specimen. Another possibility to improve the specimen homogeneity applied in laboratory is to remove the large aggregates (e.g. > 16 mm) by sieving. The grains of the referred coarser aggregates have comparably small surface area in respect to mass and therefore its removal possibly will not significantly affect the mix properties. Nevertheless, the implications of the removal of these grains have not been systematically researched yet.

Currently, gyratory compaction is becoming also available in more pavement materials laboratories, in which case test specimens of 150 mm in diameter and about 125 mm in height (h/ ϕ = 80-90 %) are usually prepared.

Taking into account this information, in the experimental studies, besides local special specimen dimensions, the following dimensions were addressed when considered appropriate and feasible:

- $\emptyset \approx 102 \text{ mm}, h \approx 64 \text{ mm} \Rightarrow h/\emptyset = 60-65 \%$
- ø ≈ 150 mm, h ≈ 95 mm ⇒ h/ø = 60-65 % or
 ø ≈ 150 mm, h ≈ 125 mm ⇒ h/ø = 80-85 %

2.3.4 Laboratory accelerated curing procedures

After cold in situ recycling or after placing in plant produced cold stabilized mixtures in the pavement, the first phase of the life-cycle of the compacted material is characterised by the



so-called "curing" process, during which the cohesion and therefore strength and stiffness of the mixture increase. During curing, excessive water (e. g. from breaking process of the emulsions) drains or evaporates. This continuously improves the contacts between bitumen and aggregates. For mixtures containing cement, a part of the water is bound during the cement hydration process. Especially in moist / cool regions the addition of cement will accelerate the water exertion from the mix resulting in an accelerated strength increase. At the end of the curing process, a continuous cohesive film that holds the aggregate in place with a strong adhesive bond must be achieved (Asphalt Institute MS14, 1990).

In the field, the curing time at which the cold mixture attains a "stable" condition can commonly take several months, depending not only on the properties of recycled material itself or on pavement layer characteristics, but also on external conditions, such as climatic conditions and traffic level. Another factor might be the moment when the cold recycled material is overlaid by next layer. According to Serfass *et al.* (2004), in temperate climate and under medium traffic at least one complete cycle of seasons is usually necessary for emulsion stabilised cold mixes to reach such "stable" condition.

Curing of cold recycled mixtures has a great impact on the evolution of the material properties and on the performance of the entire pavement as well and consequently on the quality control procedures. Therefore, it is of major importance to correctly simulate the field curing process in the laboratory. Nevertheless, taking into account, among others, time restrictions (e.g. related to road closures) and available equipment, laboratory curing procedures should be as short as possible, but without causing any significant ageing of the bituminous binder; should reproduce as close as possible the curing stage in the field; and should employ only usual equipment, i.e. equipment not too complex (Serfass *et al.*, 2004). Moreover, laboratory accelerated curing procedures should enable the simulation of different curing stages (i.e. short and long term), in order to allow the determination of performance-related properties that properly reproduce in service pavement performance along time.

As stated before, laboratory curing procedures vary considerably across different countries, not only in terms of number of days (generally ranging between 3 days and 28 days), but also in terms of conditioning temperature (from $5 \,^{\circ}$ C to $50 \,^{\circ}$ C) and conditioning relative humidity (from no requirement to values between 40 % and 100 %). Some examples of currently applied accelerated curing procedures are given bellow:

- Czech Republic: after compaction the specimens are left for 24 hours in the mould. For later curing specimens are stored the whole curing period at (20±2) °C and 90-100 % relative humidity in case of mixtures only with hydraulic binder; for 2 days at (20±2) °C and 90-100 % relative humidity + 4 days at (20±2) °C and 40-70 % relative humidity in case of mixtures with combined binder (bitumen and cement); or simply air curing for 6 days at (20±2) °C in case of mixtures where only fo amed bitumen or emulsion is used;
- France: curing for 7 days @ 18 °C and about 50 % r elative humidity, before further testing (e.g. evaluation of effect of water on compressive strength);



- Germany: curing for 2 days @ 20 ℃ & 95% relative humidity (sealed in the mould) + 26 days @ 20 ℃ & 40-70 % relative humidity (room c onditions);
- Ireland and United Kingdom: 28 days @ 40 ℃ (for e mulsion mixtures),
- Portugal and Spain: curing for 3 days @ 50 °C (for emulsion mixtures).

In most cases, these differences are not only due to climatic conditions and other geographical specific circumstances (e.g. available materials and their typical characteristics), but also because of historical reasons. For instance, in the years 1990-2000, cold asphalt techniques became popular firstly in Spain and later also in Portugal, causing that similar or even identical mix design procedures are used by both countries since then. With regard to the mix composition, laboratory studies were carried out based on standards developed for hot bituminous mixtures, but adapted to cold mixtures that require a curing conditioning period prior to testing. In 1998, Fernández del Campo recommended that after compacting cylindrical test specimens, they were subjected to laboratory accelerated curing of 24 h at 60°C. Nevertheless, the common practice in Spain and in Portugal in the latest 90's was to perform a laboratory accelerated curing procedure immediately after compaction of "2 h in the mould at ambient temperature + extraction + 24 h at ambient temperature + 3 days in oven at 60°C". Meanwhile, several research studies took place, and for example, the carried out by Tijeda (1999) found that the resistance obtained in study "immersion-compression tests" on specimens cured at ambient temperature for 21 days was similar to the resistance obtained on specimens subjected to accelerate curing of 1 day at ambient temperature and 3 days at 50°C. As a consequence, in 2001, the Spanish Road Administration issued a new technical specification document for pavement recycling (PG4) defining that after compaction the test specimens should be submitted to laboratory accelerated curing of 3 days at 50 °C previously to "immersion-compression tests".

The analysis of the collected data allows for the following comments:

- Besides historical and geographical reasons, most of the differences among curing procedures seem to be related to the presence of cement besides bituminous binder in cold recycled mixtures, leading to higher curing times (7, 14 and 28 days), relatively low conditioning temperatures (around 20 ℃) and maximum levels of humidity (95-100%) in earlier stages of curing. In fact, cold recycled mixtures with added cement are sensitive with respect to high temperatures and low humidity levels, which may inhibit their required hydration process.
- A laboratory curing of 28 days is too long for practical reasons, mainly in the case of site-produced material. Such a long period could hamper a suitable mix design before starting in situ rehabilitation works or even a quick quality check on site.
- A laboratory accelerated curing process (such as the one used in Portugal and Spain of 3 days @ 50 °C) that adequately simulates medium/fully in service cold recycled layers curing should be considered.

It is worth to be noted, that after some initial studies within the present research project, it was agreed that prior to this accelerated curing process, compact



mixtures (especially those containing cement) should be kept in the mould, at room temperature, in order to guarantee suitable hydration of the cement by conditioning them at high relative humidity.

2.3.5 Laboratory test procedures for determination of mix composition

For enhancing cold recycled mixtures design several countries developed strategies separately from each other:

- In Czech Republic, usually requirements for cold emulsion/foamed mixtures (with or without low contents of cement) refer to the indirect tensile strength (ITS) of specimens after 7 days of curing and to their retained resistance after immersion in water for additional 7 days. In addition, stiffness has also been assessed by indirect tensile tests on cylindrical specimens (IT-CY) and by four point bending tests on prismatic specimens (4PB-PR) at various temperatures, resulting in recommended values in a range of 3,500-4,500 MPa at 15 ℃ (Valentin, 2009).
- In France, the mix design of cold emulsion stabilised materials usually relies on the following tests:
 - The CGS test, which is mainly used to optimize the mix composition (namely, its granular grading curve) as well as to supply additional information on the voids content of the compacted mixtures;
 - The Duriez test (unconfined compression test on Duriez test specimens conditioned with/without immersion), that is usually applied in order to investigate the mixture compressive strength and its water resistance, in accordance with the French standard NF P 98-251-4. A schematic representation of the Duriez unconfined compression test is shown in Figure 12.



Figure 12 – Schematic of the Duriez compression test



Typically, the Duriez test requires specimens to be conditioned at 18 $^{\circ}$ for 14 days at controlled humidity, prior to testing. While "dry" specimens are kept stored at constant hygrometric conditions (50±10 % relative humidity) during all 14 days, for saturated specimens, they are conditioned during the first 7 days similarly to dry specimens and the last week submerged in water at 18 $^{\circ}$ prior to testing, as follows:

- "Dry" specimens: 14 days @ 18℃ & ≈ 50% RH ⇒ Compressive strength @ 18℃ (R)
- "Wet" specimens: 7 days @ 18℃ & ≈ 50% RH + 7 days immersed ⇔ Compressive strength @ 18℃ (r)

The loading rate is of 1 mm.s⁻¹ and, according to the Irish methodology (where Duriez test is also used); the maximum load must be obtained in the period between 5 and 60 seconds. It is also necessary to force out water to drain. Similar approach can be found in United Kingdom.

According to this procedure, requirements for cold stabilized pavement material design rely on the strength of "dry" specimens (as an approach to the mix mechanical characteristics), and on the ratio between the strength of specimens with immersion and without immersion (r/R), which refers to mix resistance to water (mix durability). Thus, these values are checked for compliance with the relevant mix design specification.

The Duriez test is also recognised as being able to deliver similar voids content as those obtained on site immediately after construction, providing that a reduced static load (of one third of the conventional compression load) is applied during compaction of the cold-mixture (Serfass *et al.*, 2009; Claudel *et al.*, 2012; Eckman *et al.*, 2012).

The mix design of this type of cold mixtures often also comprise a manual test in which the coating quality of the granular material by the bitumen emulsion (for several total water contents and residual bitumen contents) is visually evaluated (CFTR, 2007).

Moreover, several studies have been undertaken assessing the stiffness of cold recycled mixtures. For instance, in a study conducted in the sequence of the European SCORE project, the stiffness of lab specimens produced with the same mix composition as in-situ recycled materials (either with foamed bitumen or with bituminous emulsion) using the static Duriez compaction (at two different compression loads: the conventional one and 1/3 of this), resulted in stiffness values of about 1500-4000 MPa (depending on the designed type of the recycled asphalt layer) (Eckman & Soliman, 2010).

In Germany, design studies generally comprise the strength evaluation of the cold bituminous mixtures after 7 and 28 days of dry curing (room conditions), namely by indirect tensile tests (IDT) conducted at 5 °C with continuous loading according to EN 13286-42 (Unbound and hydraulically bound mixtures - Part 42: Test method for the determination of the indirect tensile strength of hydraulically bound mixtures). Further the water sensitivity is assessed by comparing the strength obtained for the dry conditioned specimens with the strength of specimens which were transferred after 14 days of dry curing into a water bath for 14 days water conditioning.



- In Ireland, by designing cold recycled mixes indirect tensile strength (ITS) at 25°C is usually required together with stiffness assessment. Alternatively, Duriez test is done with determination of compressive strength and moisture sensitivity assessment. If Duriez procedure is not applied, the test specimens are sealed and cured at 40°C for given time period.
- As referred before, in Portugal the same procedures as in Spain are used for cold recycled mix design (either new dense mixtures treated with emulsion or recycled mixtures). In both countries, laboratory mix design studies generally consist on performing immersion-compression tests according to the Spanish standards NLT 161 and NLT 162 (based on the American standards ASTM D 1074 and ASTM D 1075, respectively), which were developed for hot bituminous mixtures. As so, some adaptations were made, in order to take into account the specificities of cold mixtures, namely, in terms of the required curing process prior to mechanical testing. At the present, both Spanish and Portuguese Road Administration specifications for road works, establish a laboratory accelerated curing procedure for cold emulsion recycled mixtures of 3 days at 50 ℃.

In broad terms, the mix design procedures comprise the following steps:

- Sample reclaimed asphalt material milled from the pavement to be recycled or similar reclaimed pavement material (containing not necessarily only asphalt layers) and assess grading of the granular material (grains of aggregate and bitumen) and bitumen content as well as aggregate grading after bitumen extraction;
- Adjust the final granulate material (reclaimed asphalt + corrective material if required) in order to meet quality requirements (e.g. grading curve, ...);
- Selection of a type and grade of bitumen emulsion;
- Preliminary evaluation of the compatibility of the granulate material with the bitumen emulsion by performing manual coating tests; this can include premixing water content determination in order to achieve a satisfactory degree of coating;
- Water content determination, regarding both the mix workability and the layer compaction, by means of the modified Proctor compaction on the final granulate material or, alternatively, on the final granulate material mixed with a pre estimated amount of emulsion;
- Bituminous emulsion (or bitumen residue) content determination in order to ensure the desired mix stability and strength, by assessing the effect of water on compressive strength of compacted cold recycled mixtures (produced with different emulsion contents), as follows:
- Preparation of cylindrical test specimens by static compaction with double plunger action (based on the Spanish Specification NLT161 or on the ASTM D1074);
- Accelerated curing of test specimens by storing them in the oven at 50 ${\rm C}$ for 3 days;



- Test specimens conditioning for water sensitivity tests:
 - "Dry" specimens: 4 days in air @ 25 ℃ + 2h in wat er @ 25℃ (or alternatively:
 24h in air @ 25 ℃ + 2h in water @ 25 ℃)
 - "Wet" specimens: 4 days in water @ 50 ℃ + 2h @ ro om temperature + 2h in water @ 25 ℃ (or alternatively: 24h in water @ 60 ℃ + 2h @ room temperature + 2h in water @ 25 ℃)
- Uniaxial compression tests (v=5.08 mm/min.) in order to determine the average compressive strength of each group of "dry" and "wet" specimens and the correspondent retained strength

Consequently, in both countries, requirements for cold recycled mixtures containing bituminous emulsion usually refer to the unconfined compressive strength of cured specimens and their retained resistance after immersion in water. Besides, it is worth to be noted that, according to the described procedure, the total time between specimens' compaction and compression testing is of 7 days, for the conditioning method commonly used.

Moreover, stiffness values in the order of 3,000 MPa at 20 $^{\circ}$ C (IT-CY tests) for cured mixtures have been reported in some research studies (Batista, 2004).

• In United Kingdom, dynamic stiffness modulus has been assessed at 20 ℃ resulting in values for the foam bitumen stabilized materials of about 1.5 GPa (e.g. Khweir *et al.*, 2001).

From the gathered data, it was possible to infer that water sensitivity tests are the most commonly specified tests to investigate the effect of bituminous binder content. This is the case of Czech Republic, France, Germany, Ireland, Portugal and Spain, where requirements for cold bitumen stabilized materials regarding mixture "dry" strength of cured specimens and their retained strength after immersion in water ("wet") are established. Nevertheless, the test procedures are distinct with respect to a number of issues, such as: specimen compaction and preparation, curing, conditioning and testing (e.g. type of tests; tests criteria, mainly temperature; etc.). With respect to mechanical strength evaluation, apart from specific methods used in some of the partners' countries, water sensitivity tests based on indirect tensile tests on "dry" and "wet" specimens were carried out within this project.

2.4 Quality control procedures

For in situ cold recycled mixtures, a significant part of the raw material is usually formed by the milled existing pavement layers, which will most likely present more variable characteristics than conventional "new" aggregates. This is due to the milling process itself and to local pavement repair treatments which possibly changes the pavement structure in the recycled road section (e.g. change in the thickness of the layers, change of used mix type). For this reason, performance related tests are recommended for quality control, in addition to the usual requirements concerning aggregate gradation, binder content, density and void content. For example: in Germany requirements on bearing capacity as well as strength tests of specimens compacted from the actually prepared mix on-site are advised; in



Portugal and Spain, compressive strength and water sensitivity resistance of cold recycled mixture is required; and in United Kingdom, requirements on indirect tensile strength, stiffness and water sensitivity are established. In Ireland, stiffness and water sensitivity tests thresholds are specified. In the case that these requirements are not accomplished, then the contractor must determine compliance by taking cores from the pavement after construction and after one year in service. Pavement thickness is measured and indirect tensile stiffness modulus (ITSM) tests conducted on all cores, from which an average stiffness value of 1.9 GPa must be exceeded. Similar criteria apply in the United Kingdom, where a minimum of one core is required per 75 m² of non-compliant material. The cores are then tested using the dry ITSM test; individual test results must exceed 2.0 GPa and the mean value must exceed 2.5 GPa.

2.5 Conclusions from review on specifications and international literature

From an analysis of the data presented in previous sections, the following conclusions can be highlighted:

- Cold recycling techniques:
 - Most countries perform mainly in-situ cold recycling, generally in excess of 90 %, which is related to the following generally recognised advantages: it is a more flexible and cost-effective solution for pavement rehabilitation reducing any additional environmental impacts or additional logistic arrangements;
 - Cold recycling is commonly applied to a wide range of layers, from sub-grade material to bituminous surface. In general it is more common to use cold recycling in full-depth option combining often asphalt layers with granular or differently stabilized materials;
 - The large majority of cold recycled bitumen stabilised materials are applied in base or binder courses.
- Reclaimed Asphalt (RA) used on cold recycled bitumen stabilised materials:
 - In general, requirements on the grading curves of reclaimed asphalt material or on the final granular mix composition are specified;
 - Therefore, trial milling on the specific site may be required in order to obtain realistic reclaimed road material for conducting the mix design study;
 - Usually, well-graded materials are considered to be suitable for cold recycling;
 - It is important to reflect possible rectifications of grading curves within the mix design. Especially for foamed bitumen stabilized mixes it is crucial to secure in the final grading sufficient content of filler particles (> 5 %) for optimal coating.
- Bituminous and hydraulic binders:
 - Most countries use both bituminous emulsion (mainly C60B5, which comprises both former C60B6 and former C60B7 according to the classification of EN 13808 before its new edition in 2013) and foamed bitumen binders (being the most popular the ones produced using 70/100 penetration grade bitumen binders, with alternatives either 50/70 or 160/220 depending on the climatic conditions);



- As secondary binders, cement (mainly, Portland cement CEM I 42.5 or Portland-composite cement CEM II 32.5) and hydrated lime are commonly used. Some potential might be identified with fly-ashes or activated mineral dust particles (back filler, or fines from aggregate or cement production);
- Some countries reported the use of additives such as foaming agents, adhesion promoters and/or alternative binders like fly-ashes.
- Typical compositions of cold recycled bitumen stabilised materials:

A geographical variation among European countries was noticed, with Central European countries commonly using bituminous binder combined with relatively high contents of hydraulic binder ($\approx 3-5$ %), Northern countries commonly using bituminous binders in form of foamed bitumen, and Southern countries using just bitumen emulsion or emulsion combined with low content of hydraulic binders (≤ 1 %).

- Laboratory test procedures applied for mix design:
 - Modified Proctor compaction is often used for estimation of the optimum water content;
 - Test procedures for investigation of optimum binder content or the influence of binder content on cold recycled mixture performance differ considerably among countries, namely with reference to compaction methods, curing procedures and testing conditions and quality characteristics. Nevertheless, the most promising methods seem to be the following:
 - Compaction methods: Static pressure and gyratory compaction, with a demand for adjusting and harmonizing compaction energy (a key aspect deeply addressed in the conducted experimental studies);
 - Curing procedures: 7 days / 14 days at room conditions for early to medium specimen age, and 21 days at room conditions (for later ages) or 3 days at 50 ℃ as an accelerated curing procedure. In this later case, with a need for investigation on its applicability not only to cold recycled mixtures produced also with small cement contents (≤ 1 %) but also to mixtures produced with moderate/high cement contents (another key aspect that was addressed in more detail in the experimental studies);
 - Testing conditions and quality characteristics: Water sensitivity tests (to evaluate the effect of water on cold recycled mixtures), indirect tensile strength tests or compressive strength tests (for strength assessment), and repeated indirect tensile tests (as a functional test for stiffness assessment – essentially discussed in WP2).



3 Experimental studies

Taking into account initial conclusions summarized in the previous chapter, experimental studies were performed in order to assess the identified aspects for further research.

According to the established work program, an extended experimental program was performed focusing on the following key aspects:

- Comparison of compaction methods, including:
 - Comparison of test specimens characteristics prepared using different compaction methods (i.e. static, gyratory, impact (Marshall) and Proctor compaction);
 - Evaluation of the influence of the compaction method on the determination of the optimum water and binder content of the cold recycled mixture. Within this scope different cold recycled bitumen stabilized mixes containing either emulsion or foamed bitumen were used. Furthermore mixes with varied combinations of bituminous and hydraulic binders were used as well.
- Evaluation of curing methods, namely in terms of time, temperature and moisture, including:
 - Comparative curing studies on mixtures with emulsion and foamed bitumen, no cement or small amounts of cement (<1.5 %) acting as a reactive filler:
 - 4 days: 24 h for demoulding + 3 days @ 50 ℃
 - 7 days: 24 h for demoulding + 6 days @ room temperature and 40-70 % relative humidity
 - 14 days: 24 h for demoulding + 13 days @ room temperature and 40-70 % relative humidity

Afterwards:

- ITS according to EN12697-23,
- Water sensitivity according to EN12697-12 and modified AASHTO T283 protocol.
- Comparative curing studies on mixtures with bituminous binder and cement (≥1.5 %):
 - 7 days: 24 h for demoulding + 6 days @ room temperature and 40-70 % relative humidity (unsealed conditions) \rightarrow verify if ITS₇ > (0.3 0.5) MPa & ITS₇ ≤ 0.75 MPa
 - 7 days: 24 h for demoulding + 6 days @ room temperature and 90-100 % relative humidity (sealed specimens) → verify if ITS₇ > (0.3 0.5) MPa & ITS₇ ≤ 0.75 MPa
 - 14 days: 24 h for demoulding + 13 days @ room temperature and 40-70 % relative humidity
 - 14 days: 24 h for demoulding + 13 days @ room temperature and 100 % relative humidity (sealed specimens)
 - 28 days: 24 h for demoulding + 27 days @ room temperature and 40-70 % relative humidity \rightarrow verify if ITS_{28} > 0.75 MPa
 - 28 days: 24 h for demoulding + 27 days @ room temperature and 100 % relative humidity (sealed specimens) \rightarrow verify if ITS₂₈ > 0.75 MPa


Additional assessments have also been done with respect to the temperature used for accelerated curing.

- Assessment of cold recycled mixture characteristics and performance, comprising:
 - Characterisation of cold recycled mixtures, including its performance evaluation. Several types of laboratory tests related to specifications in various European countries (e. g. EN 12697-series, EN 13286-series were performed in order to select the most suitable to establish the mix composition, and to assist with on-site quality control. Selection of tests accounted for their simplicity and suitability to be used at the construction site. It is expected that in most European countries, where cold recycling is used, indirect tensile strength or compressive strength is used and should be applied in the future as well.
 - Evaluation of the influence of different mixture compositions in their performance in different curing phases. Special attention was paid to retained resistance of cold recycled mixtures after immersion in water, since this characteristic is considered of crucial importance in the durability and stability of cold recycled mixes. Therefore, these tests comprised water sensitivity tests and determination of stiffness modulus.
- Sensitivity study on the influence of inhomogeneous material properties of reclaimed asphalt on mix design and performance, mainly consisting in the evaluation in what extend the variation of reclaimed asphalt pavement material properties (grading, binder content, type of material) influence the mechanical properties of the cold recycled mix.

In this context, various aspects of cold recycling, from characterization of milled materials to formulation of cold recycled mixes and their properties were addressed through a range of experimental studies. Besides indirect tensile strength, other performance related characteristics, such as stiffness modulus of cold recycled mixes manufactured either with bituminous emulsion or with foamed bitumen were evaluated under various curing conditions. The impact of the addition of different quantities of cement (depending on the common practice in different parts of Europe) was also addressed.

Assessment of performance related characteristics commonly used and promoted for hot mix asphalt, according to EN 12697 series and for hydraulically bound and unbound materials according to EN 13286 series, represent key part of a functional mix design, but so far are in use for cold asphalt recycling materials only in limited extend and just in some countries. Therefore, in the CoRePaSol project experimental studies were performed using those standards whenever it was considered suitable and feasible. In some cases, some adjustments to the standards were performed in order to attend the specific characteristics of cold recycled mixtures. It is worth mentioning, that in order to diminish the variability between experimental studies conducted in different laboratories, the mixing machine used to produce cold recycled mixtures was of the same type in all of them: a mixing unit WLM30 provided by Wirtgen company. This mixing unit has two-shaft drum mixing devices with controlled mixing speed.

In the next four chapters, the following experimental studies are described:

• Test specimens characteristics using different compaction methods (chapter 4);



- Cold recycled mixture characteristics and performance, considering the effect of different curing procedures (chapter 5);
- Evaluation of the influence of the binder type on cold recycled material performance related properties (chapter 6);
- Sensitivity studies on the influence of inhomogeneous materials properties of reclaimed asphalt (chapter 7).



4 Test specimens characteristics prepared using different compaction methods

4.1 Experimental study A1

<u>Main objective</u>: Evaluate the influence of applying a static compaction based on ASTM D 1074 / NLT 161 standards, using the standard (21 MPa) or a reduced (approx. 7.5 MPa) load pressure.

<u>Used binder(s)</u>: Bituminous emulsion with or without small amounts of cement (< 1.5 %)

4.1.1 Selected materials

The reclaimed asphalt material used in this study was originated from the milling of the upper layers of a Portuguese National Road pavement, within its rehabilitation works. The grading curve of samples of the reclaimed material (RA-S1, RA-S2, RA-S3) as well as its average (RA-Average) is represented in Figure 13. This Figure also shows the Portuguese required grading envelope for the reclaimed asphalt used in asphalt layers at least 10 cm thick (EP-CETO, 2012) and the Wirtgen Manual (2012) recommended grading envelope for cold recycled mixes with bituminous emulsion.

It is worth mentioning that both Spain and Portugal specify two different grading envelopes for the reclaimed asphalt depending on the recycled layer thickness: one (RE1/I) for applications in higher thickness layers (\geq 10 cm) and other (RE2/II) for layers' thicknesses between 6 cm and 10 cm. In each of those cases, the required envelops are very similar for the two countries.



Figure 13: Reclaimed asphalt grading curves and cold recycled mixtures envelopes

With regard to achieve a grading curve fitting the Wirtgen Manual (2012) recommended envelope, certain amount of filler was added to the mixture, as shown in Figure 14. The adopted granular mix composition comprised 97% reclaimed asphalt and 3% of filler.





Figure 14: Reclaimed asphalt, filler and final granulate material grading curves

In order to investigate the optimum water content, modified Proctor tests were performed on the granular material (Figure 15).



Figure 15: Modified Proctor compaction curve

The Proctor compaction curve is fairly atypical, what may be related both to the type of tested material (which is mainly "reclaimed asphalt"), and to the dimension of its particles (which present a relatively high content of "coarse" material).

From the results obtained, an optimum water content of 5.7% (by mass of the granular material) was considered for further testing. Furthermore, the bitumen on the reclaimed asphalt was also recovered and characterized (Table 1).



Table 1: Characteristics of the bitumen in the reclaimed asphalt

Bitumen content (% in mas	Bitumen content (% in mass)				
Properties of the	Needle Penetration (EN 1426), 10 ⁻¹ mm	12			
recovered bitumen	Softening Point by R&B (EN 1427), ºC	73.4			

In the studies developed at LNEC, a cationic slow setting bituminous emulsion (C60B5 according to EN 13808:2013, which corresponds to former C60B7) was selected. Furthermore, a 42.5 resistance strength Portland cements with rapid (higher) early strength (CEM I 42.5 R) was also used. Table 2 shows the compositions that were adopted for mix design studies.

Table 2: Used mix compositions for bituminous emulsion stabilized materials and for bitumen emulsion & cement stabilized materials

Cold recycled mix ID		CM-E3	CM-E4	CM-E5	CM-E3C1	CM-E4C1	CM-E5C1
Used mix component	Reclaimed Asphalt	91.8 %	91.8 %	91.8 %	91.8 %	91.8 %	91.8 %
	Filler	2.8 %	2.8 %	2.8 %	1.8 %	1.8 %	1.8 %
	Cement	-	-	-	1.0 %	1.0 %	1.0 %
	Emulsion	2.8 %	3.8 %	4.7 %	2.8 %	3.8 %	4.7 %
	Added water	2.6 %	1.6 %	0.7 %	2.6 %	1.6 %	0.7 %

The maximum density of each of the referred six mix compositions was determined. Table 3 and Table 4 show the results obtained, respectively for the mixtures with bituminous emulsion and for the mixtures with combined binder of bituminous emulsion and cement. Those results are graphically represented in Figure 16.

Table 3: Maximum density of bituminous emulsion stabilized materials

Cold recycled mix ID CM-E3		CM-E4			CM-E5					
Sar	nple	S.A1	S.A2	S.A3	S.A4	S.A5	S.A6	S.A6	S.A8	S.A9
Maximum	ρ _{mv,i}	2.449	2.437	2.447	2,430	2,416	2,441	2,427	2,402	2,440
density Mg/m ³	Pmu / s	2.	.444 / 0.27	78	2.	. 429 / 0.28	36	2	423 / 0.28	9

Table 4: Maximum density of bituminous emulsion & cement stabilized materials

Cold recycled mix ID CM-E3C1		CM-E4C1			CM-E5C1					
Sar	nple	S.B1	S.B2	S.B3	S.B4	S.B5	S.B6	S.B6	S.B8	S.B9
Maximum	ρ _{mv,i}	2.456	2.466	2.448	2,457	2,435	2,445	2,419	2,434	2,419
density, Mg/m ³	density, Image: Provide a start of the star		2.446 / 0.277			2.424 / 0.288				





Figure 16: Maximum density of bituminous emulsion & cement stabilized materials

Previously presented results on the maximum density of cold stabilised materials show that:

- There is some variability among the values obtained from different samples of the same mixture, which may be related with the variability of the reclaimed asphalt used to produce each sample of mixture for further testing;
- As expected, there is a tendency to a decrease in the maximum densities of mixtures with an increase of the binder content.

4.1.2 Description of the study

Initial studies on compaction method carried out at LNEC aimed at comparing the effects of using static compaction but applying a reduced compressive loading (7.5 MPa) instead of the standard one (21 MPa) established in the Spanish standard NLT 161 (which was adopted from ASTM D1074).

Once the granular material (97% reclaimed asphalt + 3% filler) has a maximum dimension of the particles of 20 mm, it allows the use of cylindrical moulds of 102 mm internal diameter (NLT 162) and of Marshall test specimens ($\emptyset \approx 102$ mm and $h \approx 64$ mm).

In summary, the comparative studies on different compaction procedures initially comprised:

Standard test method:

- Static compaction: p = 21 MPa (standard pressure);
- Cylindrical test specimens: $\emptyset \approx h \approx 102 \text{ mm}$ (standard dimensions).

Modified test method:

- Static compaction: p = 7.5 MPa (reduced pressure);
- Cylindrical test specimens: $\emptyset \approx 102 \text{ mm \& h} \approx 64 \text{ mm}$ ("Marshall" test dimensions).

In this framework, bituminous emulsion stabilized mixtures were produced, according to the compositions presented in Table 2 (CM-E3, CM-E4 and CM-E5), and compacted by the above referred procedures. Furthermore, cold recycled mixtures produced using 1 % of cement besides bitumen emulsion binder (identified as CM-E3C1, CM-E4C1 and CM-E5C1 in Table 2), were also compacted using the above referred modified test method (static compaction p=7.5 MPa; $\emptyset \approx 102$ mm & h ≈ 64 mm).



Preparation & conditioning of test specimens		Cylindrical specimens: p = 21 MPa $\emptyset \approx h \approx 102 \text{ mm}^{(1)}$ 3 days @ 50 °C					Cylindrical specimens:					
Determination of bulk density	Proce (Bulk de dimer	dure D ensity by nsions)	D Procedure B Procedure D by (Saturated surface dry, SSD) (Bulk density by dimensions)				(Sa	Procedure B (Saturated surface dry, SSD)				
	ρ _t (Mg	odm J/m ³)	ρ _t (M	ossd g/m³)	(W %)	م (M	⁹ bdm g/m ³)	ρ _t (Μ	ossd g/m ³)	, ('	w %)
	2.300		2.324		0.7		2.159		2.220		1.4	
	2.311		2.337		0.5		2.163		2.225		1.3	
CM-F3	2.304	2.306/	2.344	2.335/	0.3	0.5/	2.155	2.158/	2.220	2.222 / 0.002	1.7	1.4 / 0.2
0	2.317	0.007	2.331	0.007	0.5	0.2	2.158	0.011	2.224		1.2	
	2.303		2.336		0.8		2.173		2.220		1.4	
	2.299		2.339		0.3		2.140		2.222		1.4	
	2.308		2.324		1.1		2.164	2.165 / 0.012	2.224	2.224 / 0.005	1.1	1.1 / 0.2
	2.303		2.315		0.5		2.174		2.219		0.9	
CM-F4	2.305	2.305/	2.327	2.325/	0.7	0.7/	2.176		2.228		0.9	
0	2.303	0.004	2.331	0.005	0.9	0.2	2.144		2.224		1.3	
	2.301		2.324		0.5		2.158		2.230		1.3	
	2.312		2.326		0.4		2.171		2.217		0.9	
	2.303		2.322		0.4		2.181		2.225		0.7	
	2.298		2.306		0.3		2.184		2.231		0.8	
CM-E5	2.304	2.302/	2.316	2.316/	0.3	0.5/	2.197	2.193 / 0.013	2.239	2.232/	0.6	0.6/
CM-ED	2.294	0.006	2.320	0.006	1.0	0.3	2.216		2.239	0.006	0.4	0.4 0.2
	2.305		2.316		0.6		2.183		2.227	-	0.6	
	2.310		2.320		0.4		2.197		2.230		0.8	

Table 5: Bulk density of cylindrical test specimens of bituminous emulsion stabilized materials

(1) The main average height of compacted specimens was, in fact, of 100 cm.

(2) The main average height of compacted specimens was, in fact, of 66 cm.

4.1.3 Results

Bulk density of compacted test specimens of CM-E3, CM-E4 and CM-E5 cold mixtures was determined after 3 days curing in an oven at 50 °C. Table 5 and Figure 17 synthesize the results obtained.

The results presented in Table 5 and Figure 17 are consistent with the expected, since specimens compacted with lower pressure present lower bulk densities. In addition, lower specimens densities correspond, in general, to higher water absorptions during immersion in water (in SSD test procedure), and thus to higher differences between bulk densities determined by dimensions and by immersion in water.





Figure 17: Bulk density of cylindrical test specimens of bituminous emulsion stabilized materials

Subsequently, the air voids content of each group of specimens was calculated (Table 6, Figure 18).



Preparation & conditioning of test specimens	Loose mixture	Cylindrical specimens: p = 21 MPa ø ≈ h ≈ 102 mm ⁽¹⁾ 3 days @ 50 ºC				Cylindrical specimens: p = 7.5 MPa ø ≈ 102 mm & h ≈ 64 mm ⁽²⁾ 3 days @ 50 ºC			
Voids characteristics	<mark>ρ_{mυ}</mark> (Mg/m ³)	<mark>Øbdm</mark> (Mg/m ³)	V _{m,dm} (%)	PESSD (Mg/m ³)	V _{m,SSD} (%)	Padm (Mg/m ³)	V _{m,dm} (%)	<mark>Р _{bssp}</mark> (Mg/m ³)	V _{m,SSD} (%)
CM-E3	2.444	2.306	5.7	2.335	4.5	2.158	11.7	2.222	9.1
CM-E4	2.429	2.305	5.1	2.325	4.3	2.165	10.9	2.224	8.5
CM-E5	2.423	2.302	5.0	2.316	4.4	2.193	9.5	2.232	7.9

Table 6: Voids characteristics of test s	pecimens of bituminous emulsion stabilized materials
--	--

(1) The main average height of compacted specimens was, in fact, of 100 cm.

(2) The main average height of compacted specimens was, in fact, of 66 cm.



Figure 18: Voids characteristics of test specimens of bituminous emulsion stabilized materials

From observing Table 6 and Figure 18, two comments can be made:

- Air voids content achieved on specimens compacted with the standard pressure of 21 MPa are of about 4 % or 5-6% (for bulk densities determined by SSD procedure and by dimensions, respectively), which are typical values of hot asphalt layers. In contrast, for a reduced compaction pressure (7.5 MPa), air voids ranging from about 8- 9% or from about 10- 12% were obtained (for bulk densities determined by SSD procedure or by dimensions, respectively). These last values are considered to be of the same order of magnitude as the usually achieved on field for cold recycled layers containing bituminous emulsion as a binder.
- In the case of using a lower compression load (of about 7.5 MPa), it is possible to distinguish the three mixtures in terms of their air void content, wherein the mixtures containing lower bitumen binder contents having lower levels of air voids (i.e. 12 % for CM-E3 mixtures and of about 10 % for CM-5E mixtures, when its calculation is based on the bulk density determined by dimensions).



As referred before, mixtures containing small amounts of cement (1 %) besides bituminous emulsion as binder (identified as CM-E3C1, CM-E4C1 and CM-E5C1 in Table 2) were also produced and compacted using static compaction with a reduced pressure load (about 7.5 MPa). Taking into account initial studies (summarised in chapters 2 and 3), it was decided that the accelerated curing process of these type of compacted cold recycled mixtures (containing cement, even in small amounts), should comprise a day in the mould at room temperature before demoulding and further curing for 3 days in air at 50 °C. At the end of the curing procedure, the bulk density by dimensions was determined (based on the Procedure D of EN 12697-6), as well as their void content.

Table 7 and Table 8 synthesize the results obtained, respectively for CM-E3, CM-E4 and CM-E5 mixes and for CM-E3C1, CM-E4C1 and CM-E5C1 mixes.

It should be noted that all sets of cylindrical (CY) test specimens (S1 to S5) of CM-E3, CM-E4 and CM-E5 mixes were compacted using the same compression compaction machine. The same machine was used for the sets S7 to S9 static compacted test specimens of CM-E3C1, CM-E4C1 and CM-E5C1 mixes. Nevertheless, due to temporary unavailability, another static compaction machine was used for obtain the sets S10 to S11 test specimens of CM-E3C1, CM-E4C1 and CM-E5C1 mixes.

Preparation & conditioning of test specimens		Loose	mixture	ہ ø ء 1 day in the i	nens: 4 mm ^(*) + 3 days @ 50 ⁰C	
Voids characteristics		Maximum o (Mg	density, $ ho_{mv}$ /m ³)	Bulk density, ρ _{bdm} (Mg/m³)		Voids content, V _{m,dm} (%)
		ρ_{mv}	S	Pasm	P _{bdm} S	
CM-E3	Set S1 (6 CY)	2.444	0.007	2.158	0.011	11.7
CIM-L3	Set S4 (4 CY)			2.152	0.015	12.0
CM-E4	Set S2 (6 CY)	2 420	0.004	2.165	0.012	10.9
CIVI-L4	Set S5 (4 CY)	2.429	0.004	2.176	0.007	10.4
CM-E5	Set S3 (6 CY)	2 123	0.006	2.193	0.013	9.5
	Set S6 (4 CY)	2.423	0.006	2.190	0.007	9.6

Table 7: Bulk density and voids characteristics of test specimens of cylindrical test specimens of bituminous emulsion stabilized materials (CM-E3, CM-E4 and CM-E5)

(*) The main average height of compacted specimens was, in fact, of about 66 cm for all sets of test specimens.

The results of void contents of test specimens of statically compacted ($p \approx 7.5$ MPa) cold recycled mixes containing bituminous emulsion as binder (Table 7) are quite consistent between different sets of same mixtures. The following average values were found:

- CM-E3 mixes test specimens: V_{m,dm} ≈ 12 %
- CM-E4 mixes test specimens: V_{m.dm} ≈ 11 %
- CM-E5 mixes test specimens: V_{m,dm} ≈ 10 %



Table 8: Bulk density and voids characteristics of test specimens of cylindrical test specimens of bituminous emulsion stabilized materials containing small amounts of cement (CM-E3C1, CM-E4C1 and CM-E5C1)

Preparation & conditioning of test specimens		Loose	mixture	Cylindrical specimens: p ≈ 7.5 MPa ø ≈ 102 mm & h ≈ 64 mm ^(*) 1 day in the mould @ ≈20 ºC + 3 days @ 50 ºC			
Voids characteristics		Maximum (Mg	Maximum density, $ ho_{mv}$ (Mg/m ³)		Bulk density, ρ _{bdm} (Mg/m³)		
		$\overline{\rho_{mv}}$	s	Posm	s	(70)	
CM E2C1	Set S7 (4 CY)	2.457	0.272	2.153	0.005	12.4	
	Set S10 (6 CY)			2.107	0.009	14.3	
CM-E4C1	Set S8 (4 CY)	2 446	0.277	2.157	0.006	11.8	
	Set S11 (6 CY)	2.440	0.211	2.117	0.006	13.4	
CM-E5C1	Set S9 (4 CY)	2 424	0.288	2.174	0.006	10.3	
	Set S12 (6 CY)	2.424	0.200	2.131	0.005	12.1	

(*) The main average height of compacted specimens was, in fact, of about 66 cm for sets S7, S8 and S9 and of 70cm for sets S10, S11 and S16.

The results of voids content of test specimens of statically compacted ($p \approx 7.5$ MPa) cold recycled mixes with a combination of bituminous emulsion and small amount of cement as binder (Table 8) present a relatively high variability among the two sets of test specimens produced with the same mixtures. The use of a different static compaction machines (even following the same compaction procedure) from some sets to others could have played an important role on this. Besides, slightly higher test specimens (about 4 mm thicker) were obtained for the mixtures with cement in relation to those without cement. The variability of the reclaimed asphalt may have also had some influence on the results. In spite of these differences, the average void content was also determined, being obtained the following values:

- CM-E3C1 mixes test specimens: $V_{m,dm} \approx 14 \%$
- CM-E4C1 mixes test specimens: V_{m,dm}≈ 13 %
- CM-E5C1 mixes test specimens: V_{m,dm}≈ 11 %

Figure 19 shows voids characteristics obtained for similarly compacted mixtures, but one group containing only bituminous emulsion as binder the other group containing also 1 % of cement (with the same amount of added "new" bitumen).





Figure 19: Voids characteristics of test specimens of bituminous emulsion stabilized materials

From observing Table 7, Table 8 and Figure 19, the following points can be noted:

- Among the mixtures tested (from 3 to 5% of bitumen emulsion binder content, with or without 1 % of cement), the air voids content decreases for the mixtures produced with the same mix of "aggregates" composition but containing higher bituminous binder content;
- A tendency to a slight increase of the void content from test specimens with the same amount of bituminous binder, but without or with a small amount of cement is noticed.

This is true, even if the comparative analysis is restricted to test specimens compacted with the same static compaction machine (i.e. S1 to S9) and to the same samples of reclaimed asphalt (i.e. S4 to S9). In this case, a raise of about 0.5 % in the void content of mixes' test specimens containing 1 % of cement in relation to mixes without cement is observed, as follows:

- CM-E3 (Set S4): $V_m = 12.0 \%$ & CM-E3C1 (Set S7): $V_m = 12.4 \%$
- CM-E4 (Set S5): $V_m = 10.4 \%$ & CM-E4C1 (Set S8): $V_m = 11.8 \%$
- CM-E5 (Set S6): $V_m = 9.6$ % & CM-E4C1 (Set S9): $V_m = 10.3$ %

4.1.4 Main conclusions

The analyses of the presented results allow for the following considerations:

• Static compression load of approximately 7.5 MPa would be suitable to obtain representative laboratory test specimens of on site compactions.

With respect to the particular method to be used for measuring test specimens' bulk densities, guidance is provided in EN 12697-6. According to this standard, Procedure B (bulk density – SSD) is suitable for "continuously graded materials such as asphalt concrete (with relatively small pores) with voids contents up to approximately 5 %"; Procedure C (bulk density – sealed specimen) is "not suitable for reclaimed asphalt (...)" and is less convenient to conduct than the others; and Procedure D (bulk density by dimensions) is suitable for measuring the bulk density of bituminous specimens



"whatever the voids content may be" (or greater than 15 %), providing that specimens have a "regular surface and a geometric shape". Therefore, in subsequent testing, Procedure D (bulk density by dimensions) was selected for measuring the bulk density of cold recycled mix specimens.

- While, in the case of using a very high compression load, the air voids content of the three different mixtures are quite similar among them, this is not the case when using a more representative compaction pressure. In fact, the use of a suitable compression allows distinguishing the three mixtures, yielding void contents of about 12 % for CM-E3 mixtures and of about 10 % for CM-5E mixtures. Similar variation was observed for the mixtures with small amount of cement (produced with the same set of RA and compacted by same operator in the same day), wherein the void content has decreased from about 14 % for CM-E3C1 mixes to about 11 % for CM-E5C1 mixes.
- A tendency for a slight increase in the void content of test specimens when adding a small amount of cement to mixtures stabilized with bituminous emulsion (in the order of 3 to 5 %) was noticed. However, care must be taken when discussing these results, since there are a number of other aspects that can play an important role on this, such as, the variability between the reclaimed asphalt used to produce the set of cold recycled mixtures in which only bituminous binder was used (CM-E3, CM-E4 and CM-E5), and to produce the set of mixtures in which also a small amount of cement was used (CM-E3C1, CM-E4C1 and CM-E5C1). In addition, those results could have been also influenced by some variability in the compaction of test specimens.

4.2 Experimental study A2

<u>Main objective</u>: Assess characteristics of test specimens compacted by impact method (Marshall compaction) and by gyratory compactor. In this case, different vertical pressures (600 kPa and 900 kPa) combined with varying revolutions (40, 60 and 80 revolutions)

<u>Used binder(s)</u>: Bituminous emulsion or foamed bitumen, and relatively high amounts of cement (≥ 1.5 %)

4.2.1 Selected materials

For the comparison of compaction methods, four cold recycled mixes were designed. The mix composition of two mixes is given in Table 9. Mix A contains cationic slow-breaking bituminous emulsion C60B5 (former C60B7), which is commonly used in Czech Republic. Mix B is based of foamed bitumen, which was produced by the Wirtgen WLB10S device. Within the foamed bitumen production water is injected into the 170°C hot bitumen (70/100) and that leads to the foaming effect. The foamed bitumen is then immediately dosed into the two-shaft drum mixing device with controlled mixing speed, the Wirtgen WLM 30. Additional mixes C and D are similar to presented mixes and differ only in the absence of hydraulic binder.



Mix identification	Mix A	Mix B
Reclaimed asphalt mix	91.0 %	88.5 %
Water	2.5 %	4.0 %
Bituminous emulsion	3.5 %	
Foamed bitumen		4.5 %
Cement	3.0 %	3.0 %

Table 9: Used cold recycled material mix designs

The foamed bitumen is characterized by the expansion ratio (ER) and the half-time of the foam settlement (τ ¹/₂). Both parameters are strongly dependent on the type and origin of the bituminous binder, the amount of the added compressed air and the pressure of the water injected into the hot bitumen. The intensity and efficiency of the foaming effect can be influenced by the basic physical conditions such as temperature, moisture and pressure. Optimal amount of the foaming water was specified in order to achieve the maximal expansion ratio (reached value ER=18) and the maximum half-time foam settlement (reached value of 12 seconds) according to the bitumen content. For the optimization of foaming water approach defined by Wirtgen Manual was used.

4.2.2 Description of the study

In total more than 100 cylindrical specimens were compacted from each mix by using the static pressure compactor, the Marshall impact compactor and the gyratory compactor. For some variants repeated evaluation has been done. The diameter and the height of the specimens were of about 150 mm and 60 mm, respectively. In order to find out the degree of compaction, the bulk density from the specimen dimensions and its weight was calculated for each specimen.

Specimens prepared within the activities done by the Czech Technical University were compacted by Troxler Superpave Gyratory Compactor, Model 4140, according to EN 12697-31. The gyration angle of the mould was set to 1.25° and the compaction frequency was 30 revolutions per minute. The specimens were compacted by the vertical pressure of 600 kPa and 900 kPa, both loads gradually in combination with 40, 60 and 80 revolutions.

4.2.3 Results

Following Figure 20 and Figure 21 show values of the indirect tensile strength after 7 and 14 days. Furthermore they show also the bulk density values of tested specimens, which had identical composition but were compacted by different method. Bulk density is a very important parameter and the other evaluated characteristics are related to it. Therefore also the bars representing particular compaction methods are sorted according to the bulk density value. As the figures show, the bulk density value of the specimens produced by the gyratory compactor increases with the higher pressure and the number of revolutions. The bulk



density values of the specimens compacted by the static pressure compaction and Marshall impact compactor move somewhere in the middle of the achieved range between 2.05 g/cm³ to 2.20 g/cm³, with the highest values being obtained for gyratory compaction at 900 kPa and 80 revolutions and the lowest ones for gyratory compaction at 600 kPa and 40 revolutions. Bulk density of the specimens compacted by the dynamic loading of the Marshall compaction is lower than the bulk density of specimens compacted by static pressure.



Figure 20: Indirect tensile strength and bulk density of mix A



Figure 21: Indirect tensile strength and bulk density of mix B

In terms of the bulk density determination it is quite interesting to compare the standard deviation values of the bulk density obtained from the dimension and weight of the tested specimens (Table 10). Despite the apparent material heterogeneity and the influence of water contained in the mixture the variations of the bulk density values are relatively accurate and they comply with the measurement conformity, except for the specimens of Mixture B (produced with foamed bitumen) compacted using the Marshall hammer.



	Standard devia	tion σ_R – Bulk density (g/cm ³)	
Specimen compaction		Mix A (produced with 3.5% bituminous emulsion)	Mix B (produced with 4.5% foamed bitumen)
Marshall hammer 2 x 75 blows		0.020	0.043
Gyratory compactor 600 kPa	40 revolutions	0.015	0.021
	60 revolutions.	0.008	0.022
	80 revolutions	0.008	0.015
	40 revolutions	0.011	0.014
Gyratory compactor 900 kPa	60 revolutions	0.019	0.011
	80 revolutions	0.004	0.007
Static pressure	5 MPa	0.008	0.019
Requirements on conformity a DIN 1996 7:1992 (for SSD)	ccording to	0.008 -	- 0.028

Tabla 10. C	onformity of	mossured bulk	doneity data	for cold rec	welod mixes
	conformity of	measured bulk	density data	for cold rec	sycied mixes

According to Czech technical specifications as a preferred method for compaction test specimens is considered static pressure by a suitable hydraulic press. In the past it was possible to prepare specimens also by Marshall impact compaction. Introduction of optimal method of specimen compaction in the Czech technical specification which would in best way simulate the compaction process during real site conditions was forerun by many previous testing. Despite this it is always beneficial supplement bulk density values of laboratory specimens by bulk densities of cores gained from construction sites and validate this argument. The usual problem is to get undamaged cores from the pavement since the cold recycled mix is very sensitive to water mainly during the initial stage of its curing.

4.2.4 Main conclusions

From the presented figures arises that it is possible to reach similar bulk density of test specimens for same mix design prepared by different compaction method. This is valid for mixes bond by bituminous emulsion as well as by foamed bitumen. The most proximity values of bulk density for specimens compacted by static pressure with 5 MPa are reached on specimens compacted by gyratory compactor at 600 kPa and 80 revolutions or on specimens compacted by 2x75 blows of Marshall hammer. Nevertheless, it must be stressed that the highest standard deviation values were obtained for the impact compaction (Marshall hammer).



4.3 Experimental study A3

- <u>Main objective</u>: Evaluation of the influence of the compaction method on resulting void content (v_m) and indirect tensile strength (ITS) on specimens prepared using different compactions (Marshall compaction, Proctor compaction, static compaction and compaction using a vibrating table)
- <u>Used binder(s)</u>: Bituminous emulsion or foamed bitumen, and relatively high amounts of cement (≥ 1.5 %)

4.3.1 Selected materials

For the comparison of compaction methods, two cold recycled mixes were designed with bituminous emulsion and foamed bitumen based on one grading for evaluating suitable compaction and curing procedures

In order to reach adequate content of fines according to German mix design guidelines (FGSV, 2005), 3.6 % limestone filler was added to the RA. The RA grading as well as the resulting mix granulates grading after filler addition is compared to Wirtgen Manual (2012) in Table 11. Whereas the mix gradation meets the Wirtgen requirements for emulsion BSM, the applied grading result in lower contents of fine aggregates as recommended by Wirtgen Manual.

Further the applied binder contents are identified in Table 11. These contents of bitumen and cement represent well the German mix design approach and exceed the recommendations as defined by Wirtgen Manual.



Table 11: mix design for reclaimed asphalt mixtures

The recovered bitumen characteristics of the used reclaimed asphalt material are given in Table 12.



Table 12: Bitumen characteristics (recovered)

Bitumen content (% in mass)	5.4
Needle penetration (10 ⁻¹ mm)	23.0
Т _{R&B} (ºС)	63.5

The bitumen characteristics of the bituminous emulsion are given in Table 13.

Table 13: Bituminous emulsion characteristics

Type of bituminous emulsion	C	Cationic emulsion	
	60	Bitumen content 60% by mass	
	В	Unmodified bitumen	
	1	Class of breaking value	
Residual bitumen	Penetration grade @ 25 °C (10 ⁻¹ mm)	< 100	
Residual bitumen	Softening point R&B (ºC)	≥ 43	

For foamed bitumen production water was injected into the 180°C hot bitumen (50/70 pen grade) leading to the foaming effect by using Wirtgen foaming unit WLB10S. The foamed bitumen is immediately sprayed into the two-shaft spindle drum mixing device Wirtgen WLM 30.

According to Figure 22 following parameter were applied for foaming of 50/70 in this study:

- 4.5 % (in mass) foaming water content;
- 180 ℃ bitumen temperature;
- 5.5 bar bitumen pressure.



Figure 22: Foam characteristics for 4, 5 and 6 % of water content



4.3.2 Description of the study

For evaluating the influence of the compaction method on resulting void content (V_m) and indirect tensile strength (ITS) specimens were prepared using Marshall compaction, Proctor compaction, static compaction and compaction using a vibrating table.

Marshall compaction is a dynamic compaction according to EN 12697-30 (especially TP Asphalt- StB, part 30 in Germany). Within this method the samples were prepared by 50 and 100 blows each side.

Vibrating table is a compaction method for unbound and hydraulically bound mixture. There, the samples are loaded with a defined weight by a frequency of 50 Hz for 3 minutes each side.

Static compaction is used in most European countries. The University of Kassel compared the Czech standards (5 N/mm²) and the German mix Design standard (FGSV, 2005) with 2.8 N/mm². For mentioned compaction methods, ITS and void content were tested after 7 days of dry curing.

Proctor compaction is a dynamic compaction method according to EN 13286-2. The samples (diameter 150mm) are compacted by 22 blows each side. Here it was not possible to demould specimens and no mechanical tests could be conducted for these compaction methods. The bulk density was evaluated by identifying the specimen volume in the mould.

Compaction method	Standard	Characteristic	Curing methods and results
Marshall compaction	DIN EN 12697-30 (TP Asphalt-StB, part 30)	50 and 100 beats each side, dynamic, Ø = 100 mm	
Vibrating table	DIN EN 13286-5	Vibration f = 50 Hz, t = 3 min each side, load on top 10.4 kg, Ø = 100 mm	Room conditions 7 days, afterwards testing ITS _{7,dry} , void content
Static compaction	Czech Mix Design standard	5.0 N/mm², Ø = 100 mm	
	German Mix Design standard (M KRC)	2.8 N/mm², Ø = 100 mm	
Proctor compaction	DIN EN 13286-2	RA mixture is filled in 3 layers in forms, 22 beats each layer for compacting, Ø 150 mm	Void content in compaction form

Table 14: Compaction methods used by University of Kassel

For each compaction method three specimens were prepared. The compacted specimens were stored in their moulds at 20 $^{\circ}$ C and 80 $^{\circ}$ relative moisture for approximately 24 hours and demoulded afterwards. Then they were stored under room conditions for 7 days. Bulk density was determined 1 day before conducting ITS test (before temperature conditioning 1 day prior the test).



4.3.3 Results

For evaluation between the effect of compaction procedure and void contents or resulting strength properties, an emulsion mix (which was produced with 4 % of bituminous emulsion, i.e. 2.4 % residual bitumen + 2 % of cement) and a foamed bitumen mix (which was produced with 4 % of foamed bitumen, i.e. 3.8 % residual bitumen + 2 % of cement) as described in paragraph 4.3.1 were prepared with varied compaction procedures.

The resulting void contents and ITS values after 7 days curing at room conditions, i.e. 40-70 % relative humidity and temperature of (20 ± 2) °C, are plotted respectively, in Figure 23 for the bituminous emulsion mixtures and in Figure 24 for foamed bitumen mixtures.



Figure 23: Void contents (left) and ITS (right) for emulsion based cold recycled mixtures



Figure 24: Void contents (left) and ITS (right) for foamed bitumen based mixtures

From the void content results it can be concluded, that the compaction procedures using Proctor Standard, vibrating table and Marshall method (2 x 50 and 2 x 100 blows) are not feasible to reach adequate void contents (10-15 % as usually obtained in field and recommended by Wirtgen Manual). The void content directly influences the ITS results as also indicated in the above figures. For the vibrating table compaction method (emulsion mix) and Proctor Standard compaction (both mixes, i.e. emulsion and foamed bitumen mixes) the compacted specimens didn't obtain enough strength for de-moulding and evaluation of ITS.



Therefore, in the present study compaction procedures using Proctor Standard, vibrating table and Marshall compaction method seem not to be feasible to reach adequate void contents smaller than 15 %. Such values could only be reached with static compaction conditions used in the Czech Republic (load pressure of 5.0 N/mm²).

4.3.4 Main conclusions

From the tests evaluated in this study it can be concluded that impact compaction procedures as usually applied for unbound mixtures (Proctor) or hot-mix asphalt (Marshall) as well as vibrating table method as successfully applied for hydraulically bound mixtures are not suitable for laboratory-compaction of cold recycled mixtures. The comparably higher resistance against compaction of this type of mixtures demands for static compaction procedures. For these, the increase in vertical pressure will decrease the specimen's voids content and increase its strength parameters as expected.

During compaction of the specimens on emulsion mix, it was observed that the first specimens compacted from the fresh emulsion mix were easier to compact and reached lower bulk densities compared to the last specimens compacted from the same emulsion mixture, which emulsion started to break a short time after the mixing. Therefore, the produced mix portion should be not too big considering the required time for the compaction process.



5 Cold recycled mixture characteristics and performance, considering the effect of different curing procedures

5.1 Introduction

For cold recycling mixes, curing is an important process during which water released by aradual break-down and consolidation of the bituminous emulsion or drving-up of the consolidated wet mix is drained, or such water is used for cement hydration, thus ensuring gradual stiffening thereof. Generally, lower water content of a bitumen stabilized mix (emulsion or foamed bitumen) results in an improved strength and resistance to water thanks to better adhesion. Contrastingly, in the case of hydraulic binder with content ≥ 1.5 %, it is important that the mix has sufficient moisture level and that the water can be used for hydration. This allows achieving the highest strength characteristics possible. Depending on the surrounding conditions of the environment where the material is curing, the water content in the mix can decrease more quickly or more slowly due to its evaporation; however, that is still not enough for the reclaimed material particles to form good quality bonds with the bituminous binder. The process requires roughly one to six months when the adhesion barrier is gradually broken due to the residual water film around the particles and a solid bond is formed. This leads immediately to improved strength and stiffness values. The time of the curing process is connected to that; the curing might take months with respect to the fact that reality lacks any ideal conditions that could be used to simulate the curing process in a laboratory. Due to the need to know the predicted development of strength characteristics as soon as possible, accelerated curing procedures of such characteristics that could be used in laboratory assessments while imitating the conditions of curing in construction practice have always been searched for to deliver results that the assessed mix would demonstrate if applied in practice. It is not easy to find such conditions for specimen curing in laboratory environment. At the same time, it should be pointed out that in contrast to a testing laboratory the new structure is influenced mainly by the weather conditions (temperature fluctuation, negative impact of rain water and frost, etc.). The weather conditions, or the specimen curing conditions, have been shown to have a significant effect on mix properties and, therefore, the research of their impact has been one of the main objectives of the measurements taken. Primarily in the case of mixes with higher cement contents, unfavourable curing conditions can result in insufficient hydration during the curing time, or in the occurrence of micro-cracks that deteriorate mechanical properties and overall durability of the work performed.

With respect to the current trend of construction practice where the time factor has been the most important priority for a number of years, waiting 28 days for the strength test results and only paving the subsequent cold recycling layer afterwards is impossible. Instead, the results of measurements after 7 days are used which allow conclusions on the resulting strengths to be achieved by the mix. However, these do not have the exact information value and, therefore, some countries have undertaken to determine strength characteristics after 14 days of curing. Such values should give a more precise idea of the mix properties,



particularly if combined with an assessment of water susceptibility and the strength ratio determination. Within the framework of the results presented further in this paper, measurements were taken after 7 as well as 14 days for the sake of comparison of curing options in order to give a more precise idea of mix behaviour and to demonstrate the striking difference between them (Asphalt Academy, 2004; Forde, 2009; Formanová, 2011; Wirtgen, 2012; Tebaldi *et al*, 2012). For some curing procedures this has been modified to shorten the period of determining strength and comparing the values reached after 14 days.

Assessed cold recycled mixes were observed for the impact of various combinations of curing procedures in environments of different moisture levels under temperatures of the surroundings where the specimens were cured. Based on the literature searches conducted within this project, several curing methods which are applied all over the world and represent various approaches were selected for the experimental comparison. The countries apply different curing methods with distinctive differences in the conditions to which the test specimens are exposed.

Czech Republic

The curing procedure for cold recycled mixes is described by national technical specifications, TP 208, which stipulates the temperature, humidity and time frames for test specimens. For mixes bound by cement or other hydraulic binders, test specimens are stored at 90-100 % humidity and temperature of (20 ± 2) °C. If applying cement and bituminous emulsion or foam, the test specimens are stored for 2 days at 90-100 % humidity and temperature of (20 ± 2) °C. If applying cement and bituminous emulsion or foam, the test specimens are stored for 2 days at 90-100 % humidity and temperature of (20 ± 2) °C. For mixes bound only by bituminous binders, such as bituminous emulsion or foamed bitumen, test specimens are stored in climatic chamber at (20 ± 2) °C. The reason for the different storage of specimens is the different impact of used binders. Storage under 90-100 % humidity can be replaced by the use of an impermeable cover. Contrastingly, 40-70% humidity simulates storage with the relative humidity of air, i.e. without any quick impermeable overlay (Czech Ministry of Transportation, 2008).

Great Britain and Ireland

In the case of the curing method applied in Great Britain the test specimens are left sealed for 28 days to cure at the temperature of 40 °C. For the entire duration of the process, relative humidity of 90-100 % is required (therefore curing in impermeable covers), (Tebaldi *et al*, 2012). The reason for this procedure is explained by fast overlay of the recycled layer and by the frequent rains occurring in this part of Europe.

<u>France</u>

The curing of test specimens in France is specified by the French national standard, NF P 98-251-4, which also stipulates the static compression testing performed on asphalt mixes including the modified test for cold recycling mixes. The French test method specifies



the requirements for cold mixes where the test specimens are stored at constant temperature of 18℃ in an environment with relative humidity of 40-70 % (COLAS, 2006).

<u>Australia</u>

The up-to-date summary "Austroads Technical Report AP-T178/11: 2011" recommends test specimen curing to follow the process where the test specimens are first exposed to the temperature of 40 \degree for 3 days without any impermeable covers. For the remaining time before the strength testing, the specimens are left at the temperature of (20±2) \degree , (Asphalt Academy, 2004; Tebaldi *et al*, 2012).

South Africa

In South Africa where cold recycling has long tradition, the curing process has been described within the framework of the Asphalt Academy TG2: 2009, which discussed the inappropriateness of the test specimen curing as applied since 1994, using temperature of 60°C. The new specifications have reduced the tempe rature to 40°C. It has been stipulated that test specimen enclosure in an impermeable cover for 72 hours under the temperature of 40°C maintains excess humidity and delivers very conservative results for the specimens. In contrast to that, the curing of specimens which are first exposed to the temperature of 25°C for 24 hours without being sealed and, subsequently, wrapped in plastic bags and transferred to the drying facility with temperature regulation to 40°C for 48 hours (this method better complies to the steady humidity to be achieved) does not present any evidence that the laboratory measurements are consistent with the actual conditions achieved in practice, (Tebaldi *et al*, 2012; AUSTROADS, 2011).

Portugal

The last of the aforementioned procedures described the so-called accelerated curing method which applies higher temperatures than in the preceding cases. Test specimen curing occurs for 1 day at about 90 % of relative humidity (unmolded specimens) and at room temperature (about 20°C) and, then, the specimens are demoulded and transferred to a tempered chamber that maintains the temperature of 50° where they remain for 3 days before testing (CoRePaSol Project Report D1.1). This laboratory accelerated curing procedure has proved to properly simulate on site cold recycled mixtures produced with bituminous emulsion binders (combined with small amounts of hydraulic binders, if required), providing that favourable weather conditions occur on site (i.e. no rain) and that no overlay is placed on the recycled layer for some weeks (generally, 2/3 weeks in favourable weather conditions), in order to allow a progressive reduction of its water content, which generally stabilize around 1-2 %. In the case of using bituminous binder combined with relatively high amounts of hydraulic binders (> 1.5 %), this type of laboratory accelerated process (unsealed specimens @ relatively high temperature) may result in significant lack of humidity needed for hydration, depending among other on the total water content on the mixture and on the relation between the bitumen residue and the cement content.



5.2 Experimental study B1

<u>Main objective</u>: Assessment of water sensitivity of cold recycled mixtures compacted, cured and conditioned by different procedures

<u>Used binder(s)</u>: Bituminous emulsion with or without small amounts of cement (1 %)

5.2.1 Selected materials

Same materials as described in paragraph 4.1.1 were used.

5.2.2 Description of the study

In Portugal and Spain, the determination of "optimum" emulsion/bitumen-residue content is traditionally based on the following steps:

- Preparation of cylindrical test specimens (ø≈h≈101.6 mm) by static compaction with double plunger action, applying a compression load of 21 MPa (based on the Spanish Specification NLT161 or on the ASTM D1074). Nevertheless, due to research studies in the late 1900s/early 2000s a lower pressure has been often used since then;
- Accelerated curing of test specimens by storing them in the oven at 50°C for 3 days after 1 day in the mould;
- Immersion-compression tests (NLT 162 standard 1st Procedure / ASTM D 1075 Alternative procedure):
 - "Dry" specimens: 4 days in air @ 25℃ + 2h in water @ 25℃ ⇔ compressive strength @ 25℃ (S_d)
 - "Wet" specimens: 4 days in water @ approx. 50℃ + 2 h @ room temperature + 2 h in water @ 25℃ ⇔ compressive strength @ 25℃ (S w)
- Determination of the "Index of retained strength" (IRS), which corresponds to the ratio between the compressive strength of specimens with immersion and without immersion, expressed in percent ($S_d/S_w \times 100$).

This index can be understood as a common known alternative to indirect tensile strength ratio, in asphalt mix design.

Similarly to the Duriez design method (see paragraph 2.3.5), the strength of "dry" and "wet" specimens and the retained strength of specimens after immersion are also checked for compliance with the relevant mix design specification. According to the Portuguese and Spanish procedure, the compressive strength of the specimens is determined in accordance with NLT-161 (or ASTM D 1074) test method, applying a compression load at a rate of 5.08 mm.min⁻¹. Table 15 shows the required values, for specimens tested according the above described procedure.



Compressive strength	High traffic volume (T0/T1)	Low traffic volume
Compressive strength of "dry" specimens @ 25℃ (S d), minimum	3.0 MPa (⇔ ≈24 kN for Ø≈101,6 mm)	2.5 MPa (⇔ ≈20kN for Ø≈101,6mm)
Compressive strength of "wet" specimens @ 25°C (S _w), minimum	2.5MPa (⇔ ≈20 kN for Ø≈101,6 mm)	2.0MPa (⇔ ≈16kN for Ø≈101,6mm)
Index of retained strength (IRS), minimum	75%	70%

Table 15: Characteristics of the bitumen on the reclaimed asphalt

In the present experimental study, immersion-compression tests were performed according to the specified method. Furthermore, water sensitivity tests were carried on (Figure 26), but using the following procedures:

- Static compaction with double plunger action, applying a reduced compression load of 7.5 MPa (which was found to be more suitable, as described in 4.1) of cylindrical test specimens of about 102 mm in diameter and 64 mm in height ("Marshall" test specimens' dimensions);
- Accelerated curing of the test specimens by storing them in the oven at 50°C for 3 days;
- Water sensitivity tests, according to method A of the European standard EN 12697-12:
 - "Dry" specimens: 3 days in air @ (20±5)℃ ⇒ Indirect tensile strength @ 15℃ (ITS d)
 - "Wet" specimens: Vacuum (30 min. @ 6.7 kPa in water @ 20℃) + 68-72 h in water
 @ 40℃ ⇒ Indirect tensile strength @ 15℃ (ITS _w)

Determination of the "Indirect tensile strength ratio" (ITSR), calculated as the ratio of the indirect tensile strength of "wet" specimens to that of dry specimens, expressed in percent ($ITS_d/ITS_w \times 100$).

In this context, bituminous emulsion stabilized mixtures were produced according to the compositions shown in Table 2 (mixtures in which only bituminous emulsion was used as binder: CM-E3, CM-E4 and CM-E5; mixtures in which also a small amount of cement is used; CM-E3C1, CM-E4C1 and CM-E5C1).

5.2.3 Main results

Immersion-compression tests were performed according to the "traditional" method (static compression p=21 MPa, accelerated curing 3 days @ 50° C; immersion-compression tests according to NLT 162 standard / ASTM D 1075), whose results are presented in Table 16 and Figure 25. Figure 25 also shows the threshold values required for the unconfined compressive strength of "dry" and "wet" cured specimens (3 days @ 50° C) and for their retained strength after immersion in water.



From the data presented in Figure 25, it can be concluded that the unique mixture that complies all requirements both for low and high traffic levels is the CM-E3 (2.8 % in mass of bituminous emulsion on the final mixture).

Afterwards, the above described "modified" procedure for evaluation of cold recycled mixture resistance to water (static compression applying a reduced compression load $p\approx$ 7,5 MPa, accelerated curing 3 days @ 50°C; water sensitivity tests based according to EN 12697-12) were carried on. Table 17 and Figure 26 show the results obtained for this modified mix design procedure.

Table 16: Immersion-compression test results (NLT 162 standard/ASTM D 1075) on CM-E3, CM-E4 and CM-E5 mixtures

Preparation & conditioning of test specimens	Cylindrical specimens: ø ≈ h ≈ 102 mm Static compaction: p = 21 MPa Curing: 3 days @ 50 ºC				
Testing / Mixtures ID	Determination of bulk density (based on EN 12697-6 - Procedure D)Determination of voids content (based on EN 12697-8)Immersion-compression tests (NLT 162 – 1 st Procedure / ASTM D 1075 – Alternative procedure)			ests 075 – Alternative	
	ρ _{bdm} (Mg/m ³)	V _{m,dm} (%)	S _d (kN)	S _w (kN)	IRS (%)
CM-E3	2.306	5.7	5.7 26.7 20.3 76		
CM-E4	2.305	5.1	22.3	18.1	81
CM-E5	2.302 5.0 17.4 16.8 97				



Figure 25: Compression-immersion tests according to NLT162 (adapted to cold recycled mixes by curing test specimens prior to testing) on CM-E3, CM-E4 and CM-E5 mixtures

If the same minimum of retained strength (i.e. same minimum ITSR as IRS) was required in the case of the "modified" mix design procedure, just mixtures CM-E4 and CM-E5 would meet specifications. In fact, in this case, a slight increase in ITS of wet specimens from the



mixture with lower bituminous emulsion content (CM-E3) to the mixture with higher bituminous emulsion content (CM-E5) is recorded. This is fairly consistent with the results obtained for the voids content of the different cold recycled mix specimens compacted by static pressure of 7.5 MPa (see paragraph 4.1.3), suggesting that mixtures are more resistant to water when their voids content decreases.

It is worth to highlight that both mixtures, CM-E4 and CM-E5, present ITSR of the same order of magnitude (higher then 85%). Thus, if only water sensitivity results were taken into account, the selected bituminous binder content for the final cold recycled mix composition would be CM-E4 (the one with lower bitumen content between the two that comply with required values, for economical reasons).

Preparation & conditioning of test specimens	Cylindrical specimens: ø ≈ 102 mm, h ≈ 66 mm Static compaction: p = 7.5 MPa Curing: 3 days @ 50 ºC					
Testing / Mixtures ID	Determination of bulk density (based on EN 12697-6 - Procedure D)	of / Determination of voids content (based on EN 12697-8) Water sensitivity tests (based on EN 12697-12 – method A, ITT @ 15 °C)			; , ITT @ 15 ℃)	
	ρ _{bdm} (Mg/m ³)	V _{m,dm} (%)	ITS _d (kPa)	ITS _w (kPa)	ITSR (%)	
CM-E3	2.158	11.7	11.7 1130 750 66			
CM-E4	2.165	10.9 890 770 87			87	
CM-E5	2.193	2.193 9.5 920 810 88				

Table 17: Water sensitivity tests (EN 12697-12) on CM-E3, CM-E4 and CM-E5 mixtures



Figure 26: Water sensitivity tests (EN 12697-12 - method A: ITT @ 15 °C, test specimens compacted by a static pressure of 7.5 MPa, and cured prior to testing) on CM-E3, CM-E4 and CM-E5 mixtures



Furthermore, cold recycled mixtures produced using 1 % of cement besides bitumen emulsion binder (identified as CM-E3C1, CM-E4C1 and CM-E5C1 in Table 2), where also compacted using the above referred modified test method (static compaction p=7.5 MPa, $\emptyset \approx 102$ mm & h ≈ 64 mm), cured for 1 day in the mould at room temperature and 3 days in air at 50 °C and further tested in order to evaluate its resistance to water. Table 18 shows the results obtained. In order to allow for a better comparison between cold recycled mixtures with bituminous emulsion and those with combined binder of bituminous emulsion and small amount of cement, their water sensitivity test results are graphically represented in Figure 27.

Preparation & conditioning of test specimens	Cylindrical specimens: ø ≈ 102 mm, h ≈ 66 mm Static compaction: p = 7.5 MPa Curing: 1 day in the mould @ 20 ºC + 3 days @ 50 ºC				
Testing / Mixtures ID	Determination of bulk density (based on EN 12697-6 - Procedure D)	Determination of voids content (based on EN 12697-8)	Water sensitivity tests (based on EN 12697-12 – method A, ITT @ 15 ºC)		
	ρ _{bdm} (Mg/m ³)	V _{m,dm} (%)	ITS _d (kPa)	ITS _w (kPa)	ITSR (%)
CM-E3C1	2.107	14.3	640	460	72
CM-E4C1	2.117	13.4	750	510	68
CM-E5C1	2.131	12.1	760	530	70

Table 18: Water sensitivity tests (EN 12697-12) on CM-E3C1, CM-E4C1 and CM-E5C1 mixtures

The data presented in Table 18 show that all mixtures showed similar ITSR values. However, the indirect tensile strength both for "dry" and "wet" conditioning specimens slightly increases from the mixtures with lower bituminous emulsion content to the mixtures with higher contents. This results may suggest than, when selecting a mix composition based on water sensitivity tests, besides ITSR values also ITS values of "water" conditioning test specimens should be taken into account.



Figure 27: Water sensitivity tests (EN 12697-12 - method A, test specimens compacted by a static pressure of 7.5 MPa, and cured prior to testing) on CM-E3, CM-E4, CM-E5, CM-E3C1, CM-E4C1 and CM-E5C1 mixtures



From the analysis of Figure 27, there are two main points to note:

- All the mixtures containing a small amount of cement besides bituminous emulsion as a binder, present lower ITS values (ITS_d & ITS_w) than the correspondent mixtures without. As stated before, the fact that the mixtures with the same content of bituminous emulsion have, for the tested specimens, lower void contents than those with cement could certainly have had an influence on these results. On the other hand, this may suggest that the accelerated curing of 3 days at 50 °C could simulate more advanced curing stages in the case of mixtures produced using only bituminous emulsion as binder and simulate earlier stage curing stages in the case of mixtures produced using also cement. In order to investigate this, other experimental studies were carried out, and will be presented in following sections.
- For the two mixtures with the lowest bitumen content (CM-E3 and CM-E3C1) the correspondent ITSR slightly increases when cement is added. On the other hand, for the other four mixtures (CM-E4 / CM-E4C1 and CM-E5 / CM-E5C1), the correspondent ITSR decreases when cement is added (from values about 85-90 % to values of about 65-70 %). Regarding water resistance, these results may suggest that cement plays a more important role as a binder when small amounts of the "main" binder (bituminous emulsion in this case) are relatively low.

5.2.4 Main conclusions

The results of both A1 and B1 experimental studies point out to the following conclusions:

- Static compaction of cylindrical specimens (based on the Spanish Specification NLT161 or on the ASTM D1074 methods) applying a reduced compressive loading of about 7.5 MPa provides fairly representative densities of in situ compactions;
- Water sensitivity tests performed according to the European standard EN 12697-12 on cured test specimens are suitable for distinguishing the performance of different mixtures (produced with different bituminous emulsion contents). However, besides minimum ITSR values, also minimum ITSw values should be required.

5.3 Experimental study B2

<u>Main objective</u>: Evaluate the effect of different laboratory accelerated curing procedures on the Indirect Tensile Strength of cold recycled mixtures

Used binder(s): Bituminous emulsion with or without small amounts of cement (1 %)

5.3.1 Selected materials

Same materials as described in paragraph 4.1.1 were used.

5.3.2 Description of the study

As described in chapter 3, in order to evaluate different curing methods, comparative studies on mixtures with emulsion and without or small content of cement (<1.5 %) were undertaken.



In the present study, the following curing conditions were addressed:

- 7 days in total: 24 h for demoulding + 6 days at room temperature and humidity (40-70 % relative humidity);
- 14 days in total: 24 h for demoulding + 13 days at room temperature and humidity (40-70 % relative humidity);
- 4 days in total: 24 h for demoulding + 3 days @ 50°C.

For the comparison of the above described curing procedures, six cold recycled mixes were produced according to the compositions shown in Table 2: three of them using only bituminous emulsion (C60B5) as binder (labelled as CM-E3, CM-E4 and CM-E5); and other three containing 1 % of cement (CEM I 42.5 R) besides bituminous emulsion (labelled as CM-E3C1, CM-E4C1 and CM-E5C1).

Afterwards, test specimens were prepared applying a static compaction according to the procedure described in NLT 161 standard (based on ASTM D1074), but applying a compressive load of about 7.5 MPa. Test specimens were conditioned prior to further testing.

At the end of the curing conditioning, the Indirect tensile Strength (ITS) was determined according to the method described on EN 12697-23 standard, at a temperature of 15 °C.

5.3.3 Main results

The Indirect Tensile Strength of the referred six mix compositions was determined. Table 19 and Table 20 show the results obtained, respectively for the mixtures with bituminous emulsion and for the mixtures with combined binder of bituminous emulsion and cement. Those results are graphically represented, respectively, in Figure 28 and Figure 29.

From the results presented in Table 19 and Figure 28, the following comments can be made:

- In general, all mixtures increase their ITS values from earlier to later ages of curing at room temperature (i. e. from 7 to 14 days of curing prior to testing) and from these to accelerated curing procedures by storing demoulded test specimens at higher temperatures (50 °C) even for shorter periods (3 days prior to testing). An exception is made only for mixture CM-E5, from 7 to 14 days at room temperature, which certainly is due to the higher void content of the set of test specimens conditioned during 14 days ($V_m = 10.3$ %) than the set conditioned during 7 days ($V_m = 9.5$ %). This fact highlights the elevated influence that air void content may have on the strength of tested compacted mixtures (with same compositions).
- For early ages of curing (7 days) at room temperature, all mixtures seem to present similar ITS values (about 700 kPa, i.e. higher than 0.30 MPa and lower than 0.75 MPa which are common requirements for cold recycled mixes containing bituminous binder and cement after 7 days of curing). However, when the duration of curing increases (for 7 to 14 days in total) or when the curing is accelerated due to higher levels of temperature (from approx. 20 °C to 50 °C), the mixtures with lower bitumen content (which increases from CM-E3 to CM-E5) show higher ITS values.

The fact that the volume of voids of the tested cold recycled mixes increases when their bituminous binder content decreases (e.g. $V_m(CM-E3)\approx 12$ % and $V_m(CM-E5)\approx 10$ %) may



contribute for a faster curing in forced ventilated conditions for test specimens presenting higher void contents, and therefore leading to faster strength gains.

These results are fairly consistent with the ones obtained for Indirect Tensile Strength of "dry" specimens (ITS_d) in water sensitivity tests (see paragraph 5.2.3). However, it is worth to highlight that, in spite of ITS_d values have generally increased with the bituminous binder content of the mixtures, its resistance to water has decreased due to lower ratios between ITS_w and ITS_d values (ITSR).

• All mixes present ITS values at accelerated curing stages higher than 0.75 MPa, which is a common recommended requirement for cold recycled mixes containing bituminous binder and cement after 28 days of curing.

Therefore, within the purpose of selecting an optimum bitumen content among the referred three mixtures as (CM-E3, CM-E4 and CM-E5), the selection would be the mixture with 3 % of bituminous emulsion (CM-E3) if the criteria relied exclusively on ITS tests, but if the criteria was based on water sensitivity tests, a mixture with a higher bitumen content would then be selected (i.e. CM-E4).

Preparation of test specimens	Cylindrical specimens: ø ≈ 102 mm, h ≈ 66 mm Static compaction: p = 7.5 MPa			
Testing / Mixtures ID	Bulk density (based on EN 12697 6 - Procedure D) ρ _{bdm} (Mg/m ³)	Voids content (based on EN 12697-8) V _{m,dm} (%)	Laboratory accelerated curing procedure	Indirect Tensile Strength (based on EN 12697-23, t=15 °C) ITS (kPa)
	2.162	11.5	1 day in the mould + 6 days in air @ room temperature	670
CM-E3	2.143	12.3	1 day in the mould + 13 days in air @ room temperature	770
	2.152	12.0	1 day in the mould @ room temperature + 3 days @ 50 ⁰C	1050
	2.180	10.3	1 day in the mould + 6 days in air @ room temperature	670
CM-E4	2.165	10.9	1 day in the mould + 13 days in air @ room temperature	760
	2.176	10.4	1 day in the mould @ room temperature + 3 days @ 50 °C	980
	2.193	9.5	1 day in the mould + 6 days in air @ room temperature	710
CM-E5	2.175	10.3	1 day in the mould + 13 days in air @ room temperature	650
	2.190	9.6	1 day in the mould @ room temperature + 3 days @ 50 °C	870

Table 19: Water sensitivity tests (EN 12697-12) on cold mixes with bituminous emulsion as binder, submitted to different curing procedures





Figure 28 – Indirect Tensile Strength tests (EN 12697-23) on cold mixes with bituminous emulsion as binder, submitted to different curing procedures

Table 20: Indirect Tensile Strength tests (EN 12697-23) on cold mixes with combined binder of bituminous emulsion and cement (< 1.5 %), submitted to different curing procedures

Preparation of test specimens	Cylindrical specimens: ø ≈ 102 mm, h ≈ 66 mm Static compaction: p = 7,5 MPa			
Testing / Mixtures ID	Determination of bulk density (based on EN 12697 6 - Procedure D)	Determination of voids content (based on EN 12697-8)	Laboratory accelerated curing procedure	Determination of Indirect Tensile Strength (based on EN 12697-23, t=15 °C)
	ρ _{bdm} (Mg/m ³)	V _{m,dm} (%)		ITS (kPa)
	2.153	12.4	1 day in the mould + 6 days in air @ room temperature	730
CM-E3C1	2.140	12.9	1 day in the mould + 13 days in air @ room temperature	720
	2.153	12.4	1 day in the mould @ room temperature + 3 days @ 50 ℃	1010
	2.174	11.1	1 day in the mould + 6 days in air @ room temperature	700
CM-E4C1	2.154	12.0	1 day in the mould + 13 days in air @ room temperature	780
	2.157	11.8	1 day in the mould @ room temperature + 3 days @ 50 ⁰C	1090
	2.189	9.7	1 day in the mould + 6 days in air @ room temperature	820
CM-E5C1	2.176	10.2	1 day in the mould + 13 days in air @ room temperature	790
	2.174	10.3	1 day in the mould at room temperature + 3 days @ 50 ºC	1070





Figure 29 – Indirect Tensile Strength tests (EN 12697-23) on cold mixes with combined binder of bituminous emulsion and cement (\leq 1.5 %), submitted to different curing procedures

With respect to the mixtures with combined binder of bituminous emulsion and a small amount of cement (Table 20 and Figure 29), the following findings can be drawn:

- The ITS values both for test specimens cured during 7 and 14 days are of the same order of magnitude. This may suggest that, even small amounts of cement, changes the process of curing of cold stabilized mixtures, which is mainly attributed to the need for cement hydration.
- On the other hand, when test specimens are submitted to an accelerated laboratory curing procedure of 1 day in the mould at room temperature and 3 days @ 50°C, an improvement in their indirect tensile strength is observed.
- Analysing the results in relation to recommended indirect tensile strength requirements for cold recycled mixes containing bituminous binder and cement, ITS₇ > 0.3 MPa & ITS₇ ≤ 0.75 MPa for 7 days of curing and ITS₂₈ > 0.75 MPa for 28 days of curing, it can be concluded that CM-E5C1 achieves quickly a relatively high strength values, which don't allow this mixture to comply with the referred maximum strength at 7 days.
- There may be a slight tendency for the ITS values of CM-E4C1 and CM-E5C1 being somewhat higher than those of CM-E3C1, but even so, for each type of laboratory curing procedure, all three mixtures show same levels of ITS values.

These results are quite consistent, once again, with the ones obtained for Indirect Tensile Strength of "dry" specimens (ITS_d) in water sensitivity tests (see par. 5.2.3).

Bearing in mind the above referred with respect to mixtures containing a small amount of cement besides bitumen emulsion as binder, the results of both water sensitivity and ITS tests didn't allow for a clear selection of the optimum bituminous binder content.

In order to evaluate the influence of adding small amounts of cement on cold recycled mixtures using bituminous emulsion as main binder, their ITS test results, for groups with the same "new" bituminous binder content, were graphically represented in Figure 30.



From the data presented in Figure 30, the following comments can be made for particular bituminous emulsion content used as binder in cold recycling:

- Cold recycled mixtures with 3 % of bituminous emulsion (CM-E3 & CM-E3C1): The addition of 1 % of cement seems only to have some influence on ITS test results, by increasing the mixture' strength, on early curing stages (7 days at room temperature). This, nevertheless, could be an important factor to cold recycled layers, since in the field is usually essential to have faster curing processes in order to cold mixtures reach higher strength as soon as possible.
- Cold recycled mixtures with 4 % of bituminous emulsion (CM-E4 & CM-E4C1): In this case, the addition of 1 % of cement seems to have some influence on ITS results, by increasing the mixtures' strength in all addressed curing procedures, with emphasis for the one that simulates later curing stages (1 day in the mould + 3 days @ 50°C).
- Cold recycled mixtures with 5 % of bituminous emulsion (CM-E5 & CM-E5C1): Similarly to the previous mixtures, the addition of 1 % of cement seems to play a relatively important role on ITS results, since higher mixtures' ITS values were obtained in all addressed curing procedures.



Figure 30: Indirect Tensile Strength tests (EN 12697-23) on test specimens of CM-E3, CM-E4, CM-E5, CM-E3C1, CM-E4C1 and CM-E5C1 mixtures (static compacted & cured prior to testing)

Conversely to the above results, the water sensitivity test results suggested that cement would play a more important role as a binder for the mixtures with lower bituminous binder content (mixtures with 3 % of bituminous emulsion).

In order to evaluate which binder composition would be the optimal, the ITS values for each of the six types of mixtures (CM-E3, CM-E4, CM-E5, CM-E3C1, CM-E4C1 and CM-E5C1) test specimens were again graphically represented, but this time in groups within the same curing stage (Figure 31).



Taking only into account the results presented in Figure 31, the following comments can be made:

- At an early curing stage of 7 days at room temperature, CM-E5C1 mix distinguishes from the others by showing the highest ITS value (above 800 kPa), while the remaining five mixtures have values of the order of 700 kPa.
- At a curing stage of 14 days at room temperature, only one mixture (CM-E5) shows an ITS value below 700 kPa, while four of the mixtures (CM-E3, CM-E4, CM-E4C1 and CM-C5E1) show ITS values in the order of magnitude of 800 MPa.
- At a further curing stage (1 day in the mould + 3 days @ 50 °C), CM-E5 mix is again the one presenting the lowest ITS value (below 900 kPa). All the remaining five mixtures (CM-E3, CM-E4, CM-E3C1, CM-E4C1 and CM-E5C1) show ITS values of about 1000-1100 kPa.



Figure 31: Indirect Tensile Strength tests (EN 12697-23) on CM-E3, CM-E4, CM-E5, CM-E3C1, CM-E4C1 and CM-E5C1 test specimens (static compacted), cured under different procedures

If the results from water sensitivity tests, which were performed for an unique curing process (3 days @ 50°C) (Figure 29) are considered together with the results from Indirect Tensile Strength tests (Figure 31), then the following conclusions can be drawn:

- Among the mixtures with bituminous emulsion as a binder, CM-E4 mix would be selected, because it shows a relatively good ITSR value (above 85 %) and quite satisfactory values of ITS for the three different stages of curing considered.
- Among the mixtures with combined binder of bituminous emulsion and a small amount of cement, the obtained ITSR values didn't allow for a distinguish between mixtures, and only a slightly tendency for an increasing of values of ITS_d and ITS_w values from CM-E3C1 to CM-E5C1 would suggest that CM-E5C1 would have a better performance. From the ITS test results, only the curing conditioning for 7 days at room temperature allows to clearly distinguish CM-E5C1 mix from the other two. Thus, in this case, other tests should be carried out in order to support the selection of optimum binder content.


5.3.4 Main conclusions

The results of B2 experimental studies, together with previously presented studies (A1 and B1), point out to the following conclusions:

- In general, cold recycled mixtures using bituminous emulsion as binder, increase their ITS values from earlier to later ages of curing at room temperature (i.e. from 7 to 14 days of curing prior to testing). On the other hand, cold recycled mixtures with combined binder of bituminous emulsion and 1 % of cement, showed ITS values of the same order of magnitude for test specimens cured during 7 and for 14 days at room temperature. This suggests that, even small amounts of cement (which require relatively long periods of hydration in order to get strength), may change the process of curing of cold stabilized mixtures.
- As regards to the accelerated laboratory curing procedure of 1 day in the mould at room temperature and 3 days @ 50 °C, an improvement in both type of mixtures (with or without cement) indirect tensile strength was observed in relation with the obtained at earlier curing stages (7 and 14 days at room temperature).
- Moisture susceptibility tests (ITSR) and ITS tests didn't allow for a distinction between mixtures containing 1 % of cement and varying contents of bituminous emulsion.
- The addition of small amounts of cement seems to provide the following advantages:
 - Increase the early strength of the mixtures (from lower to higher mix bitumen contents);
 - Increase the strength of advanced cured mixes produced with relatively high contents of bituminous emulsion (about 5 %).

5.4 Experimental study B3

<u>Main objective</u>: The main focus of this experimental study was based on the systematic measurement of ITS values and stiffness modulus of test specimens compacted by different procedures (see study A2 in section 4.2)

<u>Used binder(s)</u>: Bituminous emulsion or foamed bitumen, and high content of cement (3 %)

5.4.1 Selected materials

The same materials presented in 4.2.1 were used.

5.4.2 Description of the study

This experimental testing was based in systematic measurements of the Indirect Tensile Strength and the Stiffness modulus. Both values were measured on specimens cured by different procedures, covering a wide range of varying conditionings, such as: duration, temperature and relative humidity.



In the present study, cold recycled mixtures were produced using a combination of bituminous (3.5 % of emulsion or 4.5 % of foamed bitumen) and hydraulic (3 % of cement in mass of the final mixture) binders.

As reported in chapter 3, comparative curing studies on those types of cold recycled mixtures, addressed the following variations:

- 7 days in total: 24 h for demoulding + 6 days at room temperature and 40-70 % relative humidity (unsealed conditions) \rightarrow verify if ITS₇ > (0.3 0.5) MPa & ITS₇ ≤ 0.75 MPa
- 7 days in total: 24 h for demoulding + 6 days at room temperature and 90-100 % relative humidity (sealed specimens) \rightarrow verify if ITS₇ > (0.3 0.5) MPa & ITS₇ ≤ 0.75 MPa
- 14 days in total: 24 h for demoulding + 13 days at room temperature and 40-70 % relative humidity
- 14 days in total: 24 h for demoulding + 13 days at room temperature and 100 % relative humidity (sealed specimens)
- 28 days in total: 24 h for demoulding + 27 days at room temperature and 40-70 % relative humidity \rightarrow verify if ITS₂₈ > 0.75 MPa
- 28 days in total: 24 h for demoulding + 27 days at room temperature and 100 % relative humidity (sealed specimens) \rightarrow verify if ITS₂₈ > 0.75 MPa

5.4.3 Results

For evaluation the effect of curing conditioning on the indirect tensile strength (ITS), cold recycled mixtures using bituminous emulsion or foamed bitumen and cement (as described in paragraph 4.2.1) were compacted by static compaction, applying 5 MPa, according to the method generally used in Czech Republic.

The ITS results of the specimens versus conditioning times applied with varied moisture content and temperature conditioning are plotted in Figure 32.







Figure 32: Overview results for ITS of a bitumen emulsion-cement cold mix and a foamed bitumen-cement cold mix

Figure 33 illustrates ITS values after 14 days under conditions with 40 %, 55 % and 70 % humidity at 20°C and under room conditions for cold recycled mixture containing either foamed bitumen or bituminous emulsion besides cement.



Figure 33: Comparison ITS after 14days for different curing conditions

For all conditioning procedures conducted at 20°C a strength increase can be observed with conditioning time.

For these mixtures, which contained a high cement content (3 %), the raising of the conditioning temperature from 20°C to 60°C didn't a ffect the strength development in the first three conditioning days significantly, as indicated by similar ITS values obtained for the 20°C conditioned specimens and 60°C conditioned specimens a fter 3 days. For the further interpretation of the results it has to be taken into account, that the tested material contained 3 % cement which is significantly more than cold emulsion mixes (without cement or with small amounts of cement) usually applied in Portugal or Spain, from where the 60°C conditioning procedure was adapted. Besides, in those Southern European countries, it is also common to leave the specimens on the mould for 1 day, previously of demoulding and submitting to accelerate curing at 50°C or 60°C.



The time-dependent strength increase with similar results in dry (40 %) and humid (70 %) conditions shows the predominately effect of cement hardening due to hydration.

5.4.4 Main conclusions

The controlled humidity in climate chamber conditioning has only a limited effect on the resulting ITS for mixtures with high cement content. In tendency, an increase of humidity at constant temperature (20 ± 2) °C from 40-70 % will re sult in a decrease of ITS.

5.5 Experimental study B4

<u>Main objective</u>: Assessment of moisture during specimen curing for cold recycling, namely for sealed and unsealed test specimens

<u>Used binder(s)</u>: Bituminous emulsion, foamed bitumen and intermediate content of cement (1.5 %)

5.5.1 Selected materials

For further determining the effect of moisture in terms of rather uncontrolled room moisture as well as the moisture condition resulting by specimen sealing in a plastic bag, the mix variations as summarised in Table 21 were prepared.

For this study, the reclaimed asphalt as sampled from a stockpile of milled road materials (see sections describing study A3 and study B3) was complemented by 5 % of inactive limestone filler.

Water was added to the samples in order to achieve a constant water content of 5 %.

Sample	B3-E2.1	B3-E1.2	B3-F2.0			
Bituminous binder	Bitumen emuls	Foamed Bitumen (50/70)				
Bitumen content, % (emulsion content)	2.1 (3.5)	1.2 (2.0)	2.0 (n.a.)			
Cement content, %	1.5 (CEM I 42.5 N)					

Table 21: Samples in curing study B3

5.5.2 Description of the study

After mix preparation, cylindrical specimens (diameter 150 mm) were compacted by applying a constant force of 50 kN for 3 minutes resulting in a specimen height of 90 \pm 3 mm.

After compaction, the specimens were stored in their moulds for one day in laboratory room conditions. After demoulding the specimen curing conditioning was applied.



For each sample mix, one set of specimen was sealed by a plastic bag, whereas another set was left unsealed. Both sets of specimens were stored in laboratory room conditions (22 ± 3 °C; 40-70 % relative humidity), see Figure 34. For the curing, the specimens were stored for 7, 14 and 28 days.



Figure 34. Storing of sealed (right) and unsealed (left) specimen

After the curing time, the sealed specimens were taken from the plastic bags. The dimensions of the specimens were measured and then they were transferred to a temperature control chamber for temperature conditioning to 15 °C. After 3 \pm 1 h the indirect tensile strength of the specimens were tested and the moisture content was evaluated from the tested specimens.

For each mix sample, curing procedure and curing time, three specimens were tested.

5.5.3 Results

The results of the experimental curing study B3 are summarised in Table 22. For each specimen the resulting void content and its indirect tensile strength which was evaluated at the test temperature of 15 $^{\circ}$ C as well as the mean of three experiments are given. The resulting indirect tensile strength values are plotted in Figure 35.

Sealing of the specimen in a plastic bag generally results in significant lower indirect tensile strengths for all mixtures compared to unsealed curing. Only the foamed bitumen mix tested after a curing time of 28 days shows higher strength values in the sealed specimen compared to the unsealed specimen. Though, whereas all other samples indicate an increasing strength with curing time, the strength of the foamed bitumen sample cured unsealed is reduced between 14 and 28 days. This may be a sign for irregular results.

The moisture content of the specimen after the curing are plotted in Figure 36. The initial moisture content of the cold recycling mixture was defined to 5 %. After compaction, the moisture content was not evaluated, therefore, the moisture content of the specimens at the beginning of the curing was not evaluated.



The sealed specimens showed after the indirect tensile strength tests moisture contents between 2.0 and 3.5 %. Here no relevant time effect on the moisture content can be observed. On the other hand, the specimens, that were cured unsealed, indicate significantly lower moisture contents of about 1.0 %.

nc			Curing time										
onditie	ple	2 7 days			14 days			28 days					
ring c	sam	voids	s [%]	ITS [MPa]		voids	s [%]	ITS [MPa]	voids	s [%]	ITS [MPa]
Cu		Individual value	mean	Individual value	mean	Individual value	mean	Individual value	mean	Individual value	mean	Individual value	mean
	DO	12.3		0.562		12.4		0.617		12.9		0.651	
	БЭ- Е2 1	12.1	12.1	0.544	0.551	12.8	12.5	0.598	0.602	12.5	12.8	0.613	0.656
	L2.1	11.7		0.546		12.4		0.592		13		0.705	
g	D2	15		0.459		16.3				16.9		0.493	
sale	В3- F1 2	15.4	15.3	0.451	0.455	17.3	16.7	0.5	0.473	17.7	17.4	0.535	0.526
Š	L1.2	15.5		0.455		16.6		0.446		17.6		0.549	
	БЭ	16.3		0.471	15		0.516		16		0.638		
	Б3- F2 0	22.9	18.8	0.351	0.413	14.2	14.7	0.47	0.495	15.5	16.3	0.578	0.590
	12.0	17.2		0.416		14.9		0.499		17.4		0.554	
	D 2	11.8		0.741		13.1		0.694		14.8		0.816	
	E2 1	11.5	11.9	0.712	0.721	13.1	13.2	0.792	0.760	14.7	14.6	0.931	0.882
	L2.1	12.4		0.709		13.3		0.793		14.2		0.898	
led	БЭ	15.8		0.553		16.4		0.697		16.6		0.711	
sea	БЭ- F1 2	15.2	15.4	0.544	0.538	16.8	16.6	0.65	0.664	17.6	17.3	0.641	0.670
Un	L 1.2	15.2		0.518		16.6		0.644		17.8		0.659	
	БЭ	13.8		0.54		14.4		0.641		17.0		0.516	
	БЭ- F20	14.5	14.3	0.539	0.533	16.5	15.8	0.575	0.600	16.7	17.2	0.541	0.539
	12.0	14.7		0.521		16.3		0.584		17.8		0.559	

Table 22: Results of curing study B3



Figure 35: Effect of curing conditions (sealed / unsealed) on indirect tensile strength





Figure 36: Effect of curing conditions (sealed / unsealed) on moisture content of the specimens

The dry conditioning (unsealed specimens at room conditions, i.e. 22 ± 3 °C & 40-70 % relative humidity), which resulted in moisture contents of approximately 1 %, led to significantly higher strength values when compared to severe moist curing conditions (sealed specimens from 7 days to 28 days, i.e. about 90 % of relative humidity). For the tested cement content of 1.5 %, a higher strength provided by improved cement hydration (which is usually stronger with increased moisture content) can't compensate, in such severe moisture conditions, for the reduction of the strength provided by the bituminous binder (since an increase water content in the mixes hinders the coalescence of the bitumen particles after emulsion breaking or foamed dissipation).

5.5.4 Main conclusions

The comparison analysis of moisture curing conditions (unsealed or sealed specimens during 7-28 days) for three cold recycled mixtures with 1.5 % of cement showed that a faster elimination of water content after the construction of the cold recycled material allows for higher strengths of the mixture due to improved bituminous binder curing. In the case of relatively low water contents, the increased strengthening capacity provided by the bituminous binder seems to be able to compensate for a possible decrease of the strength provided by the cement due to worst conditions of hydration. Therefore, in extremely severe moisture conditions (simulated by unsealed specimens over long periods) seems to be necessary to add higher cement contents in order to reach similar strength levels to that obtained in drier moisture conditions.

5.6 Experimental study B5

Main objective:Evaluation of the effect of the accelerated curing procedure of curing
unsealed test specimens at 50° for 3 days, after o ne day in the mould

Used binder(s): No additional binders have been used



5.6.1 Selected materials

To evaluate the effect of accelerated curing procedure for cold recycled mixes with bituminous binder and no cement or small amounts of cement, which was based on Spanish and Portuguese experience, a cold recycled mix containing only reclaimed asphalt material and mixing water was designed. This mix contains 97.5 % of RAP (Reclaimed Asphalt Pavement) and 2.5 % of mixing water. In addition, grading curve of used RAP was determined as well and is presented in Figure 37. Used RAP fulfils the Czech criteria given by the grading curve envelope for cold recycled mixes containing bituminous and hydraulic binder.



Figure 37: Reclaimed asphalt grading curves and cold recycled mixtures limits

5.6.2 Description of the study

For the purpose of verification, whether accelerated curing at 50°C can really correspond to the real conditions, a simple comparison has been done using cold recycled mix containing only RAP to exclude the effects of binders. Therefore no other bituminous or hydraulic binders were used for the mix designs and testing.

Curing procedure	Bulk density (Mg/m³)
no curing – 4 hours (40-70%, 15℃)	
standard curing – 24 hours (90-100%, 20℃) + 6 days (40-70%, 20℃)	2.016
standard curing – 24 hours (90-100%, 20℃) + 13 days (40-70%, 20℃)	2.016
standard curing – 24 hours (90-100%, 20℃) + 20 days (40-70%, 20℃)	2.011
standard curing – 24 hours (90-100%, 20℃) + 27 days (40-70%, 20℃)	2.013
standard curing – 24 hours (90-100%, 20℃) + 6 days (40-70%, 20℃) + 7 days in water	2.021
accelerated curing – 24 hours (90-100%, 20°C) + 3 d ays (40-70%, 50°C)	2.041

Table 23:	Comparison	of curing	procedure and	related b	ulk density



From the mix cylindrical specimens with 65 mm height and 150 mm diameter were prepared and compacted in same way like for standard cold recycled mixes (static pressure compaction). The specimens were cured according to the procedures described in Table 23.

Two types of testing were conducted – stiffness modulus by IT-CY test method and indirect tensile strength. In both cases 15° was the testing temperature.

5.6.3 Results

Results are summarized in Figure 38 and Figure 39, respectively to stiffness tests and to indirect tensile tests, showing the impact of different curing periods and the difference between accelerated and standard curing containing values of stiffness and ITS after 7, 14 and 28 days.



The impact of accelerated curing is clearly demonstrated in those Figures.

Figure 38: Stiffness results for test specimens cured for different period at 15°C



Figure 39: Indirect tensile strength results for test specimens cured for different period at 15°C

5.6.4 Main conclusions

From the presented results, it is obvious that in the present case the accelerated curing doesn't correspond to the traditional curing procedure for short-term properties and the acceleration effect might be overestimated. The key effect in this respect might be the partially reactivated binder in the RAP, which is softened at the applied curing temperature of



50°C and the bitumen is helping to bound individual particles better together since probably reaching the range of its softening point. Therefore the used mix consolidates better showing improved bonds between the aggregate particles. Of course this usually doesn't happen during a few days within the pavement. For bituminous materials it is known that their viscous material properties allow the application of time-temperature superposition principle. Therefore, the same effects visible at high temperatures within small time intervals can also be observed at lower temperatures but substantially increased time period. This is known for permanent deformation as well as stiffness properties. Therefore, also the strength development due to long-term bonding of particles as observed in the test for the curing procedure of 50°C will also develop at lower temperature reduction of 30°C will result in an increase of time of the factor 2700. For this reason additional assessments are necessary and further cut-down of the temperature applied during accelerated curing (e.g. 30°C or 40°C) might be reasonable to avoid any effects related to softened bitumen in countries where reclaimed asphalt materials commonly have relatively low softening temperatures.

5.7 Experimental study B6

<u>Main objective</u>: Comparison of maximum density testing procedure done in different states of the tested material (wet, air dried, oven dried, *etc*.)

<u>Used binder(s)</u>: Cationic slow-breaking bituminous emulsion C60B5 (former C60B7) or foamed bitumen using 70/100 pen grade bitumen, without or with 1 % or 3 % cement

5.7.1 Selected materials

For the cold recycled mixes tested within this experimental study reclaimed asphalt material of 0/22 mm grading from the Středokluky (CZ) mixing plant was applied. The grading curve of the RAP is given in the Figure 37, as shown in study B5. As used binders bituminous emulsion or foamed bitumen has bee used. In some mixes cement CEM II B32.5R according to EN 197-1 was applied as well.

5.7.2 Description of the study

One of the basic parameters, which have to be determined for every asphalt mixture, including cold recycled mixes, is maximum density. It is important to exactly calculate the voids content in a mix. Determination of this parameter follows the standard EN 12697-5 which is applicable to cold recycled mixes as well. Nevertheless, primarily this standard is dedicated to hot asphalt mixes. For cold mixes containing mainly reclaimed asphalt material (RAP) and mixing water the determination might be a bit more complicated. Several mixtures were therefore tested with different types of used binders. Used mix designs are summarized in Table 24.



Mix	A°	В	S	Р
Reclaimed asphalt	90.0	90.0	94.5	93.0
Cement	3.0	3.0	1.0	-
Bituminous emulsion	4.5	-	-	4.5
Foamed bitumen	-	4.5	2.0	
Water	2.5	2.5	2.5	2.5

Table 24. Mix	designs used in cor	nnarability study	of maximum	density testing
	ucalgina uacu ili cui	mparability Study		uchaily lealing

The mixtures were cured and tested by different type of maximum density testing procedure¹:

- from a wet (fresh) mix determination of maximum density from a wet mix immediately after its production;
- from a dried mix determination of maximum density from a dried mix at the day, when it was produced and dried at 105-110℃ until constant mass was reached;
- from a cured mix determination of maximum density from a dried mix one week after producing and cured at laboratory conditions (t=20±2)℃ and relative humidity of 40-70 %;
- from a dried mix + vibration determination of maximum density from a dried mix at the day, when it was produced and dried at 105-110°C un til constant mass was reached + using vibration table during filling the pycnometer by loose material and water.

5.7.3 Results

The results which are shown in the Table 25 illustrates that the highest maximum density was always achieved for the first type of testing, when a wet fresh mix was used. The reason is obvious. A wet mix absorbs better additional water which is mixed with the loose material during maximum density determination. For the mixture S we can see, that vibration contribute to higher and therefore also more accurate maximum density value.

Nevertheless, the most interesting results are given for the comparison of dried mixes tested immediately after the mix was produced and a mix cured for one week. The values for one week cured mix are always significantly higher. If we used this values for determination of voids content (and it might be in many countries probable), we would get more or less significantly different values. These different values can be important in cases, when a mix has to comply with some requirements sets in national or European specifications.

Testing procedure where designed mix is dried after production leads to the lowest maximum density values. In this case the material water absorption is slightly limited (compared to first procedure) and the results will lead to unduly decreased voids content value.

¹ EN 12697-5+A1 states a vibration plate as an equipment. For the volumetric process the test is performed on the plate. The Department of Road Structures CTU unfortunately doesn't have such vibrating plate. The vibrations are made by hands or by a non-standard old table. A process with vibration is therefore mentioned above in bullets, because in this case a special vibrating equipment was used.



Mix	Testing method	Testing method Maximum density (Mg/m ³)		0
	from wet mix	2.458	100 %	
А	from dry mix (age 0 days)	2.411	98 %	-
	from dry mix (age 7 days)	2.423	99 %	
	from wet mix	2.398	100 %	
В	from dry mix (age 0 days)	2.358	98 %	-
	from dry mix (age 7 days)	2.373	99 %	
	from wet mix	2.465	100 %	
Р	from dry mix (age 0 days)	2.392	97 %	-
	from dry mix (age 7 days)	2.427	99 %	
	from wet mix	2.468	100 %	-
S	from dry mix (age 0)	2.416	98 %	100 %
	from dry mix (age 0 days) + vibration	2 426	98 %	100 %

Table 25: Maximum density tests result
--

5.7.4 Main conclusions

Based on the results of this single study, in which different types of cold recycled mixes where used, whereas similar ratios have been found between the three procedures for maximum density testing, it is recommended to carry out this test after 7 days cured cold recycled mix which has been stored uncompacted (loose) and unsealed at laboratory conditions, temperature of (20 ± 2) °C and relative hu midity of 40-70 %. Then the mix is dried at 105-110°C and maximum density is determined using the test procedure defined in EN 12697-5.

5.8 Experimental study B7

<u>Main objective</u>: The work focused on investigation of relation between cement content, curing regime and test temperature on the recycled cold emulsion material mix properties

<u>Used binder(s)</u>: Bituminous emulsion (C60B4) with varying cement contents up to 3.0%

5.8.1 Selected materials

For this investigation material was obtained from an Irish road site where cold recycling was used. Figure 40 shows the grading of the combined surface and granular material obtained from the site. The grading was created by taking in account recycling depth of 300mm where material ratio surface to granular material is 1:2. The Wirtgen cold mix grading envelope (Wirtgen, 1998) was used as a guide in analysing the material grading mix.





Figure 41: Material mix grading

The optimum moisture content of the material was obtained using a procedure based on the standard soils Proctor test, BS 1377- 4: 1990. The moisture content was gradually increased within samples of this material and the corresponding density measured. The results for the test are presented in Figure 42, which shows the dry density - moisture content relationship. From the graph we can conclude that optimum moisture content for the material mix is around 4%. This moisture content was adopted for the material mix designs from all of the five pits as it is just on the dry side of optimum as recommended in the IAN.



Figure 43: Determination of optimum moisture content

5.8.2 Description of the study

Three emulsion mixes were designed, Table 26 summarises the mix designs. The material mix designs are classified in the IAN (NRA, 2011) as Quick Viscoelastic Mixes (QVE) as 3% of cement was included in their design. The mixes were designed both with 3% cement included and also without any cement included. Additional, stiffness and dry strength tests were performed on mixtures containing 1.5% cement.



		Mix Constituents (%)							
Mix	Mix type	Surface Material	Granular Material	Cement	Water/ Moisture	Binder	Emulsion		
А	Emulsion Mix without cement	33.3	66.7	0	4	2.2	3.5		
В	Emulsion Mix with cement	32.8	65.7	1.5	4	2.2	3.5		
С	Emulsion Mix with cement	32.3	64.7	3	4	2.2	3.5		
 Note: 1. All percentages contents are by mass, 2. The emulsion used in the mix is the Irish Tar and Bitumen Suppliers Ltd. product: 'Fuarflex', of the binder specification C60B4. 3. The amount of added water in emulsion mixes, was adjusted per mount of water in the emulsion and moisture content of the surface and granular material. 									

Table 26: Mix Designs density tests results

Mixing Procedure

granular material.

A cold emulsion mixing procedure was used for the study. The mixes were mixed using a Wirtgen foam material mixer at maximum speed of 30 rpm. The reason for the slow speed is that at higher mixing speeds the emulsion breaks down and the binder separates from the water, thus inhibiting adequate coating of the aggregates.

The following steps were taken during the mixing procedure:

- i. dry aggregates are pre-mixed for about 1 minute;
- ii. half of the water is added into the mix;
- iii. the emulsion is gradually added to the mix;
- iv. the remaining water is added to the mixture.

Compaction Procedure

All of the specimens were compacted in accordance with EN 12697-31:2007. A Coopers Technology gyratory compactor was used to compact the test specimens. Figure 44 illustrates the kneading motion used in the compaction process, whereby a simultaneous static compression and shearing force, resulting from the rotation of the top surface of the mould, is used to compact the mixture.

The static compaction pressure was set at 0.6 MPa with an angular velocity of 30 gyrations per minute and the gyratory angle set at 1.25°. A s et number of gyrations were used as the compaction control target, in this case 100 gyrations. For this study the cylindrical test specimens were compacted to the target dimensions of 150 mm in diameter and 75 mm in height. Slotted moulds were used in the compaction process in order to allow for the drainage of the excess moisture and thus better compaction. The same weight of material, 3 kg, was placed into the mould for all of the specimens. Six specimens of each mix were produced, in total 30 specimens. After compaction, test specimens were left in the mould to cure for 24 hours. The test specimens were then extruded, and their dimension and weights were recorded. The specimens were then ready for curing.





Figure 45: Basic gyratory compaction concept with insert of Coopers Technology gyratory compactor (P_G =compaction pressure (0.6MPa), β_G =gyratory angle (1.25°), ω_G =angular velocity (30 gyrations/minute)

Curing Procedure

Two different curing programmes were adopted for curing test specimens. For water sensitivity test specimens (WP2) a Portuguese and German curing procedure for emulsion mixtures were adopted. The Portuguese curing procedure, of 3 days at 50° C – unsealed, was adopted for emulsion mixtures without cement. The German curing procedure, of 14 days at 20° C – unsealed, was adopted for emulsion mixtures with cement (3 %). Additional curing procedure, of 7 and 14 days at 20° C – unseal ed, for emulsion tests specimens with 1.5% cement and without cement was adopted. This procedure was used for test specimens used for the investigation of the dry indirect tensile strength test (WP1).

5.8.3 Testing

Three tests were used to evaluate the mixes performance and one was used to determine its maximum densities, there were:

- Indirect tensile stiffness modulus test (ITSM) -EN 12697-26: 2012;
- Water sensitivity test (WST) EN 12697-12: 2008;
- Indirect tensile strength tests (ITS) EN 12697-23: 2003;
- The maximum densities test EN 12697 5: 2009.

Indirect Tensile Stiffness Modulus Test (ITSM)

The non-destructive ITSM test was conducted which complied with EN 12697-26: 2012. The Cooper Research Technology NU-10 testing apparatus with a pneumatic close loop control system was used. Two linear variable differential transformers (LVDT) were used to measure the horizontal deformation. After the curing period, test specimens were removed from the sealed plastic bags and had their dimensions and weights were re-recorded, they were placed in a temperature controlled chamber for ITSM test temperature curing at 20°C, for three hours prior to testing. The stiffness value was recorded on two diameters orientated at 90° to each other, and an average of these two values was reported as the specimen stiffness. The results are summarised in Table 28.



Water Sensitivity Test (WST)

The water sensitivity test was performed in accordance with European standard EN 12697-12: 2008. After the curing period, the set of six specimens were then divided into two subsets of three specimens. The first set was stored in a temperature control chamber at 20°C. The second set was placed under distilled wat er and subjected to a vacuum of 6.7kPa for 30 minutes, and then left submerged in water for another 30 minutes at atmospheric pressure. After this, the wet conditioned set of specimens was placed into a water bath at 40°C for 72 hours. Both sets of test specimens were then conditioned at a test temperature of 25°C or 15°C for three hours prior to testing. The dry set was conditioned in a temperature controlled air chamber, and the wet set conditioned in a temperature controlled water bath.

Indirect Tensile Strength Test (ITS)

A control testing system was employed to complete the Indirect Tensile Strength Test (ITS) in accordance with EN 12697-23: 2003. The ITS test is conducted by applying a vertical compressive strip load to a cylindrical specimen. The load is distributed over the thickness of the specimen through two loading strips at the top and bottom of the test specimen.

The critical stresses and strains within the indirect tensile specimen are computed using following analytical formulation based on linear elastic theory:

$$\sigma_{xy}(\max) = \frac{2P}{\pi dt} \tag{1}$$

where P is load (N), d specimen diameter (mm) and t specimen thickness/height (mm).

Using equation (1), the maximum tensile strengths of both wet and dry conditioned test specimens were calculated and an indirect tensile strength ratio was calculated for each. Table 29 summarises the ITS and ITSR test results.

Maximum Density Test (MDT)

The maximum densities test were conducted on the laboratory compacted specimens in accordance to the EN 12697-5: 2009. The procedure for this process was that the maximum density of the cold asphalt test specimens was determined from the volume of the sample without voids and from its dry mass. The volume of the sample was measured as the displacement of water by the sample in a pycnometer. After the sample was placed in the pycnometer and it had been filled with water to within 30mm of the top of it, a vacuum was applied to it for 15min while the pycnometer was vibrated on a vibrating table to ensure the evacuation of air from inaccessible pores. The pycnometer was then entirely filled with water and weighted. The maximum density was then calculated. This test was employed in order to determine void content in the mix. Table 27 summarises the results of the, mixes moisture content for all mixes, between 1.0% and 1.6%. Mixtures containing cement did not show lower moisture content after conditioning period. Air voids content was lower than



expected (4%) though during the compaction it was noticed that small over compaction was achieved this was due to the targeted number of gyrations rather than density value. The results show that cement content in the mix did not affect air void content either.

Mix	Mix type	Curing method	Moisture content (%)	Max density (kg/m³)	Bulk density (kg/m³)	Air voids content (%)
Δ	Emulsion mix without	7 days at 20℃	1.6	2351	2279	3.1
A cement	cement		1.0	2287	2210	3.4
С	Emulsion mix with 3% cement	14 days at 20℃	1.0	2303	2236	2.9
в	Emulsion mix with		1.5	2343	2278	2.8
В	1.5% cement	7 days at 20℃	1.4	2291	2204	3.8

Table 27: Max and Bulk densities and air voids content of test specimens.

5.8.4 Results

The testing programme was split in two parts, water sensitivity tests (reported in WP2) and dry indirect tensile strength test (reported below). ITSM tests were performed on all of the specimens at the end of their curing programme. Table 28 summarises the stiffness (ITSM) values of all mixtures at varying curing programmes. The results illustrates that the emulsion mixes without cement reached the optimum stiffness value at curing procedure of 3 days at 50°C. However, as the Irish summer environmental co nditions are not same as in continental Europe, where more rain prevailing conditions with temperature in range of 20°C, ITSM results at curing temperature of 20°C would be more acceptable. The results of test specimens conditioned at 7 and 14 days at 20°C show mix stiffness values below one year requirement, of 2000 MPa. However, results of the mix containing 1.5% cement show that stiffness value has been achieved by seventh day of curing at 20°C. Mix containing 3% cement illustrated very high stiffness values. These high stiffness values could lead to highly brittle material and such high cement content in the mix would not be recommended.

Mix	Mix type	Curing Method	ITSM (MPa) @ 20 ⁰C
A		3 days at 50℃	2254
	Emulsion Mix without cement	7 days at 20℃	1302
			1975
С	Emulsion mix with 3% cement	14 days at 20℃	6258
В	Emulsion mix with 1.5% coment		3526
		7 days at 20℃	2911



Mix	Mix type	Curing method	Test temperature (℃)	ITS (MPa)	ITSR (%)
			25	0.28 _(wet)	80
Δ	Emulsion mix without	3 days at 50℃	23	0.35 _(dry)	
~	cement		45	0.58 _(wet)	
			15	0.74 _(dry)	
			25	0.47 _(wet)	85
C	Emulsion mix with 3% cement	14 days at 20℃	20	0.65 _(dry)	
C			15	0.83 _(wet)	
			15	0.94 _(dry)	00

Table 29: Water sensitivity (ITS and ITSR) test results

The water sensitivity tests and indirect tensile strength tests were performed at two different temperatures, a standard test temperature of 25°C and lower temperature of 15°C.

Table 29 shows the water sensitivity test results. The results show a very good ITSR values for both mixes at all test temperatures slightly higher at 15°C test temperature for mix "C" (mix containing 3 % cement). For the mix without cement ITSR values were very close. However, ITS values are much higher at lower test temperature. This was expected, as at lower temperature bituminous material will experience more brittle like behaviour. This was confirmed by the additional ITS test on the mix A and B (mix A = 0 % cement and mix B = 1.5 % cement), see Table 30. The results also illustrate the influence of the curing time period and cement on the mix strength. Where the increase in the curing time period and cement with the ITSM test results.

Table 30: ITS test results

Mix	Mix type	Curing method	Test temperature (°C)	ITS _(dry) (MPa)
A		7 days at 20⁰C	25	0.24
	Emulsion Mix without coment		15	0.52
		$14 \text{ days at } 20^{\circ}\text{C}$	25	0.31
		14 days at 20 C	15	0.61
В	Emulsion Mix with 1.5% cement	7 days at 20%	25	0.33
		7 days at 200	15	0.63
		14 days at 20%	25	0.41
		14 days at 200	15	0.74

5.8.5 Main conclusions

The following points can be made based on the work described above:

• The emulsion mix with reduced binder/emulsion content (2.2 % - BC /3.5 % - EC) performs well.



- The Portuguese curing period for emulsion mixtures without hydraulic binder (cement) shows that mix can reach yearly stiffness value of 2000 MPa. However, a caution must be exercised as this curing period does not reflect typical environmental conditions in Ireland. The German curing period for cold recycled emulsion mixtures with and without hydraulic binders (cement) of 14 days at 20 °C, would be more realistic and suitable curing period for Irish studies.
- All of the mixtures illustrated good stiffness values, where mix without cement have lowest value and mix with 3 % cement highest.
- The mix with 3 % cement was considerably stiffer than the other materials, and the impact of this should be considered.
- Both mixtures with and without cement showed good ITSR values, at and over 80 %.
- ITS results showed increase in material strength with:
 - o increase of curing period (7 14 days);
 - o decrease of the test temperature (25 $^{\circ}$ C 15 $^{\circ}$ C);
 - o inclusion of cement in the mix.

5.9 Experimental study B8

<u>Main objective</u>: This study focused on evaluation of the influence of different curing procedures on strength and stiffness characteristics of cold recycled mixes using foamed bitumen, bituminous emulsion or combination with cement.

<u>Used binder(s)</u>: Bituminous emulsion (C60B7) or straight-run bitumen 70/100 with varying cement contents up to 3.0 %.

5.9.1 Selected materials

The presented experimental study comprised the use of the following materials:

- Screened reclaimed asphalt of 0-22 mm and 0-11 mm grading
- Cationic slow-breaking bituminous emulsion (C60B7)
- Foamed bitumen using a 70/100 penetration grade bitumen
- Cement (CEM II B 32.5R)

5.9.2 Description of the study

In the present study, the influence of different curing laboratory procedures on the strength and stiffness of four different cold recycled mixtures is evaluated. The addressed curing procedures were the ones described in section 2.3.4 for the different countries. As referred before, most available procedures require 7 or 14 days of curing at 18-40°C, previously to testing. In Spain and Portugal however, an accelerating curing procedure of 1 day in the mould at room temperature followed by three days (demoulded) at 50°C is of current practice for cold recycling mixtures using bitumen emulsion. In order to test specimens of the same



age, i.e. with 7 or 14 days, test specimens cured according to the Spanish/Portuguese curing procedure for 4 days were again stored under (20±2)℃ for the remaining time.

Presented assessments were performed on cylindrical specimens prepared with four mix designs. Separately recycled mixes with reclaimed asphalt of 0-22 mm and 0-11 mm grading were used. The designs differ in the bituminous binder type and in the presence or absence of a hydraulic binder. The compositions of the individual versions are indicated in Table 31. Mix A and E contains cationic slow-breaking bituminous emulsion C60B7 commonly used in the Czech Republic. In mixes B and F, the bituminous emulsion was replaced by foamed bitumen (with 70/100 straight-run bitumen) prepared with Wirtgen WLB10S laboratory equipment. These mix types used cement CEM II/B-M 32.5 R, according to European product standard EN 197-1. The composition of mixes C and G corresponds with the composition of mixes A and E which contain cationic bituminous emulsion; there is not hydraulic binder though. The same applies to mixes D and H, which were stabilised by foamed bitumen.

Identically for individual options, a set of selected characteristics was verified which included both determination of bulk densities and calculation of void content in the mix, and verification of the initial moisture content of the mix and determination of strength characteristics by means of an indirect tensile stress test. Stiffness was determined at the same time by means of the non-destructive method of indirect tensile stress test (IT-CY method listed in European standard EN 12697-26).

Four typical variants covering the possible designs used in road practice not only in the Czech Republic were chosen. Tables 32 and 33 state the basic volumetric characteristics – densities – with respect to the type of mix and curing method applied to the individual test specimens. The void contents of the cold recycling mixes was then calculated based on the bulk weights of the specimens and maximum bulk weight of the mixes.

Mix	R	٩P		Water		
IVIIA	0-22 mm 0-11 mm		Cement	Bituminous emulsion	Foamed bitumen	content
A ⁽¹⁾ /E ⁽²⁾	91.0 %		3.0 %	3.5 %	-	2.5 %
B ⁽¹⁾ /F ⁽²⁾	90.5 %		3.0 %	-	4.5 %	2.0 %
C ⁽¹⁾ /G ⁽²⁾	94.0 %		²⁾ 94.0 % - 3.5 %		-	2.5 %
D ⁽¹⁾ /H ⁽²⁾	93.	5 %	-	-	4.5 %	2.0 %

Table 31: Designed experimental	cold recycled mixes
---------------------------------	---------------------

(1) Mixture produced with RAP 0-22 mm; (2) Mixture produced with RAP 0-11 mm

Within the framework of the comparisons performed, bulk density is an important parameter which the remaining values measured relate to. As can be noted, maximum density of the cold recycled mixes with reclaimed asphalt 0-22 mm whether using emulsion or foamed bitumen with and without cement (indicated in brackets) is of the same order of magnitude if cement is used.



Mix	Type of curing	Maximum density [kg/m ³]	Bulk density [kg/m ³]	Voids content [%]
	Czech Republic		2 124 (2 124)	12.3 (12.2)
A (C)	UK and Ireland		2 102 (2 102)	13.3 (13.2)
mixes with	France	2 422 (2 421)	2 104 (2 104)	13.2 (13.1)
cement (or	Australia	2 423 (2 421)	2 097 (2 097)	13.5 (13.4)
cement)	South Africa		2 140 (2 140)	11.7 (11.6)
	Portugal		2 093 (2 071)	13.6 (14.5)
B (D)	Czech Republic		2 116 (2 116)	9.3 (9.5)
Foamed	UK and Ireland		2 152 (2 152)	7.8 (8.0)
bitumen	France	2 241 (2 246)	2 175 (2 174)	6.8 (7.1)
mixes with cement (or	Australia	2 341 (2 340)	2 115 (2 115)	9.3 (9.5)
without	South Africa		2 121 (2 121)	9.1 (9.3)
cement)	Portugal		2 085 (2 072)	10.6 (11.3)

Table 32: Basic volumetric characteristics; cold recycled mixes with RAP 0-22

Note: The values in brackets () correspond to mixtures where only bituminous binder was used, i.e. with no cement.

Table 33: Basic volumetric characteristics; cold recycled mixes with RAP 0-11

Mix	Type of curing	Maximum density [kg/m ³]	Bulk density [kg/m ³]	Voids content [%]
	Czech Republic		2081 (2137)	13.5 (11.2)
E (G)	UK and Ireland		2121 (2157)	11.8 (10.4)
mixes with	France	2 406 (2 407)	2125 (2078)	11.7 (13.7)
cement (or without cement)	Australia	2 400 (2 407)	2100 (2064)	12.7 (14.2)
	South Africa		2153 (2149)	10.5 (10.7)
	Portugal		2058 (2122)	14.4 (11.9)
F (H)	Czech Republic		2118 (2120)	9.0 (8.6)
Foamed	UK and Ireland		2122 (2099)	8.8 (9.5)
bitumen	France	2 224 (2 228)	2111 (2118)	9.3 (8.7)
cement (or without cement)	Australia	2 334 (2 328)	2107 (2100)	9.4 (9.5)
	South Africa		2136 (2099)	8.2 (9.5)
	Portugal		2118 (2126)	9.0 (8.4)

Note: The values in brackets () correspond to mixtures where only bituminous binder was used, i.e. with no cement.

For cold recycled mixes with RAP of 0-11 mm grading, same conclusions can be achieved. Nevertheless, in this case, some slightly differences between the bulk density and therefore between the void content of mixtures with or without cement can be noticed.

For a complex overview of all results Figure 46 summarizes void contents for designed and tested mix variants divided into groups according to curing method which has been used. It should be pointed out, that bulk densities have been determined always before the indirect tensile strength test. Therefore residual water captured in the test specimens plays an important role as well.







5.9.3 Results

Curing method impact on indirect tensile strength

The determination of indirect tensile strength was performed in accordance with the method stipulated by technical conditions TP208 (Czech Ministry of Transportation, 2008). Cylindrical test specimens of 150 mm diameter and minimum 60 mm in height were used. As a standard, the test is taken at 15°C. The measurements *per se* were taken after 7 and 14 days of curing. In total, strength was determined for four types of mixes with different curing methods and times. The average values of the indirect tensile strengths (ITS) obtained are indicated in the following Tables 34 and 35. The indirect tensile strength values reach fundamentally different results in comparison to the findings related to the bulk densities and void contents that depend on the curing methods chosen. During the research, this presented the motivation to carry out extensive assessment. As ensues from the results presented, the weather conditions (simulated by the curing method) have significant impact on the mix properties and the selection of the right method could have a distinctive influence on material characteristics.

If ITS results are compared to the requirements given in TP208 the following findings can be noted. Based on use binder following is required:

- In the case of bituminous emulsion or foamed bitumen application, the minimum indirect tensile strength R_{it} required after 7 days of curing amounts to 0.3 MPa;
- In the case of using a combination of cement and emulsion or foamed bitumen, the value falls within the range of 0.3 MPa to 0.7 MPa.

When a comparison is made for mixes with RAP 0-22 and same bituminous stabilising agent (emulsion or foamed bitumen, with or without cement), the use of 3 % cement is noticed to increase the ITS achieved in contrast to the mixes without hydraulic binder (cement). In the case of the accelerated Portuguese method (1 day in the mould + 3 days @ 50°C), the mix achieved almost identical results with and without cement which is caused by a different effect during hydration. In the cases of the remaining curing methods, the least increase of strength due to cement was recorded for the Australian method (3 days @ 40°C) which



delivered an improvement of 35 % in comparison to the mix without cement after 7 days and of 27 % after 14 days. The most marked difference was delivered by the Irish/British method (28 days @ 40 °C for emulsion mixes with no cement & 90-100 % humidity) where the application of 3 % cement resulted in ITS increase by over 400 % after 7 days; however, after 14 days, the value only amounted to 79 %. The Czech curing method (2 days @ 20°C & 90-100 % humidity + storage @ 20°C & 40-70 % humi dity) applied to the mix with added cement improves ITS after 7 and 14 days, on average roughly by 90 %. If indirect tensile strengths after 14 days of curing are compared, it should be concluded that the most beneficial (i.e. with the highest increase) percentages, is the application of the Czech curing method with an improvement of 85 % after 14 days' curing. The decrease of indirect tensile strength of mix A for the curing method according to the French approach seems illogical within the overall comparison of methods.

Type of	Mix A - Emulsion mix & Mix B - Foamed mix, with 3 % cement		Mix C - Emulsion mix & Mix D - Foamed mix, without cement		ITS improvement A vs. C and B vs. D [%]	
curing	ITS [MPa]	ITS [MPa]		
	7 days	14 days	7 days	14 days	7 days	14 days
	Bitur	ninous emulsi	on stabilized	mixes (A and	C)	
Czech Republic	0.67	0.87	0.35	0.47	93	86
UK and Ireland	0.83	1.00	0.16	0.56	405	79
France	0.84	0.65	0.44	0.51	92	29
Australia	0.85	0.87	0.63	0.68	35	27
South Africa	0.90	0.83	0.45	0.54	101	52
Portugal	0.86	0.85	0.82	0.88	4	-4
	Foa	amed bitumen	stabilized m	ixes (B and D)	
Czech Republic	0.65	0.84	0.26	0.41	150	105
UK and Ireland	0.93	1.17	0.22	0.49	325	140
France	0.88	0.95	0.33	0.46	163	106
Australia	0.91	0.95	0.52	0.63	74	51
South Africa	0.84	0.87	0.48	0.59	76	49
Portugal	1.12	1.14	0.87	1.06	28	7

Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of indirect tensile strength (ITS) for mixes with RAP 0-22 mm; Table 34: Results of	=15	C
---	-----	---

Comparing cold recycled mixes using foamed bitumen, the addition of cement seems to improve the indirect tensile strength again. In the case of some curing methods, the improvement is quite significant. Again, like in the preceding case, it is visible that the highest ITS improvement with cement application was achieved by the British curing method where the strength exceeded the mix with no hydraulic binder by more than 300 % after 7 days' curing; in the case of the 14 days strength, the value was lower but still improved by approx.



140 %. The indirect tensile strengths measured on specimens cured by the Czech and French curing methods were almost identical; after 7 days, strength improved by roughly 150 % and after 14 days, by an average of 100 %. In the case of the Portuguese method, only a small increase by the effect of cement addition to the cold recycling mix was noticed.

In the case of comparing cold recycled mix variants with RAP 0-11 mm results are similar. Influence of cement has a positive effect on gained strength values and mixes with hydraulic binders again reach higher values. The assumption made for mixes with RAP 0-22 mm was confirmed. Focusing on comparison of recycled mixes containing bituminous emulsion and cement it can be stated that highest values were gained after British curing method. Cement improved the ITS values after 7 days curing by 263 % and after 14 days curing there was additional slight increase in strength values. Czech and Australian curing methods have shown identical values for mixes with cement addition. On the other hand the least suitable curing method if cement is applied might be the Portuguese accelerated curing procedure, where test conditions are not introducing best environment for cement hydration.

Type of curing	Mix E - Emulsion mix & Mix F - Foamed mix, with 3 % cement ITS [MPa]		Mix G - Emulsion mix & Mix H - Foamed mix, without cement ITS [MPa]		ITS improvement E vs. G and F vs. H [%]	
	7 days	14 days	7 days	14 days	7 days	14 days
	Bitum	ninous emulsi	on stabilized	mixes (E and	G)	
Czech Republic	0.47	0.58	0.37	0.49	26	17
UK and Ireland	0.93	1.05	0.26	0.27	263	291
France	0.67	0.82	0.38	0.52	76	59
Australia	0.84	0.91	0.65	0.76	31	19
South Africa	0.78	0.81	0.55	0.61	41	34
Portugal	0.62	0.64	0.55	0.65	14	-2
	Foa	amed bitumer	stabilized m	ixes (F and H)		
Czech Republic	0.73	0.87	0.39	0.52	87	66
UK and Ireland	0.90	1.11	0.30	0.73	204	52
France	0.74	0.79	0.40	0.53	85	49
Australia	0.93	0.95	0.62	0.67	51	41
South Africa	0.97	1.04	0.48	0.55	101	88
Portugal	1.08	0.82	0.86	0.64	26	28

Table 35: Results of indirect tensile strength	I (ITS) for mixes with RAP 0-11mm; T=15℃
--	--

If focusing only on recycled mixes with foam and cement, improvement is reached by the British curing method (increase of ITS after 7 days curing about 204 %). In the case of 14 days strength properties the improvement was smaller and shoed only 52 %. On the other



hand curing method used in South Africa increased the strength value after 7 days curing about 101 % and after 14 days curing about 88 %. For the latter conditioning time this method seems to be slightly more promising. Almost same improvement level was reached for Czech and French curing method, showing about 86 % increase in strength value after 7 days curing. For 14 days curing the Czech method showed a little bit higher increase level (about 66 %).

When assessing the ITS after 14-day curing from the point of view of curing regardless of the bituminous binder applied, the methods used in Australia, South Africa, CZ and Portugal basically deliver comparable results. Minor differences appear in the case of mixes with foamed bitumen. With respect to cold recycling mixes with no cement, the approaches applied in Portugal, South Africa and Australia seem to be comparable. The British method showcases the effect of water which cannot be freely released in the surrounding environment. Primarily the 7-day strengths are low in comparison to the other methods.

Curing method impact on stiffness

Stiffness modulus represents an important characteristic that describes the behaviour of cold recycled mixes and can be used from the perspective of pavement structural design. The requirements applicable to stiffness modules are similar to those of ITS. Czech technical specifications do not require determination of stiffness modulus value and specify no minimum thresholds required; however, the characteristic is examined as a standard within experimental research. Stiffness measurements were taken for all mixes after 7 and 14 days of curing. A summary of the results is given in Tables 36 and 37. The measurements were taken under 15°C using the method of repeated indir ect tensile stress method. Again, the results had to be compared from the point of view of values after 7 and 14 days of curing.

From the perspective of time-development of stiffness modulus, it can be note that there is usually a considerable increase of stiffness between the values after 7 and 14 days; however, a slight decrease was recorded for some curing methods. In the cases of the French and Australian curing methods, a rather significant decrease was found and could have been caused, identically to the Portuguese method, by the heterogeneity of the reclaimed material which constitutes a risk difficult to control. It can be concluded that the values obtained after 7 days' curing give just a rough idea of the final mix properties and, therefore, the values after 14 days of curing should be relied on more since they proved to have greater indicative value.

The results of stiffness copy relatively well the ITS results. Unexpected is the low modulus values for the Czech curing method. Lower values of the British method related to the mixes with no cement just confirm the findings applicable to ITS (negative impact of condensing water). In the case of mixes with cement, it is obvious that the water enhances cement hydration. The high stiffness modules for mixes with cement that were exposed to the Portuguese curing method are illogical from the perspective of curing. The lack of moisture led to poorer results with respect to the cement's limited ability to hydrate. The Australian and South African methods are mutually very well comparable for basically all mix types.



Type of curing	Mix A - Emulsion mix & Mix B - Foamed mix, with 3 % cement		Mix C - Emulsion mix & Mix D - Foamed mix, without cement		Stiffness improvement A vs. C and B vs. D [%]			
	Stiffness mo	odulus [MPa]	Stiffness mo	odulus [MPa]		P		
	7 days	14 days	7 days	14 days	7 days	14 days		
	Bituminous emulsion stabilized mixes (A and C)							
Czech Republic	4 212	4 997	1 594	1 838	164	172		
UK and Ireland	4 926	6 223	897	2 345	449	165		
France	4 486	3 803	2 073	2 108	116	80		
Australia	5 038	4 448	3 359	3 411	50	30		
South Africa	5 169	5 223	2 369	2 893	118	81		
Portugal	5 573	6 633	4 157	5 363	34	24		
	F	oamed bitumer	n stabilized mix	kes (B and D)				
Czech Republic	3 756	4 760	1 082	1 573	247	203		
UK and Ireland	5 174	6 389	1 116	2 108	364	203		
France	4 155	4 380	1 490	1 880	179	133		
Australia	4 920	5 511	2 266	2 575	117	114		
South Africa	4 191	4 492	2 279	2 454	84	83		
Portugal	6 304	6 673	4 698	5 327	34	25		

Table 36: Results of stiffness assessment for mixes A and C; T=15°C

Table 37: Results of stiffness assessment for mixes E and G; T=15 °C

Type of curing	Mix E - Emulsion mix & Mix F - Foamed mix, with 3 % cement Stiffness modulus [MPa]		Mix G - Emulsion mix & Mix H - Foamed mix, without cement Stiffness modulus [MPa]		Stiffness improvement E vs. G and F vs. H [%]	
	7 days	14 days	7 days	14 days	7 days	14 days
	Bitu	uminous emuls	ion stabilized r	nixes (E and G		•
Czech Republic	2 891	3 699	1 252	1 597	131	132
UK and Ireland	5 500	6 385	831	1 044	562	511
France	3 503	4 313	1 204	1 647	191	162
Australia	4 287	5 000	2 513	2 714	71	84
South Africa	4 611	4 165	1 983	2 163	133	93
Portugal	4 064	4 172	2 899	3 091	40	35
	F	oamed bitume	n stabilized mix	xes (F and H)		<u>.</u>
Czech Republic	4 189	5 127	1 749	2 038	139	152
UK and Ireland	4 691	5 679	1 025	2 717	358	109
France	3 655	4 019	1 466	1 953	149	106
Australia	4 538	4 961	2 198	2 329	107	113
South Africa	5 135	5 519	2 270	2 410	126	129
Portugal	6 790	5 144	4 014	2 850	69	81



5.9.4 Main conclusions

As is obvious from the performed assessments, the selected curing method has a major effect on the resulting characteristics of cold recycling mixes. The choice of a suitable method cannot be made on the basis of historic analogies or acceptance of other countries' approaches. At least three perspectives must be taken into consideration. First, whether the mix is based solely on bituminous binders with a maximum of 1.5 % by mass of cement, or whether it is a mix with combined binder where cement plays an important role. Secondly, the approach to the structure of the recycled layer from the point of view of its subsequent overlay - if another layer is laid to cover it soon after the cold recycling completion, the phenomenon can be simulated by enclosing the test specimens sealed by plastic bags as the water content will be mostly retained in the recycled layer and the evaporation of the excess water takes much longer. The third point of view is the region where the cold recycling technology is being implemented, and its characteristic climate. Based on these aspects, the accelerated curing method where the test specimens are put in an air-conditioning chamber at relatively high temperature (e.g. $\geq 40 \, \text{C}$) to provide good drying-up and, consequently, faster bituminous emulsion or foam consolidation, seems appropriate for cases where cement is not used (or is used in small quantities). Contrastingly, a different procedure should be applied in case of using relatively high contents of hydraulic binders (e.g. ≥ 1.5 % by mass); with respect to the course of construction and the climate, it is to be discussed whether the test specimens should be left uncovered in an environment with relative humidity of 40-70 % or whether they should be put in plastic bags which is likely to give a better simulation of prompt overlay by the next structural layer. However, it is questionable whether this should occur immediately or for instance after 1-2 days break. In general, we can note that for mixtures with an amount of cement higher than bitumen residue content, the approach wherein the specimens remain unsealed is more conservative and will probably yield lower strength and deformation characteristic values. Due to that, the approach appears preferable. Nevertheless, it is important that reasonable simulations of accelerated curing will not be possible for this combination of binders since it is complicated, if not impossible, to accelerate the cement hydration process. Besides, it must be highlighted that on site weather conditions should also be taken into account when selecting a laboratory curing procedure.

5.10 Experimental study B9

<u>Main objective</u>: This study focused on evaluation of the effect of curing storing conditions (sealed and unsealed test specimens in the first 24 hours) on strength and stiffness of cold recycled mixtures

<u>Used binder(s)</u>: Bituminous emulsion (3.5 %) combined with a high content of cement (3 %)



5.10.1 Selected materials

For further determining of the effect of the moisture condition resulting by specimen sealing in a plastic bag in the first 24 hours after compaction, mixtures with a cationic slow-breaking bituminous emulsion (C60B7) and cement (CEM II B 32.5R) were prepared. The mixtures composition produced within this experimental study corresponds to mix A in Table 31, i.e. cold recycled mix with 3.5 % of bituminous emulsion and 3 % of cement.

Cold recycled mixtures were produced using 0-22 mm reclaimed asphalt.

5.10.2 Description of the study

Two sets of 12 laboratory specimens of mix A (3.5 % bituminous emulsion and 3 % cement) were produced. After compaction cylindrical test specimens with approx. 60 mm of height and 150 mm of diameter, one set of specimen was sealed by a plastic bag, whereas another set was left unsealed. For the curing, the specimens were stored for 7, 14 and 28 days at room conditions. Furthermore, studies were also conducted by storing the specimens for 11 days at room conditions (sealed and unsealed) and for further 3 days in water in accordance to the procedure described on the water susceptibility standard EN 12697-12 for the wet specimens (specimens submitted to a water vacuum for 30 minutes and immersed in water @ 40 \mathbb{C} for approximately 3 days).

The specimens from the first set (AS) were cured in sealed conditions for the whole time, while the specimens from the second set (AU) were cured sealed for the first 24 hours and then removed and cured for the rest of their curing time in laboratory conditions.

5.10.3 Main results

The results obtained are synthesized in Figure 47 and Figure 48, respectively, for cold recycled mixtures strength and stiffness values.









Figure 48: Comparison of stiffness values for sealed and unsealed test specimens

The sealed curing during the first 24 hours resulted in lower ITS and stiffness modulus values in all curing patterns applied. The difference between average values was from 7 % to 30 %, but in most cases around 10 %. The specimens which were cured at room conditions for 11 days and in water for 3 days (according to EN 12697-12 procedure for wet specimens) showed the most significant differences depending on the sealed or unsealed curing conditions.

5.10.4 Main conclusions

The former research which was performed in the framework of curing conditions impact showed that in case of mixes with cement higher moisture brings higher ITS and stiffness values, because the moisture is supportive for the hydration process. On the other hand even then was clear that the moisture value must be regulated. Reason for this necessity is that in general moisture interferes with bonding of the asphalt binder with the aggregate. In this case and in other experiments carried out in CTU and in University of Kassel the detrimental effect of moisture on the bond was bigger than the positive influence on the hydration process.



6 Effect of binder type on cold recycled material performance related properties

6.1 Use of different bituminous binders for foamed bitumen production and the influence on cold recycled mixture properties

6.1.1 Introduction

The foamed bitumen technology is experiencing a global renaissance and rather extensive development of innovative approaches with respect to both its application and research. However, the variability of available bituminous binders for utilisation within the technology is quite large; therefore, a general methodology of quality parameter determination for foamed bitumen and the requirements for mixes bound by this type of binder must be specified.

Generally, any bituminous binder which, when hot, comes into contact with water, starts foaming and increases its volume several-fold. However, this is not desirable in most cases. The essence of foamed bitumen preparations thus lies in driving air (under the pressure of approx. 10 bar) and water (2-5 % by mass under the temperature of 15-25 ℃ and pressure of 4-5 bar) in hot bituminous binder (commonly pen grade bitumen 50/70 - 160/220 under 160-190 °C) (Jenkins et al., 1999). In contact with the hot bituminous binder, water transforms into steam which is gradually captured and closed in tiny foamed bitumen bubbles and, having transformed into steam, the foam expands and its volume multiplies. In this state, the foamed bitumen is dosed and added to the aggregate. The entire process of foaming takes place in an expansion chamber to which the hot bituminous binder is carried and the water vapour and pressurised air are injected. Upon contact thereof, foamed bitumen is formed immediately and injected under high pressure to the wet aggregate mix, reclaimed asphalt material (RAP) or a mixture of these two components. Once the maximum level of foaming has been reached, the foamed bitumen bubbles gradually collapse and attempt to closely adhere primarily to the fine aggregate particles which creates bituminous binder droplets that link to one another to form a thin bitumen film coating the larger aggregate grains. In comparison to bituminous binder or emulsion, foamed bitumen has a much larger area and surface tension, which facilitates sufficient coating of tiny aggregate grains in particular with a smaller quantity of bitumen. In this respect, the fine particle content of the designed mix is important in particular since the mix is based on the principle of coating such particles with foam and the mortar formed subsequently binds the larger aggregate grains. Naturally, a proportion of the binder is caught on the surface of larger aggregate grains, too.

The quality of foamed bitumen is generally formulated as the proportion of the maximum volume of foamed bitumen achieved and the volume of the original bituminous binder on foaming, i.e. the expansion ratio (ERm). With premium quality foamed bitumen, the expansion ratio should amount to at least 8-15. Another qualitative parameter of foamed bitumen is defined as the foam half life time (HLT) which is the time (in seconds) in which the maximum volume of foamed bitumen decreases by 50 %. The longer the half life time, the better quality the foamed bitumen is; a time exceeding 15 seconds is judged as superior quality (as a standard, the $t_{\frac{1}{2}}$ value should be in the range of 8-15 seconds). The half time is



an expression of the foamed bitumen stability and is indirectly proportionate to the expansion rate (Jenkins *et al.*, 1999; Iwański, 2013; Mofreh, 2007; Sunarjono, 2008).

The expansion rate and the half life time are dependent parameters affected by the water content (FWC) added to the bituminous binder, and by the binder type and temperature (Brennen et al., 1983), as well as the settings of the foaming device, i.e. water, air pressure, and nozzle dimensions (Castedo-Franco & Wood, 1983). Ruckel et al. (1982) mentioned that the size of the cylindrical measuring vessel also affects the foamed bitumen parameters. Softer bitumen and higher temperatures generally produce better-quality foam. Based on his findings, Abel (1978) adds that acceptable foaming values are only achieved under temperatures exceeding 149°C. It should be mentioned, that the present effort to define global parameters of acceptable foamed bitumen is not easy. It should be figured out, that the foamability of bitumen is influenced by many factors and entry conditions (bitumen temperature, bitumen properties, foaming water content, characteristics of the foaming unit etc.). Usually determined empirical foam characteristics like expansion ratio and the half time are dependent parameters, nevertheless they show opposite trend. According to the table 1 presented in this study, in different countries minimum recommended or required values are specified for both characteristics. But it is guestionable how these foam characteristics do correlate with the resulting behaviour of the mix where foamed bitumen is applied (coating guality, strength properties, water susceptibility etc.), especially if the mix production process is considered as well (type of the mixing plant, mixing process). It is therefore still unclear if presently applied foamed bitumen design is optimal or not. It should be mentioned, that the present effort to define global parameters of acceptable bitumen foaming is not easy. It should be figured out that the foamability of bitumen is influenced by many factors and entry conditions (bitumen temperature, bitumen properties, foaming water content, characteristics of the foaming unit etc.). Usually determined empirical foam characteristics like expansion ratio and the half time are dependent parameters, nevertheless they show opposite trend. According to the Table 33 presented in this study, in different countries minimum recommended or required values are specified for both characteristics. But it is questionable how these foam characteristics do correlate with the resulting behaviour of the mix where foamed bitumen is applied (coating quality, strength properties, water susceptibility etc.), especially if the mix production process is considered as well (type of the mixing plant, mixing process). It is therefore still unclear if presently applied foamed bitumen design is optimal or not. Nevertheless, according to Ruenkrairergsa et al. (2004), softer bitumen and higher temperatures do not always guarantee improved foam quality, or in other words, foams with lower viscosity and relatively low surface tension are more likely to be susceptible to premature collapse prior to reaching the maximum volume than higher-viscosity foams (He & Wong, 2007). According to Jenkins (2000) is foamed bitumen decomposition depending on time and lower foam temperature, due to the bubbles being in contact with the surrounding air or aggregate, is one of the factors impacting foamed bitumen collapse. The intensity and efficiency of the foaming effect can be regulated by modifying the basic physical conditions like temperature, moisture and pressure (Iwański, 2013). Therefore, from the point of view of a sufficiently homogeneous mix, the size and arrangement of the mixer (e.g. the injection zone dimensions) are important as well as the expansion ratio.



The typical properties of foamed bitumen, as already noted above, are affected by the foaming water content (FWC, % by mass of the total asphalt quantity), or the maximum expansion ratio ERm and the half time of foam collapse, HLT (Jenkins et al., 1999). As can be generalised, ERm increases with increasing FWC while the HLT values tend to drop. Foamed bitumen with higher ERm and longer HLT currently represents the preferred combination of these properties which, from the perspective of the ultimate characteristics, should deliver a superior quality product. Unfortunately, the ERm and HLT values very often follow opposite trends and, therefore, it is difficult to stipulate the optimum of the FWC water content. According to Sunarjono (2009) and as stated earlier, good-quality foam tends to have an ERm within the 10 to 15 range. There are various empirical approaches to foam design as is indicated in Table 33, which stipulate the limit ERm and HLT values. The methods are appropriate primarily for field applications but, as a tool to assess and decide on the top quality selection for the foamed bitumen based on the FWC optimum (between the minimum ERm and HLT values), they are not exact; in other words, with respect to those parameters, the relation is not one of direct proportion. Another suitable characteristic for foamed bitumen specification is the parameter reflecting the ability of foamed bitumen to be mixed with mineral materials. The parameter has been defined as the foam index FI (s) (Jenkins et al., 1999). The expansion ratio is a gauge of foam viscosity while the value at which the foam collapses is defined by the half life time, i.e. an indication of the time available for mixing. The FI is determined as the area below the curve that represents the expansion ratio in time while the area might correlate with the surface energy of the foam. Nevertheless, an FI-based approach cannot be applied to all types of bituminous binders as, with increasing ERm, not all binders demonstrate continuously falling HLT trends (He & Wong, 2007; Lesueur et al., 2004), which means that the FI values grow with increasing FWC. According to Jenkins (2000) the foam index should be >125 seconds.

Data source	Limit for expansion ratio	Limit for half time (s)	
CZ (TP208)	10	10	
TRL Report 386 (Milton & Earland, 1999)	10		
Wirtgen (Wirtgen Cold Recycling Technology 2012)	0	6	
Asphalt Academy (TG2) 2009	O	O	
Asphalt Academy (TG2) 2002	7	7	
CSIR (Muthen 1998)	10	12	
Ruckel <i>et al.</i> (1983)	10 - 15	20	
Austroads (Austroads technical report AP-T178/11)	15	30 - 45	
Chiu and Huang (2002)	8	8	
Sunarjono (2009)	10	6	
Maccarrone, (1994) – for activated binders	15	60	

Table 33: Minimum quality parameters for foamed bitumen – different specifications



6.1.2 Experimental study - scope and found impacts

Within the framework of the CoRePaSol project, foamed bitumen was prepared using the Wirtgen WLB 10S laboratory-scale mobile foamed bitumen plant in which water was injected into the bitumen under the pressure of 5.5 bar and air under the pressure of 5.0 bar. The foamed bitumen injection time in a calibrated cylindrical metal vessel was 5 s. The foamed bitumen characteristics (ERm and HLT) were achieved through water injection within the interval of (0.5-6.0 % by mass of bituminous binder) under the temperature of 170°C, aiming to find the optimum between the two parameters researched.

Figure 49 shows the effect of water on the foamed bitumen quality characteristics for the bituminous binder and foaming additives researched. The individual types of bituminous binders assessed originate from different producers - Nynas, Total and Shell - as the objective was assessing the effect of the origin of the bituminous binder (and, therefore, of the input raw material) on the resulting foamed bitumen characteristics. In one case, the binder was supplied by emulsion producer Vialit Asphalt, where the original manufacturer could not be determined with absolute certainty. In total, distilled bituminous binders of penetration grades 50/70, 70/100 and 160/220 were obtained. In several cases, the bituminous binders were based on Russian crude oil (Russian Blend). In some bitumen variants there was a blend of several sorts of crude oil with a significant representation of North Sea raw material (Mittelplatte crude oil). The EM mark with some of the binders assessed means "fit for emulsification"; the crucial factor being, in this case, the salt content and neutralising number of these bituminous binders, which are usually different for each sort of crude oil. Bitumen 160/220 was compared to the binder allegedly based on Venezuelan crude oil which, from the perspective of bitumen exploitation rate and quality of the resulting distilled bitumen, is among the best. Only the characteristics of softening point and penetration under 25°C were examined for all of the bituminous binders assessed.

However, not all bituminous binders have the properties required for utilisation in foamed bitumen, i.e. the minimum ERm and HLT (or the minimum FI) are not always met. The reasons of poor foaming ability are not always clear; they are likely to be affected primarily by the distillation and other processing of crude oil. Due to that, alternative solutions are available where the bituminous binder may be improved for the purposes of foamed bitumen production by the application of a foaming agent.

As has been identified in the past, the addition of foaming agents can significantly improve the foaming properties of bitumen – particularly increase the foaming volume and slow down the foam collapse which also extends the interval for preparation of mixes with the binder and improves the level of coating (Jenkins *et al.*, 1999).





Figure 49: Foamed bitumen characteristics dependent on FWC



Within this study two additives have been tested as part of the research framework. Additive Iterfoam (Iterchimica) is used as a foaming thixotropic agent. The additive is of a dark, oily colour and from the chemical perspective it is a combination of different amine compounds. In the case of additive Evotherm MA3 (MeadWestVaco), the additive is an oily liquid of amber colour, based on derivatives of fatty acid amines. The additive is intended for applications in the field of low temperature asphalt mixes. Its application as a potential foaming agent constituted an assumption, not verified as yet, with respect to improving the surface activity (which is improved by this additive). The producer declares a 30-45 $^{\circ}$ C decrease in the production and laying temperature for the asphalt mix. The additive has volume weight of 970 kg/m³ and flash point CoC over 204 $^{\circ}$ (Iterlene, Evother m).



Figure 50: Foam index FI for bitumen 70/100 (Total)

Foamed bitumen optimisation was carried out under the methodology given in Wirtgen Cold Recycling Manual (2012). For the sake of comparison, the FI was determined, too. Nevertheless, as indicated in Figure 50, the values continued to grow with increasing FWC for all bituminous binders with no additives researched; therefore, the optimum FWC value could not be stipulated based on the global maximum. Contrastingly, FWC can be determined for bitumen 70/100 with additives and it is more fitting than following the methodology specified by Wirtgen Cold Recycling Manual (2012). Table 34 gives the quality parameters of the binders assessed and the optimum parameters of the foamed bitumen produced. It states the relationship between the qualitative parameters prior to foaming (penetration, softening point) and the foamed bitumen properties. Bissada (1987) proved that softer bituminous binders or binders of lower viscosity during foaming generate superior guality foams. However, based on the results shown above, the situation is guite the opposite or, within the framework of the research conducted, harder binders produce betterquality and more stable foams. In this context, it should be mentioned and emphasised that this can be affected by other factors (lower salt content for better emulsification and adhesion of the binders researched, generally different quality of bituminous binders in comparison to research conducted more than 25 years ago), or other causes which affect foamed bitumen formation and stability. The optimal water content in the foam for binders with the additive marked Iterfoam is determined according to the FI concept. The binder with additive produces a better quality of foam primarily from the perspective of stability (the foam collapse is higher by an order of magnitude). Contrastingly, Evotherm MA3 is absolutely unsuitable for



foamed bitumen production and, therefore, its application was excluded from further research while the objective remains identifying other additives which will have an effect similar to that of the first additive mentioned.

	Penetration (mm ⁻¹)	Softening point (⁰C)	Foam bitumen				
Bitumen			Optimum water content (% of bit.)	Expansion ratio	Half-life time (s)	Foam Index (s)	Foam suitability
50/70 (Nynas Nyfoam 60)	58	49.5	2.1	19	9.0	220.7	Very good
50/70 (Shell)	55	50.9	3.0	24	9.0	301.6	Very good
70/100 (Total)	83	46.8	3.8	21	7.0	239.3	Very good
70/100 EM (Total)	79	47.4	2.4	22	8.0	254.1	Very good
70/100 EM (Vialit)	86	46.2	1.1	12	8.0	112.2	Poor
70/100 (Total) + 0.4 % Iterfoam	-	-	2.7**	21	95	1277.2	Very good
70/100 (Total) + 0.6 % Iterfoam	-	-	2.6**	23	132	2498.5	Very good
70/100 (Total) + 0.4 % Evotherm MA3	-	-	3.8*	10	3.8	78.1	Very poor
70/100 (Total) + 0.6 % Evotherm MA3	-	-	3.8*	8	4.7	59.1	Unsuitable
160/220 EM (Total)	156	41.6	1.8	17	8.0	193.7	Good
160/220 (Nynas)	165	40.8	3.0	-	-	-	-
* Cannot be determined – estimate based on binder without an additive **Set based on the Foam Index parameter							

Table 34: Qualitative parame	ters of the bituminous	binders and fo	amed bitumen

6.1.3 Mix design

There is currently a wide range of design methods for foamed bitumen mixes. Each one differs by the grading of the filler (aggregate) used, methodology to determine the percentage of the foamed bitumen, filler type and content, water content and the issues related to the test specimen dimensions, mixing, compaction and curing. The design methods for foamed bitumen mixes also strongly depend on the type and quality of recycled aggregate used since different countries have different pavement structural design from the point of view of mix compositions (Chandra, 2013).

From the perspective of composition of the mineral skeleton in the recycled mix, sorted RAP material of 0/22 mm grading from the Středokluky mixing plant was chosen for the laboratory study and extraction was performed to determine the soluble binder content. Grading analysis has been done as well as depicted in Figure 48.




Figure 51: RAP Grading Analysis RAP

The binder content was observed as 5.6 % by mass. The optimum water content of the recycled mix was determined as the optimum according to the modified Proctor test. The water content is the most important factor from the perspectives of the course of mixing (quality of aggregate coating by binder, binder dispersion), compaction and resulting mix properties perspective (Sunarjono, 2009).

Generally, the optimum binder quantity is recommended in Cold Recycling Manual (Wirtgen, 2012) within the interval of 2-5 % by mass of the mix.

Table 35 stipulates the indirect tensile strength and stiffness modulus values for mix BSM-FB (cold recycled mix stabilised by bituminous binder using foamed bitumen) under the temperature of 15 °C while using bitumen 70/100 (To tal). However, the information did not allow determining the optimum quantity of the foamed bitumen. It should also be borne in mind that for such mixes, water susceptibility is a rather non-negligible parameter which, however, depends on the quantity of binder and quality of coating, and thus also on the foamed bitumen quality. Therefore, it was decided to dose the optimum foamed bitumen quantity in BSM-FB mixes to the amount of 4.5 % by mass of the mix.

Characteristic	Mix variants							
Foamed bitumen content (%)	2.0	2.5	3.5	4.5				
Bulk density (g/cm ³)	2.203	2.194	2.211	2.209				
Indirect tensile strength (MPa)	0.45	0.41	0.43	0.41				
Stiffness modulus @15℃ (MPa)	2333	1596	1631	1701				

Table 35:	Variants of recycled	asphalt mix ((BSM-FB) an	d characteristics	after 7days	air curing
			(·····,	

The mix itself was mixed by means of laboratory-scale twin-shaft compulsory mixer, Wirtgen WLM 30 for approximately 60 s. The resulting recycled mix was subsequently put in cylindrical moulds and compacted by static pressure of 5.0 MPa in compliance with technical conditions TP208. The cold recycled mixes prepared were compacted in cylindrical moulds of 150±1 mm diameter and 200-300 mm height. Specimens were tested after 7, 14 days curing on air under the temperature of 20±2°C and a fter 14 days on air and in water. The water-related curing conditions changed in the course of curing, in other words, on the first day after mixing and compaction, the specimens were left in 100 % relative humidity



(enclosed in the test mould or plastic bag) and, subsequently, for the rest of the curing period, in relative humidity of 40-60 %. One day after production, the basic volumetric parameters were determined for the test specimens. Once the curing period finished, indirect tensile strength (ITS) and stiffness modulus S_m were determined by a non-destructive test of indirect tensile stress at 15°C. Last but not least, water susceptibility was determined for the individual cold recycling mix variants.

6.1.4 Results and Conclusions

The summary results of the researched parameters for the recycled mixes are given in Figure 52.



Figure 52: Strength and deformation parameters of assessed cold recycled mixes BSM-FB



Cold recycled mixes with softer bituminous binders demonstrate lower values of indirect tensile strength from the perspective of resulting indirect tensile strength and, simultaneously, within the framework of test specimen curing in air. This contradicts the results presented in Lee (1981), which state that there are no perceptible differences between the measured properties of mixes prepared by the foamed bitumen technology with varying bitumen penetrations. However, the trend is not too pronounced and may be affected, with respect to the void content achieved, by a lack of homogeneity in the recycled material. Contrastingly, mixes containing softer bitumen are less sensitive to the effects of water. This indication might support the theory of better aggregate coating by the binder. The stiffness modulus values copy the trend mentioned within the part on indirect tensile strength. On the other hand, harder bituminous binders demonstrate higher indirect tensile strengths but lower water susceptibility.

The effect of bituminous binder penetration on the properties of bitumen foam could explain the entire mechanism of behaviour of BSM-FB mixes. According to Merrill *et al.* (2004), the choice of penetration of the bitumen applied is a compromise between the foam quality and the stiffness of the resulting mix. The ERm of the tested binders ranges from 12 to 24, and the half life time HLT falls within the interval of 7 to 9 seconds. It can be concluded that the effect of ERm on mix properties is more dominant than that of HLT. Therefore, the approximate minimum ERm value of \geq 15 can be recommended.

The foaming additive researched did not demonstrate any effect, as it did not influence the cold recycling mix properties as examined; therefore, only its application can be considered from the perspective of a possible longer mixing time, albeit with no real changes. This indicates that the HLT value is not a principal change from the perspective of foam design. Another factor could be the differences in the material inputs in the individual countries which, according to the assessment obtained, can be rather significant. The relation between the bitumen properties and the resulting foam characteristics, depending on the strength and deformation parameters of the cold recycled mixes, is not quite clear.

6.2 Use of alternative hydraulic binders: fly-ash

6.2.1 Introduction

In the research done during last few years in the field of road construction increased focus on environmental aspects and the protection of the environment is more and more visible. This is closely related to the necessity to limit consumption of natural non-renewable resources, to decrease amount of waste material disposed on land-fills and at the same time to identify suitable techniques which would help to reduce the energy demand during construction of transport networks. In case of waste there is a European wide target for construction and demolition waste reaching a level of their recyclability and reuse about 70 % until 2020. Similar criteria can be found or it would be wise to promote them also for other waste materials (Piau, 2006). Simultaneously some other initiatives and programs originated within the EU during last few years (especially as part of the activities done by the European centre for advanced studies), which focused on defining and describing suitable end-of-waste criteria. Such criteria will have in economic terms an important impact on easier use of such



materials in further production chains and for various applications. One of the areas gaining actually increased attention is represented by so called mineral waste materials. In this group also fly-ashes and solid reactive products based on calcium from desulphurized flue gasses can be included in.

Despite of R&D activities in the area of fly-ash utilization as an alternative binder or geopolymer especially for concrete, the possibilities to exploit bigger amounts of this material are limited. Various results can be found also in the research quite successfully realized recently also in the Czech Republic. The smaller applicable amounts of fly- ashes are determined by the mix composition and expected function of the fly-ash in mixes like concrete, usually having a role of a binder. Following the situation of fly-ash producers these trends cannot significantly solve the persisting problem of land-filling large amounts of this energetic by-product. So far gained results of ongoing research presented and discussed in this paper therefore aim on the possibility use fly-ash as an alternative binder in cold recycled mixes as well as on the possibility to substitute or supply part of the recycled material by fine-graded (pulverized) particles. Although it is realistically possible to use effectively only 5-15 % by mass of such material, this level represents an indispensable portion how to utilize especially more problematic fly-ashes from soft coal combustion and power production. In this area the Czech Republic shares front brackets in a worldwide comparison, especially in terms of per capita production.

Assessed application of the tested coal combustion by-products (CCB) was in cold recycled mixes as an alternative to more common variations with bituminous binders and cement. In this case, the fly-ash from fluidized combustion is used as a substitute for the hydraulic binder. The fly-ash tested was subjected to mechanical activation in high-speed disintegrators while the parameters of the mix under scrutiny reflected the impact on the strength and deformation parameters of the mix; last but not least the water susceptibility indicator was also monitored. From the environmental perspective of practical applicability of the mixes a chemical analysis and some simple leaching tests have been done.

A limiting factor for the use of some CCB types is their relatively low resistance in repetitive contact with water and frost (Chen *et al.*, 2009; Sear, 2001), volumetric changes and in some cases the risk of partly unsatisfactory health and environmental parameters (Bin-Safique *et al.*, 2006; Aydilak & Cetin, 2006]. Particularly for fly-ashes from fluidized combustion, ettringite (high-calcium sulfo-aluminate mineral) might be formed in case of long-term contact with water (Řezník, 2011; Chindaprasit *et al.*, 2013). Extensive analyses of CCB chemical characterization have been done, e.g. during the planning and preparation of embankment structures 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 (Vaníček, 2008).

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 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.

6.2.2 Used materials

Analyses of applied fly-ash

In the first step examination of used fly-ashes from fluidized combustion of the thermal power plants CEZ Tisova, CEZ Hodonin and the Plzen generation (heating) plant has been done. These plants are all equipped with the fluidized 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 brown (soft) coal very typical for Czech Republic and pulverized limestone which, from the perspective of potential for further application in the construction industry, is rather significant. The fly-ash 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 m.s⁻¹ and power consumption at the level of approx. 20 W per kg of pulverized fly-ash.

X-ray diffraction and chemical analysis was performed for the obtained material in cooperation with the Faculty of Natural Science at Charles University Prague. The chemical analysis is indicated in Table 36.

Fly-ash from fluid.	SiO ₂	AI_2O_3	CaO _{Total}	MgO	TiO ₂	Fe_2O_3	SO ₃	Na ₂ O	K ₂ O	P_2O_5	MnO	Cl	F [.]	Annealing loss
comb.							[%]							
Tisova	39,50	24,70	15,45	0,70	5,47	7,63	3,06	<1,0	0,49	0,35	0,058	-	-	2,05
Hodonin	43,16	16,27	16,83	1,86	1,28	3,62	5,08	0,7	3,58	1,53	-	0,71	0,34	4,85
Plzen							NDF							

Table 36: Chemical composition of activated fly-ash from fluidized combustion (NDF – not defined)

Fly-ashes from fluidized combustion from the Tisova power plant have a standard composition in comparison to other ashes. The only unusual feature is the higher content of non-decomposed limestone and anhydrite. The fly-ashes from the Hodonin power plant and the Plzen heating plant contain unusually small quantities of free lime; such fly-ash loses its self-binding ability and an addition of a certain quantity of lime or cement would be highly recommended in a certain stage of compacted mix production. The contents of the amorphous phase are rather high in such type of fly-ashes. The results of XRD analysis for mechanically activated fly-ashes are indicated in the following Table 37 and Figure 53.



Ref. Code	Compound Name	Score	Total Lines	Scale Factor	Semi-Quant [%]
	Fly-ash from fluidize	ed combus	tion – power pl	lant Tisova	
01-086-2334	Calcite	66	9	0.801	26
01-085-0796	Quartz	48	7	0.492	3
01-074-2421	Anhydrite	53	16	0.974	57
01-079-0007	Hematite	37	7	0.130	4
01-078-0315	Portlandite, syn	53	5	0.239	7
01-078-0649	Lime	42	2	0.152	3
	Fly-ash from fluidize	d combusti	ion – power pla	ant Hodonin	
01-089-8935	Quartz - alpha	72	7	1.002	46
01-072-0503	Anhydrite	69	16	0.294	23
01-079-1741	Hematite, syn	47	7	0.104	4
01-082-1691	Lime	47	2	0.084	3
00-005-0586	Calcite, syn	34	9	0.052	4
00-009-0478	Anorthoclase, disordered	30	28	0.042	9
00-046-1311	Muscovite, ammonian	36	20	0.204	11
	Fly-ash from fluidized	l combustic	on – generatior	n 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	36	17	0.427	18
01-083-0578	Calcite	43	9	0.071	2
01-089-6423	Albite	26	83	0.076	9
00-026-0911	Illite	36	17	0.427	18

Table 37: Evaluation of phase composition of mechanical chemically activated fly-ashes from fluidized combustion based on XRD data record

Mechanical activation process

Mechanical activation can be defined as a mechanical process under specific conditions or interference with the structure of a substance that increases its chemical reactivity. Such intervention in the substance structure can consist of grinding/pulverization. According to the traditional 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 (Faltus, 2009; Sekulić *et al.*, 1999; Blanco *et al.*, 2006; Baláž, 2008).

Grinding might be a possible solution for rough fluid combustion separation process residuals. The grinding process causes large plerospheres (porous particles) to disintegrate and reduce 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 hydration water (Hela, 2013).



Figure 53: X-ray diffraction (XRD) analysis data record for mechanically activated fly-ash from fluidized combustion a) Tisova, b) Hodonin, c) Plzen

Cold recycled mixes

The main application for assessed fly-ashes was a compacted mix using the cold recycling approach. This technology involves the milling of the original structural pavement layers (mainly asphalt layers or combination with granular base layers, but applicable also to e.g. cement stabilized material), mixing thereof with bituminous or hydraulic binders followed by levelling and compacting. Sorted reclaimed asphalt material (asphalt mix obtained by milling the asphalt layers or crushing blocks knocked from old asphalt pavements or asphalt mix from non-identical or excess production - RAP) of grading 0-11 mm from Porr asphalt mixing



plant in Běchovice, was used to prepare the cold recycled bitumen based mix. For the RAP bitumen content of 7.3 % by mass was detected by bitumen recovery analysis. Cationic bituminous emulsion C60B7 was applied as the bituminous binder for cold recycled mixes. This is a standard emulsion used for this type of cold recycling applications in the Czech Republic. Similarly, the commonly used Portland slag cement CEM II/B – S 32.5 was used in the reference mix.

A set of various cold recycled mixes with a variable representation of fly-ashes from fluidized combustion modified by mechanical activation were designed for the laboratory testing; the fly-ashes from fluidized combustion play the role of hydraulic binder substitute or reactive filling admixture in this case. At the same time, a set of laboratory tests was defined to verify the impact of the material on mix properties. The compositions of the assessed mixes are indicated in Table 38. The optimum water content of the cold recycled mix for the composition as indicated was determined according to the standard EN 13286-2.

Component/Mix	REF	MCAT 1a	MCAT 1b	MCAH 2a [%]	MCAH 2b	MCAP 1	MCAP 2
Bituminous emulsion (C60B7)	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Cement CEM II/ B-S 32.5	3.0						
Mechanically activated fly-ash; fluidized							
comb. – power plant Tisova		3.0	10.0				
Mechanically activated fly-ash; fluidized							
comb. – power plant Hodonin				3.0	10.0		
Mechanically activated fly-ash; fluidized							
comb. – generation plant Plzen						3.0	10.0

6.2.3 Cold recycled mix assessment methodology

Test specimen preparation methodology

Optimum water content of the mixes was determined according to EN 13286-2. The valid Czech technical conditions for cold recycled mixes TP 208 (Czech Ministry of Transportation, 2009) stipulate test specimen preparation by means of cylindrical moulds of 150.0 ± 1.0 mm diameter and 200-300 mm height. For the purposes of experimental assessment, cylindrical test specimens of 101.60 ± 0.1 mm diameter and 63.5 ± 2.5 mm heights were chosen as more appropriate. During the processing, the mix was compressed in the moulds by means of static pressure of 5.0 MPa. The test specimens were left in the mould at the temperature of (20 ± 2) °C for (24 ± 6) hours; subsequently, the specimens are stored at 90-100 % relative humidity and a temperature of (20 ± 2) °C for another two days. Then, the test specimens are stored at 40-70 % relative humidity and a temperature of (20 ± 2) °C. The mixes examined were left to cure under the aforementioned conditions for 7 and 14 days. From the point of view of water susceptibility detection, the set of samples was soaked in water for another 7 days after seven days' curing under 40-70 % humidity and a temperature of (20 ± 2) °C.



Indirect tensile strength, stiffness modulus and water susceptibility

Determination of indirect tensile strength is done on test specimens, which have been prepared as already described. Testing is proceeded according to TP 208 (Czech Ministry of Transportation, 2009), whereas the test specimens are air conditioned for at least 4 hours to the temperature of (15 ± 1) °C. Specimens are then te sted at constant speed of the laboratory press jaws (50±1) mm/min until the total specimen failure.

Stiffness modulus of cold recycled mixes has been done according to EN 12697-26, applying the method of non-destructive cyclic indirect tensile stress on the Nottingham Asphalt Tester apparatus. Stiffness is an important strain characteristic, which is used together with Poisson's ratio in multilayer pavement structure design. The principle of stiffness modulus determination is to apply direct compressive stress, which is transferred in the plane of vertical cut of test specimen to deduce indirect tensile stress perpendicular to the loading direction. The stress applied is causing horizontal deformation of the specimen. The stiffness modulus is thereafter defined as a ratio between stress and strain at a given temperature, for which a particular value of Poisson's ratio can be defined.

Water susceptibility according to TP 208 (Czech Ministry of Transportation, 2009) normally reflects coefficient of indirect tensile strength decrease. Generally this is defined as a ratio of indirect tensile strength of specimens cured 7 days vs. indirect tensile strength of test specimens after seven days curing at 40-70 % relative humidity and temperature of (20 ± 2) °C and additional seven days water immersion. In this paper the water susceptibility is evaluated only as a strength characteristic of combined curing.

Chemical Analysis

Chemical analysis have been carried out in cooperation with the Geological Institute of the Academy of Sciences of the Czech Republic (AV CR) for fundamental analysis of elements represented in the used materials and partly as a basis for describing leaching effects. Particularly selected samples of waste granular material, by-products, and reclaimed materials the use of which is being considered or expected in pavement structures, or where such construction applications already exist, has been selected for the analyses done. The set of tested materials involves waste filler from aggregate production, fly-ashes and reclaimed asphalt material.

In connection to the list of technical standards governing the leaching methods for various types of mineral materials different analytical procedures are used (Valentin, 2009). None of the methods governed by any standard concerned has been used; a modified procedure was preferred and applied based on analytical spectroscopy method. For this test procedure samples were analysed with IRIS Intrepid II XPS spectrometer (ICP-EOS = spectroscopic analytical technique optical emission spectroscopy with inductively coupled plasma) manufactured by Thermo Electron Corporation, using axial plasma view and cyclone type nebulizer. The standard operational conditions were used (plasma power 1150 W, nebulizer pressure 25.0 psi, auxiliary gas flux 1.0 ml/min, sample uptake 2.40 ml/min). For the analytical purposes wavelengths recommended by manufacturer for each element were used. The calibration curves were constructed using four points (blank and multi-element



standards in 1% supra-pure nitric acid) covering full range of the concentrations measured. Concentration of macroelements and microelements were calibrated and measured in separate experiments. Each sample was analysed three times. Quality control was ensured inserting QC sample into analytical run after each ten unknown sample.

For analytical purposes and as a basis for future leaching tests, the total elements contents in the solid samples were measured, after total decomposition of the solid samples in nitric acid/hydrofluoric acid/perchloric acid mixture. In this way the solid samples went into acidic solutions, in which elements concentrations were measured by ICP EOS. The concentrations of basic elements (macroelements) were estimated as well as the trace elements (microelements) by ICP EOS technique. Among others, the macroelements aluminium, calcium, ferrum, kalium, magnesium, manganese, natrium, phosphor, sulphur, silicium and the microelements arsen, boron, barium, beryllium, cadmium, cobalt, chromium, cuprum, lithium, molybdenum, nickel, strontium, titanium, zinc were analysed.

For the future alternative leaching tests distilled water (pH value ~6.5) and aqueous solution of acetic acid (pH value ~ 4.5) will be used as leaching agents. Acetic acid was chosen to approximate the effect of leaching by acidic rain water. Similar testing procedure and environment can be found, e.g. in (Townsend, 1999). Analytical experiments were performed on samples of the materials pulverised and homogenised, fraction of grain size < 0.1 mm was used. Even if such testing sample preparation is not usual in the standardised leaching test methods for granular materials, the pulverisation has been decided for the total inorganic analysis. Sample weight 1-5 g and 100 ml of the leaching solution was used. Weighted samples were covered with the leaching solution and agitated on an overhead shaker for 2 h at room temperature. Leachates were then filtered over 0.45 μ m filter and analysed. No leaching solution exchange was performed in the course of the test. Thus, the test can be characterised as a short-time procedure without dynamic character, i.e. without leaching solution exchange.

Diffusive Test

Within the framework of possible utilisation of cold recycled mix using coal combustion byproducts in pavement base courses, it is necessary to take into consideration the environmental aspects of such applications too; the necessity of determining the leaching of potentially hazardous substances in the surrounding environment arises. In a laboratory environment, the leaching can be simulated by a diffusion test. The essence of a diffusion test according to EA NEN 7375:2004 is monitoring the long-term effect of water on the product from the chemical (contents of analytes in the leach) and mechanical perspective (content of the solid part separated). The diffusion test is carried out statically to avoid influencing the natural diffusion and, at the same time, to avoid disturbing the sample surface. The nature and properties of the basic material with the entire sample (monolithic cylindrical specimen) placed in the leaching liquid (demineralised water of neutral pH) and additions of the leaching liquid at certain time intervals for the period of 64 days, the quantity of the leach per surface unit is determined. The qualitative evaluation of the leach observed the limits stipulated in the regulation concerning construction materials of the Dutch Ministry of Housing, Spatial Planning and the Environment issued in 1999, as amended. The leaches



were compared to the limits for pavement base courses (Building Materials Decree BMD, 64 days). Then, the results were compared to the Landfill Regulation Amendment for England and Wales no. 1640 of 2005 for waste deposited at landfills – leach after 64 days).

6.2.4 Results and Discussion

Strength and strain characteristics of recycled mixes

As is obvious from the results obtained and indicated in Figure 54 and Figure 55, substitution of cement by activated fly-ashes from fluidized combustion improves mix strength; the strength is reached already in the first 7 days of curing and does not increase significantly afterwards. The mixes assessed with 3.0 % fly-ash meet the strength parameters set by technical specifications TP 208 (Czech Ministry of Transportation, 2009), namely min. 0.3 MPa and max. 0.7 MPa, mixes with 10 % fail to meet the upper limit of the strength criterion, or the fast increase of strength demonstrates a potential risk of huge hydration heat generation and, potentially, formation of shrinkage micro-cracks during the curing. From the point of view of deformation parameters, the increase of the stiffness modulus is insignificant in relation to the reference mix. The indirect tensile strength and stiffness modulus values of water-soaked specimens show a positive trend with activated fly-ash application, namely no significant drop in water susceptibility of the mixes was observed as is the case of the reference mix. From the point of view of the parameters observed, it has to be mentioned that for the sake of improved resistance of the mixes the fly-ashes from fluidized combustion should be dosed for cold recycled mixes within the range of 2.0-5.0 % of mass of the mix.





Figure 54: Strength characteristics of cold recycled mixes



CEDR Call 2012: Recycling: Road construction in a post-fossil fuel society



Figure 55: Strain characteristics of cold recycled mixes

Results of Chemical Analysis

The results of performed tests are summarized in the tables presented below. The concentrations of particular elements are normalized by the unit mg/g per sample. Due to this normalization easy and well understandable comparison of particular macro- and microelements is possible. It is possible to transform the normalized amounts into relative expression showing that besides the analyzed elements the material usually contains also some other.

For the macro-elements aluminium, calcium and partly silicon seems to have the highest concentration (Table 39). The elements are represented for all chemical compounds in crystalline or amorphous phases. Aluminium represented in both fly-ash samples is a puzzle because the source material burnt was soft coal and limestone used as catalyst. Only in case of fly-ash Hodonin sample the energy producer is trying to modify the burn process by including partially municipal waste. The content of calcium can be easily explained by the burning process as already stated previously. This explanation might be used for sulphur as well. Silicon is represented in the RAP materials. This can be explained by the source of mineral materials.

Element Sample	AI	As	Ca Total	Fe content	K (mg) ol	Mg ⁱ eleme	Mn ent per	Na 1 g of si	P ample	S	Si
sorted RAP material 0/11	55.01	0.10	56.06	31.02	17.61	12.6	0.66	21.84	0.35	2.82	72.47
Fly-ash Plzen (fluid comb.)	105.95	-	166.36	40.39	1.55	9.88	0.96	8.20	1.58	32.29	0.51
Fly-ash Hodonin (fluid comb.)	108.33	-	141.67	36.11	1.80	9.44	0.43	5.11	0.54	29.64	0.35
"<" stays for below the detection limit, generally < 0.020 ppm.											

Table 39: Total sample analysis - macro-elements



From the micro-elements analysis following conclusions can be made. Increased content of hazardous/toxic micro-elements (observed) was found in total sample analyses as part of analyzed material:

- Both fly-ashes cadmium
- Reclaimed asphalt material 0/11 barium

In this context, it should be emphasized that in the case of beryllium (Be), cadmium (Cd) the elements in question are highly poisonous and toxic and are distributed mainly in dust.

Significantly different content of titanium in fly-ash from generation and cogeneration plant in Plzen cannot be explained easily there might be an important amorphous compound rich on this element. The reason for that might probably be in the source material of soft coal. In terms of total analysis reclaimed asphalt material can be described as a material rich on arsenic. During the respective leaching test of RAP samples it was however found, that arsenic is released rather from brown coal dust sample (not presented here). In this connection it is necessary to emphasize, that in terms of total extracted content of particular elements an important role will play the type of bond between such element and the analyzed material. Beside the elements shown in Table 40, for both fly-ash samples also zirconium has been detected in amount of 0.08-0.34 mg/g.

Element Sample	В	Ва	Ве	Cd Total c	Co ontent	Cr (mg) of	Cu element	Li per 1 g	Mo of samp	Ni ble	Sr	Ti	Zn
sorted RAP 0/11	1.89	0.30	0.0004	0.006	<	0.03	0.02	0.03	0.002	0.05	0.11	1.73	0.06
Fly-ash Plzen (fluid.													
comb.)	-	0.41	0	0.01	0.07	0.11	0.19	0.24	0.008	0.05	0.54	18.4	0.10
Fly-ash Hodonin													
(fluid. comb.)	-	0.31	0	0.01	0.02	0.13	0.13	0.12	0.01	0.07	0.29	5.42	0.30

Table 40: Total sample analysis – micro-elements

Diffusive test results

As ensues from the results indicated in Table 41, higher sulphates and arsenic values were detected in mixes where fly-ash from fluidized combustion was applied. The highest sulphate quantity is seen in the mix with fly-ash from the Hodonin power plant (the higher value is probably caused by burning brown coal together with unsorted municipal waste). The highest arsenic content in the leach was shown by the mix with fly-ash from fluidized combustion from the Plzen heating plant. The leach water from all mixes examined demonstrated alkaline pH within the range of 8.0 - 11.0; the pH values increased within the 14 days of test duration; subsequently, slight decreases were recorded.



CEDR Call 2012: Recycling: Road construction in a post-fossil fuel society

Determine monitored	d leach of elements	REF	MCAP2	MCAH2b	BMD 64 days	Land filled waste (leach after 64 days)
Chloride	[mg/m ²]	2 809.80	2 552.90	2 516.10	-	10 000
Sulfate	[mg/m ²]	2 390	71 397	126 308	27 000	10 000
Arsenic	[mg/m ²]	0.45	8.712	2.48	41	1.3
Cadmium	[mg/m ²]	0.09	0.086	0.085	1.1	0.2
Chromium	[mg/m ²]	0.6	0.6	0.6	140	5
Copper	[mg/m ²]	4.3	4	3.6	51	45
Mercury	[mg/m ²]	0.03	0.03	0.02	0.4	0.1
Nickel	[mg/m ²]	0.9	0.9	0.8	50	6
Lead	[mg/m ²]	0.2	0.31	0.3	120	6
Zinc	[mg/m ²]	8.3	5.2	6.1	200	30

Table 41: Values from	leaching test	ts for cold rec	cycled mixes

Test specimens of mixes REF and MCAP 2 demonstrated matt sediment on the water surface at the end of each leaching period (mostly in the ultimate periods).

6.2.5 Conclusions

Experimental results of the assessment of cold recycled mixes with application of combined binder formed by bituminous emulsion and mechanically activated fly-ash showed a benefit that can be delivered by materials otherwise rather difficult to use (often classified as waste or by-products) if mechanical and chemical activation process is employed. The refinement achieved during the process and, primarily, the structural changes caused by high-speed pulverization allow both applications of the materials examined as a partial substituent of traditional binders, particularly in the case of fluidized fly-ashes. In the case of cold recycled mixes, they potentially eliminate even the much more fundamental problem of water susceptibility and, therefore, poor durability of such mixes. However, with respect to the reclaimed material fraction used, we can assume that with more coarse-grained reclaimed material, the effect should be greater. Based on the findings, further assessments are being carried out to evaluate the stability of the strengths or stiffness characteristics over time. From the environmental perspective, the application of the binder concerned in cold recycled mixes is associated with certain problems; further risk identification in relation to the contamination of the environment, primarily ground and surface waters, is necessary.

The experimental results of the cold recycled mixes bound by mechanically activated flyashes as presented demonstrate a contribution that materials can show if the mechanical activation process is employed. These materials are otherwise difficult to apply and often qualified as waste or residuals. The fineness achieved by the process and, primarily, the structural changes caused by high-speed pulverization result both in the possible utilization of the assessed materials as a partial substitute for traditional binders, particularly in the case of fly-ashes from fluidized combustion. In the case of cold recycled mixes, they potentially eliminate even the much more fundamental problem of water susceptibility and, therefore, poor durability of such mixes. However, with respect to the fractions of reclaimed materials used, we can assume that a greater effect should be visible in the case of reclaimed materials with particles >11 mm. Based on these findings, further assessments are in progress which should evaluate the permanence of the strength or stiffness characteristics in



time. From the environmental point of view, utilisation of fly-ash as a binder or active filler in cold recycled mixes can be partly a problem; further identification of risks associated with contamination of the surrounding environment, particularly underground and surface waters, is needed.



7 Sensitivity studies on the influence of inhomogeneous materials properties of reclaimed asphalt

7.1 Introduction

During mix design of cold recycled materials the composition of the cold recycled mixture (content of additional aggregates, water content, bitumen content and cement content) is optimised for a representative sample milled from the original pavement (FGSV 2005, Wirtgen, 2012). For plant-mixed cold recycled material, the road granulate can be homogenised prior to the mix application in order to maintain constant material properties in this constituent as also in the resulting mixture.

However, for cold-in-place recycling the actual composition of milled road material can vary because of fluctuations in pavement structure in cold recycling site. Usually, in full depth reclamation the entire bound surface is milled and recycled. When the layer thickness varies in the run of a road or in transversal direction, it may happen, that also a significant portion of the unbound road base will be milled and therefore, the proportion of unbound material in the mix granulate will increase.

The aim of this study is to evaluate on the composition of milled granulate materials influencing the mechanical properties of cold recycled layers. By these results it can be estimated, in what extend heterogeneities in road structures can be tolerated for in-situ cold recycling works. Experimental work and laboratory test.

7.2 Experimental work and laboratory tests

7.2.1 Mix design

Cold recycled mixtures used in this study was designed according to German Mix Design standard (FGSV, 2005) for a cold recycled mix with maximum content of reclaimed asphalt (RA). In order to meet the specification regarding a minimum content of fines (< 0.063 mm), 3.6 % of limestone filler was added to the granulated RA. Figure 56 indicates the resulting grading for the mix granulates. By conducting Proctor compaction tests according to DIN EN 18127 the optimum water content was estimated to 7.8 %. The cement (CEM I 42.5 N) content was fixed to 2.0 %. The mix design study conducted resulted in optimum residual (virgin) bitumen content of 4.0 %. To reach this bitumen content, 6.4 % of bitumen emulsion (C60B1-BEM) was added to the mixtures. For the foamed bitumen mixtures, the foamed bitumen was produced at 180 °C with a water content of 4.5 % by bitumen mass (50/70) and a pressure of 5.5 bar.



CEDR Call 2012: Recycling: Road construction in a post-fossil fuel society



Figure 56: Particle size distribution of reclaimed asphalt

7.2.2 Mix variations

For each bituminous binder (bituminous emulsion and foamed bitumen) applied, eight mix granulate variations were used for the preparation of the cold recycled mixtures in laboratory. The eight variations were prepared by varied contents of reclaimed asphalt (RA), reclaimed cement concrete (RCC) originating from a crushed concrete pavement and basalt aggregates (for simulating reclaimed unbound base layers RUM). The contents of each type of granulate material in the eight mix granulates are summarised in Table 42. These mixes simulate different pavement structures which may be used for cold recycling.

Mix variations		Reclaimed asphalt (RA)	Reclaimed cement concrete (RCC)	Reclaimed unbound material (RUM)		
Ι	100/0/0	100%	-	-		
Ш	0/100/0	-	50%	50 %		
Ш	0/0/100	-	-	100%		
IV	50/25/25	50%	25%	25%		
Va	50/0/50	50%	-	50%		
Vb	75/0/25	75%	-	25%		
VI	0/25/75	-	25%	75%		
VII	40/20/40	40%	20%	40%		

Table 42: Varied cold recycling mixtures - Composition of mix granulate

7.2.3 Sample preparation and curing procedure

The cold recycled mixtures were produced using a Wirtgen compulsory mixer WLM 30. For the foamed bitumen mixes, the bitumen was foamed in a laboratory foaming unit WLB 10 S.

Each freshly produced mixture was compacted to 9 cylindrical specimens (diameter 149.6 mm. height 80 mm) by applying repeated static forces of 45.9 kN. After compaction, the moulded specimens were stored in a climate control chamber at 20°C and 80 % relative humidity for 1 day. After demoulding, the specimens were cured for additional 2 days in humid conditions (20°C, 80 % relative humidity). The additional curing was varied according to the mechanical tests to be conducted.



7.2.4 Laboratory tests

Following tests were applied for evaluating the mechanical properties of cold recycled mixes:

- Indirect tensile strength (ITS) tests acc. EN 13286-42 ITS conducted at 5℃ for evaluating the indirect tensile strength after 7 days of curing (ITS₇) - 2 specimens were stored at room conditions for 4 days and after 28 days (ITS₂₈) of curing - 3 specimens at room conditions for 25 days.
- California Bearing Ratio CBR was evaluated according to EN 13286-47 2 specimens were cured at room conditions for 25 days.
- Water susceptibility was evaluated by ITS tests on immersed specimen (ITS_{28,wet}) 2 specimens were stored for 11 days at room conditions and afterwards 14 days immersed in water (20℃). The water susceptibility is evaluated by comparing the strength values obtained on the immersed specimen compared to the dry conditioned specimen (strength loss = (ITS₂₈ ITS_{28,wet})/ITS₂₈ [%]).
- After dry conditioning the bulk density ρ_{b,dim} was evaluated according to EN 12697-6. From these values and the calculated maximum densities, the void content of each tests specimen was calculated.

7.2.5 Void content

The results of void content are shown in Figure 57 (left). The mix variation I (100/0/0) for which the mix granulate is composed of 100 % reclaimed asphalt reaches the specification limit for maximum void content of 15 %. The other mix samples (except for sample Vb) for which the mix granulate composition was varied, show higher void contents and would not comply with the mix design specification. These results indicate the importance for specific mix design to be conducted for each pavement to be recycled.



Figure 57: Voids content of emulsion mixtures (left) and foamed bitumen mixtures (right)

An increase in RA content in the mix granulate will improve the compactibility of the cold recycled mix and therefore results in significant lower void contents.



7.2.6 Indirect tensile strength

Results for indirect tensile strength (ITS) are given in Figure 58 for the bituminous emulsion mixtures and in Figure 596 for the foamed bitumen mixtures. The indirect tensile strength values vary significantly regarding the mix granulate composition. For better comparing the test results, the mix variations are sorted regarding the RA content, beginning with mixture I (100 % RA), Vb (75 %), IV and Va (50 %) and VII (40 %). The mixtures II, III and VI without RA are marked grey.

Similarly to voids content, the indirect tensile strength depends on the RA content in the mix granulate. A decreasing RA content in the mix granulates results in decreasing indirect tensile strength values of the cold recycled mixtures.



Figure 58: ITS after 7 days and 28 days for the bituminous emulsion mixtures sorted according to the RA content



Figure 59: ITS after 7 days and 28 days for the foamed bitumen mixtures sorted according to the RA content

On the other hand, the composition of the non-bituminous granulate material doesn't affect the indirect tensile strength values as indicated by similar strengths obtained for the mix samples II, III and VI (0 % RA) as well as for the samples with 50 % RA content (IV and Va).



7.2.7 Water susceptibility

The comparison of ITS values obtained on dry conditioned specimen as well as on water immersed conditioned specimen is summarised in Figure 60.

All mixtures with bitumen emulsion show a decrease in ITS after dry/wet conditions. The mixtures without RA (II, II and VI) indicate only a slight strength loss of below 10 %, whereas for the mixtures containing both, RA and non-bituminous mix granulate, the strength loss is higher than 10 %, except for sample I for which the mix design was optimised.

For the foamed bitumen mixtures, the water immersed curing results in an increase of ITS for four mix samples (Va, Vb, VI and VII). The strength increase can be explained by further hydraulic activity of the cement in the cold recycled mixture in case that the original water content was not high enough for allowing a complete hydration of the active cement content.

All samples tested comply with the mix design specification of a maximum strength loss of \leq 30 % (FGSV, 2005).



Figure 60: Results of water susceptibility tests: ITS after 28 days of dry conditioning and 14 days of immersed conditioning. The relative difference is given in [%]. Left: bituminous emulsion mixtures, right: foamed bitumen mixtures

7.2.8 California bearing ratio (CBR)

The results of CBR tests are summarised in Figure 61. The highest CBR values are obtained for the mixtures without RA components in the mix granulate (II, III, and VI).







7.3 Discussion and Conclusions

Figure 62 shows the results of void content evaluation and ITS versus the RA content in the mix granulate. The RA content affects both properties significantly. A decrease of RA content will result in an increase of void content (left) and a decrease of ITS. The figure also contains the specification requirements according to German mix design standard (FGSV, 2005) for (a) the mix design values and (b) for the compliance tests on samples cored from the pavement. From these results it can be concluded that, for emulsion mixtures, non-homogeneous pavement structures may be tolerated if the RA content in the mix granulate is higher than 50 %, whereas for foamed bitumen mixtures the limit value is 70 %. In both cases the voids content requirement is the limiting property. Based on the performed study found differences won't lead to incompliance for water susceptibility as all designed and evaluated cold recycled mix variants fulfilled the mix design requirements. Furthermore, the bearing capacity of the assessed mix variants with reduced or absent RA content repeatedly showed higher values in terms of CBR or indirect tensile strength compared to the reference mix with 100 % RA material. It can be therefore stated that even in the case of using other than RA materials, the overall pavement structure won't be under-designed.



Figure 62: Void content (left) and ITS after 28 days of dry conditioning (right) versus the RA content in the mix granulate for bituminous emulsion and foamed bitumen mixtures. German specification requirements are plotted in dotted lines

However, also in full-depth reclamation homogeneous bearing capacity especially transversal to the lanes is a requirement in order to avoid longitudinal cracking in the asphalt surface layers. Therefore, if a pavement structure indicates inhomogeneous transversal structure (perhaps of former road widening), a recycling device which allows a transversal mix of the cold recycling material is recommended.

Furthermore, the results showed, that the composition of the non-RA granulate (reclaimed cementious material or unbound material) doesn't affect the pavement performance.

Curing in water immersion usually reduces the ITS but also may result in increasing strength due to remaining active hydraulic binders.

An increased content of reclaimed asphalt in the mix granulate will decrease the CBR value and therefore the bearing capacity of the cold-recycled layer. Therefore, the effect of granulate composition is contrary compared to the indirect tensile strength.



8 General conclusions and recommendations

8.1 Cold recycled asphalt materials

The term "cold recycled asphalt materials" defines a pavement material which is composed of high proportions of reclaimed asphalt pavement material, as well as added bituminous and/or hydraulic binders; this composite is then in-situ or in-plant mixed, paved and compacted at ambient "cold" temperatures. As bituminous binders either bitumen emulsion or foamed bitumen are applied. Hydraulic binders, such as cement, fly-ash etc., are generally in small proportions applied as "active filler" for influencing the early-life properties of the cold recycled materials and/or in higher contents as binding agents, resulting in more rigid mixes.

European countries have different approaches to cold-recycling practices, applying a vast range of binder contents or binder combinations. Grilli *et al.* (2012) classified specific cold-recycled materials regarding the bituminous and hydraulic binders' contents. Within CoRePaSol project, the defined materials are further supplemented by cold recycled materials applied in Germany, the Czech Republic and a few other countries for the recycling of tar-contaminated road materials, which need to be sealed in the new pavement and therefore demand increased content of bituminous binders. Table 43 summarises the considered types of cold recycled materials classified according to their content of (residual) bitumen and mineral binder.

Table 43: Types of cold recycled materials according to content of bituminous and hydraulic	
binders	

Cold recycling material: Definition	Content of (residual) added bitumen	Content of hydraulic binder
Unbound (U)	0 %	0 %
Cement stabilisation (CS)	0 %	1 to 6 %
Lean concrete (LC)	0 %	≥6%
Bitumen-stabilised material (BSM)	1 to 3 %	< 1.5 %
Bitumen-cement-stabilised material (BCSM)	1 to 3 %	1 to 3 %
Cold asphalt mix (CAM)	≥ 3 %	0 %
Sealing cold recycled material (SCRM)	3 to 6 %	1 to 6 %

Differences found within European countries can result from:

- Climatic conditions: e.g. moist regions requiring use of higher amounts of cement, cold northern countries making impossible the use of cement;
- Geographical and historical reasons: e.g. recycling of tar-based pavements with the use of higher bituminous binder content in order to ensure a complete coating of the RA, cold recycled mixes with increased cement content for base or binder layers with higher bearing capacity.

All these aspects imply different mix design approaches, namely in terms of compaction methods, curing procedures and performance evaluation tests, for which some recommendations will be presented in following sections.



Regarding the various climatic conditions within Europe, following recommendations regarding the applicability of various cold recycled materials could be made:

- Bitumen stabilised materials BSM (cement content < 1.5 %):
 - applicable with foamed bitumen for flexible pavements in cold climate;
 - applicable with bituminous emulsion in dry or moderate climate;
 - not applicable with bituminous emulsion in moist climate;
 - conditionally applicable with bituminous emulsion in cold climate.
- Bitumen-cement stabilised materials BCSM (cement content > 1 %):
 - applicable for moist climate either with foamed bitumen or bituminous emulsion;
 - adequate for high early-life strength or if increased bearing capacity is required.

8.2 Laboratory mixing

Standard laboratory mixers used for hot mix asphalt can be applied for cold recycled mixes. It is always necessary firstly to mix the granulate material (RAP or RAP with aggregates) together with hydraulic binder and mixing water. In the second step bituminous emulsion or foamed bitumen are added. The applied mixing energy should be kept at moderate level of added mixing energy to avoid e.g. fast breaking of the emulsion.

In laboratory is advantageous the use of twin-shaft mixers which additionally are fitted to correct dosage and spreading of foamed bitumen. This type of mixers simulates very well the process of mixing cold recycled material if produced by the in-situ technique. Compared to traditional laboratory mixers for HMA twin-shaft mixer is not equipped with heating and therefore no additional technical problems are created, e.g. with switching off the heating unit or system, etc.

Nevertheless, it is worth mentioning that the production of cold recycled mixtures produced with foamed bitumen demand a special device for the production of the foam immediately before its introduction in the mixer. Commercial devices are available on the market.

8.3 Laboratory compaction

Hydrostatic pressure plays an important role in the compaction of cold recycled materials, thus static compaction and gyratory compaction are the most suitable methods, which additionally simulate well the way the material is compacted in the pavement.

With respect to <u>static compaction</u>, two test methods were further addressed in this research and final report. Based on the findings following recommendations could be drawn:

- Method used in Czech Republic (technical specifications TP 208) and Germany (DIN 1048 standard):
 - The referred standards / specifications already apply to cold recycled mixtures; therefore there is no need of adapting their procedures, except possibly for specimen's dimensions and load pressure.



- A final pressure of 5.0 MPa (approx. 88.5 kN for specimens of 150 mm in diameter) has demonstrated to be suitable for representing field compactions (i.e. voids contents of the same order of magnitude).
- Method used in Portugal and Spain (based on NLT 161/ASTM D 1074 standards):
 - As the initial standards were developed for hot mixes, some adaptations to the standard procedures are needed, namely in terms of temperatures of materials and of devices (e.g. when compacting cold recycled mixtures there is no need to heat the moulds).
 - A final pressure of 7.5 MPa (approx. 135 kN for specimens of 150 mm in diameter), instead of the standard pressure of 21 MPa, has demonstrated to be suitable to obtain representative laboratory test specimens of on site compactions.

It is propose to add a new part of EN 12697, on static compaction and comprising two levels of load: the one used in Germany and the Czech Republic applying 5 MPa static load to test specimen; and the one used in Portugal and Spain but for a compressive load of 7.5 MPa.

In case of **<u>gyratory compaction</u>**, the same test procedure as described in the European standard EN 12697-31 (developed for hot bituminous mixtures) can be used, but some adaptations to ambient compaction conditions must be performed (e.g. there is no need to heat materials/devices). Usually the static compaction pressure is set at 600 kPa with an angular velocity of 30 gyrations per minute and the gyratory angle of 1.25°. Some studies pointed out that representative test specimens of field conditions are achieved for a total number of 80 revolutions.

In both types of compactions, the entire set of mould devices (cylindrical mould and top & base plates) should allow for water to drain during the compaction procedure. These could be achieved in several ways, namely by:

- Using perforated top and base plates (e.g. Figure 5);
- Using top and base plates with an external diameter slightly lower than the inner diameter of the mould;
- Using slotted or perforated cylindrical moulds (e.g. Figure 10 and Figure 11).

8.4 Curing

Cold recycling materials show a significant time-dependent strength development after paving and compaction. For materials with bituminous emulsion, the bitumen particles start to coalesce after breaking of the emulsion, which usually occurs during compaction. This curing process can last several months until the strength of the pavement material is fully developed and stabilized. This is one of the main reasons why e.g. permanent deformation determination by wheel tracking tests is not suitable for this type of asphalt mixtures. The free emulsion water has to drain or evaporate from the mix. For foamed bitumen mixtures, curing also is required for added water evaporation and drainage. The addition of



mineral/hydraulic binder (cement, lime, HRB or fly-ash) can accelerate the curing process (by providing a reduction in the water content, which is used for their hydration) and will increase the early-life bearing capacity. However this will increase the rigid bonds in the material and introduce brittleness into the cold recycled materials.

The curing procedures applied in laboratory according to the national mix design specifications show a wide variety of curing parameters (duration, temperature, moisture). However, this variety can be explained by specific differences in climate and site curing conditions as well as the preference for specific types of cold recycled materials. These aspects were assessed in previously described experimental studies, which allowed for the following recommendations:

- After compaction the specimen shall be stored in the mould (or sealed in a plastic bag) for one day at room condition for allowing a demoulding without deteriorating the specimen.
- Curing at elevated temperature (40°C or 50°C) of s pecimens and unsealed condition:
 - Suitable only for cold recycled materials with a hydraulic binder content not exceeding 1.5 %. The fast drying of the specimen won't allow hydraulic reactions of cement occurring in field under moist conditions.
 - Curing duration of 3 days is adequate and represents minimum 21 days of normal curing at ambient conditions.
 - Medium-term strength development by consolidation effects in reclaimed asphalt can also be simulated.

With respect to the temperature to be used for accelerated curing, the selection between the range of 40°C to 50°C should be made ta king into account the specific characteristics of the road to be rehabilitated (e.g. temperatures in the cold recycled layer) and the type of bituminous binder to be used (e.g. residual bitumen softening point etc.). So far used 50 $^{\circ}$, which are based on experience gained in Portugal and Spain, might not optimally simulate real conditions in regions which are not in southern Europe, like UK or Ireland. It should be born in mind that in central/northern European countries, softer bituminous binders are commonly used, and the average temperatures in the binder or base course of a cold recycled pavement, even in hot summer periods, barely reaches 50 $^{\circ}$ C. Thereby, if the accelerated curing is done in laboratory at this temperature, the softening point of new binder can be reached for the bitumen which is used in bituminous emulsion or foamed bitumen. Such binder is melted during accelerated curing and might lead to a better coating of mineral particles in the mix. This most probably does not reflect the real conditions in the pavement and can lead to overestimated test results. For this reason it is continuously assessed if 40°C as, e.g. used in the UK or Ireland, is not a more suitable option particularly for central/northern European countries.



- Curing of unsealed specimen at room conditions as defined by temperatures around 20℃ and relative humidity between 40 % and 70 %:
 - Procedure is feasible for unsealed specimens with an adequate content of cement (more than 1.5 %).
 - Moisture between 40 % and 70 % is suitable since variations within that range won't affect significantly specimen properties.
 - The assessed strength development identifies a curing duration of 14 days as recommended while 28 days is not required.
- Curing of sealed specimens at room conditions:
 - Emulsion based BSM (cement content ≤ 1.5 %) identified a slow strength increase. Therefore sealed curing is not feasible for these types of cold recycled materials. The explanation might be found in the water to be released during emulsion breaking and mix consolidation.

The laboratory-applied curing procedures try to simulate and accelerate the curing conditions in filed. In-situ the particular conditions are of course strongly affected by climate and paving conditions. Therefore, the curing experiments allow further conclusions on the applicability of specific cold recycled materials regarding the climate conditions:

- Emulsion based BSMs (cement content ≤ 1.5 %) need drying for strength development and therefore are not applicable in moist regions.
- Foamed bitumen BSMs show adequate strength development also at moist curing conditions.
- However, in dry, warm climate, the addition of cement as in BCSM may not be required or useful for reaching adequate stiffness and strength properties.

8.5 Test methods

In general, same test methods can be used for cold recycled mixtures as for HMA (EN 12697 standard series), providing that test specimens are previously submitted to curing in some extent. The following general recommendations can be drawn:

Voids characteristics

Voids content of cold recycled layers containing a bituminous binder (emulsion or foamed bitumen) in-situ typically vary between 8-16 %-vol. Therefore, laboratory-prepared test specimens must have voids content within this range.

With respect to the particular method to be used for measuring test specimens' bulk densities, guidance is provided in EN 12697-6 depending among others on the voids characteristics of the compacted bituminous materials. According to developed studies, Procedure D (bulk density by dimensions) is recommended for measuring the bulk density of cold recycled mix specimens as the most appropriate. The bulk density should be determined always after the given curing period.



• Moisture susceptibility

Moisture susceptibility (water sensitivity) tests performed according to the European standard EN 12697-12 on cured test specimens are suitable for distinguishing the performance (durability) of different mixtures produced with different bituminous emulsion contents and no cement. However, besides minimum ITSR values, also minimum ITS_w values should be required.

On the other hand, even a small amount of cement (1 %) affects the behaviour of the cold recycled mixture and its curing process. Moisture susceptibility tests (ITSR) and ITS tests didn't allow for a distinction between mixtures containing 1 % of cement and varying contents of bituminous emulsion.

Indirect tensile strength

Indirect tensile strength tests on cold recycled mixtures can be performed according the European standard EN 12697-23, as long as specimens have been previously (partially) cured. It is the most commonly used performance test for determining the quality of cold recycled mixture.

• Stiffness modulus (optional test)

Stiffness can be determined by means of the non-destructive method of indirect tensile stress test (IT-CY method) listed in the European standard EN 12697-26. The experience and recommendations with this test are more in detail discussed in reports D2.1 and D2.2 of CoRePaSol project. Importance in performing these tests is in collecting long-term necessary data.



9 Harmonized advanced mix design procedure proposal

Based on previously presented relevant findings, a six-step mix design principle could be derived and recommended as best practice for cold recycled asphalt mixtures. Within these steps the variety of test methods and parameters shall be reduced in order to allow in the future comparisons of gained experience.

The following recommendations can be drawn for drafting a harmonised mix design approach for cold recycled materials:

Step 1: Analysis of reclaimed road materials

Reclaimed road materials shall be always analysed for suitability as mix granulate, namely in terms of its aggregate grading (before and after bituminous binder extraction), bitumen content and natural water content.

Regarding the grading curve requirements of the mix granulate following threshold values shall be applied:

- content of fines (< 0.063 mm): 4 10 %;
- content of fine aggregates (< 2 mm): 15 % 40 %.

Step 2: Selection of suitable binder(s)

Depending, among others, on the type of the required characteristics for the cold recycled layer (e.g. more flexible/rigid/semi rigid) and on the specific conditions of the road to be recycled (e.g. climatic conditions, traffic loadings) suitable bituminous and mineral/hydraulic binders shall be selected.

In the case of foamed bitumen being selected as binder, then an optimisation of its properties has also to be made (foaming water, necessity of using foaming agents). In case of bituminous emulsion the actually amplified concept focuses on the possibility to integrate rejuvenating agents in the emulsion and improve later the activity of the bitumen in the reclaimed asphalt material.

Step 3: Evaluation of optimum compaction water content and reference bulk density

An evaluation of optimum compaction water content shall be made taking into consideration both the mix workability and the layer compaction. Afterwards, the reference bulk density should be determined.

The application of modified Proctor test is the most applied procedure internationally. However, shortcomings from an impact compaction procedure are reported (mainly when relatively high amounts of binder are applied). Therefore, an alternative compaction method might be more feasible for conducting tests for evaluating optimum compaction water content and reference density. Some additional experiments are necessary to define a suitable compaction procedure based on controlled compaction energy. Finally, it is recommended not to overestimate the role of water content since it is only one of several factors influencing good or bad mix design. In the practice, contractors might use water



content slightly above or below the optimum content because of the overall conditions on the site (wind, sunshine, relative humidity, *etc.*).

Step 4: Mix preparation and specimen compaction

After mixing the cold recycled material in laboratory test specimens need to be compacted. It has been found that it is not possible to define for Europe just one laboratory compaction method, simply because of national experience and available equipment of road laboratories across Europe. Adequate compaction procedures identified within CoRePaSol project are:

- Gyratory compaction according to EN 12697-31 (which needs to be adapted to cold recycled mixtures: e.g. perforated moulds or base plates, no need for heating of the moulds and materials to be compacted);
- Static compaction with double-plunger and a compaction stress of 5.0-7.5 MPa, depending on the type of equipment/method of applying the static load (e.g. loading rate) and best national technical practice. In this case a new compaction standard needs to be drafted and prescribed for Europe.

Step 5: Curing of specimens

For simulating site-development and consolidation of cold recycled mixture properties (e.g. strength, stiffness), suitable laboratory curing procedures are required and can be seen as one of the most critical parts of cold recycled mix design. From the laboratory comparisons, following curing procedures are recommended for specimens, which are demoulded 24 ± 1 h after compaction:

- for BSM (cement content ≤ 1.5 %): curing of unsealed specimen at 50°C (or 40 °C) for 3 days,
- for CBSM and SCRM (cement content > 1.5 %): curing of unsealed specimens at room conditions for 14 days, whereas it is up to each country if additionally strength properties after 28 days are required or not. For quality assessment strength or stiffness values after 14 days curing should be prescribed as decisive.

Step 6: Mechanical tests

Firstly bulk density of test specimens has to be determined for each cold recycled mix. It is recommended to determine the bulk density before the indirect tensile strength test is done. Because of typical air voids content usually in a broad range of 8-16 % in volume, the test procedure calculating bulk density from test specimen dimensions has to be used. Then in order to assess the mechanical properties, the indirect tense strength as well as moisture/water sensitivity shall be assessed.

Depending on the type of mixtures and the specific requirements of the given road and its pavement (e.g. traffic level – intensities, type of traffic and its typical loading, *etc.*), other performance evaluation tests can be specified (stiffness modulus, crack propagation, *etc.*).



10 Acknowledgement

The research presented in this deliverable was carried out as part of the CEDR Transnational Road research Programme Call 2012. The funding for the research was provided by the national road administrations of Denmark, Finland, Germany, Ireland, Netherlands, Norway list funding countries.

Furthermore, the authors would also like to express their gratitude to all those who contributed to this report, namely:

- Infraestruturas de Portugal, S.A. (former Estradas de Portugal, S.A.) for providing reclaimed asphalt pavement for the experimental studies carried out at LNEC;
- CEPSA Portuguesa Petróleos, S.A. for providing bitumen emulsion to LNEC tests.



11 References

Abel, F. (1978). Foamed Asphalt Base Stabilization. Proc. of 6th Annual Asphalt Paving Seminar, Colorado State University, Dec.7-8.

Asphalt Academy (2009): Technical guideline: bitumen stabilised materials, TG2, Asphalt Academy, CSIR Built Environment, Pretoria, South Africa.

AUSTROADS (2011): Technical Report - Review of Foamed Bitumen Stabilisation Mix Design Methods, Austroads Publication No. AP-T178/11, ISBN 978-1-921709-77-7.

Aydilek, A.H., Cetin, B. (2013)., Geoenvironmental Impacts of Using High Carbon Fly Ash in Structural Fill Applications, Maryland State Highway Administration, MD-13-SP909B4P, Final report.

Balaz, P. (2008). Mechanochemistry in Nanoscience and Minerals Engineering, Chapter 2, High Energy Milling, Springer, Hardcover, Netherland, ISBN: 9783540748540.

Batista, F. (2004). Novas técnicas de reabilitação de pavimentos - Misturas betuminosas densas a frio (Innovative pavement rehabilitation techniques - Dense asphalt cold mixtures). PhD dissertation prepared under the cooperation between LNEC and FEUP, Porto, Portugal.

Batista, F.A.; Antunes, M.L (2005). Asphalt Cold Mixtures for Pavement Rehabilitation: Curing and Mechanical Characteristics. Proceedings of the 7th International Conference on Bearing Capacity of Roads, Railways and Airfields, BCRA'05, Trondheim, Norway.

Batista, F.A.; Antunes, M.L.; Mollenhauer, K.; McNally, C. (2012). Building blocks for a best practice guide on cold in-place recycling. Proceedings of the "5th Euroasphalt & Eurobitume Congress", Istanbul, Turkey, USB Pen Disk, 10p.

Bin-Shafique, S., Benson, C. H., Edil, T. B., Hwang, K. (2006). Environmental Engineering Science, 23(1), pp. 53-67, ISSN: 1557-9018, pp 253.

Bissada, A.F. (1987). Structural Response of Foamed-Asphalt-Sand Mixtures in Hot Environments. In: Asphalt materials and mixtures. Washington, DC: Transportation Research Board. (Transportation Research Record, 1115), pp 134-149.

Blanco, F., Garcia, Ma P., Ayala, Ma J., Mayoral, G., Ángeles, F. G. M. (2006). The effect of mechanically and chemically activated fly ashes on mortar properties, Fuel, Vol. 85, Issues 14-15, pp. 2018-2026, ISSN 0016-2361.

Brennen, M., Tia, M., Altschaeffl, A.G., Wood, L.E., (1983). Laboratory Investigation of The Use of Foamed Asphalt for Recycled Bituminous Pavements. In: Asphalt materials, mixtures, construction, moisture effects and sulfur. Washington, DC: Transportation Research Board. (Transportation Research Record; 911), pp 80-87, 1983.

Castedo-Franco, L.H., Wood, L.E. (1983). Stabilisation with Foamed Asphalt of Aggregate Commonly Used in Low Volume Road. In: Low volume road: 3rd International Conference, 1983.

CFTR (translated from French to English in 2007). Technical Guide "Valorization of local materials". French Road Engineering Committee, Sétra (ed.), Paris, France, 41p. (<u>http://www.setra.equipement.gouv.fr</u>)

Chamot, S., Romero, P. (2009). Fracture Energy Evaluation of Cold In-Place Recycling Mixtures. In: Advanced Testing and Characterization of Bituminous Materials. Leiden: CRC Press/Balkema, p. 1123-1130.



Chandra, R., Veeraragavan, A., Murali Krishnan, J. (2013). Evaluation of Mix Design Methods for Reclaimed Asphalt Pavement Mixes with Foamed Bitumen, Procedia - Social and Behavioral Sciences, Volume 104, pp 2-11, ISSN 1877-0428

Chen, Z., Wang, Z., Wang, X., and Zhao, Y. (2009). Study on the Frost Resistance Test Method of Lime-Fly Ash Stabilized Material Base Course. ICCTP 2009: pp. 1-9., ISBN: 978-0-7844-1064-6.

Chindaprasirt, P., Thaiwitcharoen, S., Kaewpirom, S., Rattanasak, U. (2013). Controlling ettringite formation in FBC fly ash geopolymer concrete, Cement and Concrete Composites, Volume 41, pp. 24-28, ISSN: 0958-9465.

Chiu, C.T., Huang, M.Y. (2002)- A Study on Properties of Foamed Asphalt Treated Mixes. Chung Hua University, Department of Civil Engineering, No. 30 Tung Shiang, Hsin Chu, 300, Taiwan.

Claudel, D., Valery, L., Triquigneaux, J.P. (2012). Which grade of bitumen for cold asphalt concrete? Proceedings of the "5th Euroasphalt & Eurobitume Congress", Istanbul, Turkey, USB Pen Disk, 7p.

COLAS (2006). Tenue à l'eau des enrobés à chaud et à froid.

Collings, D.C., Jenkins, K.J. (2008). Characteristics of materials stabilised with foamed bitumen. Proceedings of the "4th Euroasphalt & Eurobitume Congress", Copenhagen, Denmark, Paper 402-113, 12p.

CSIR Transportek, 1998. Foamed Asphalt, Mix Design. www.foamasph.csir.co.za:81/chap4.htm

Eckmann, B. (2012). Report on "Warm Mix Asphalt and Low Temperature Techniques". Proceedings of the "5th Euroasphalt & Eurobitume Congress", Istanbul, Turkey, USB Pen Disk, 11p.

Eckmann, B., Delfosse, F; Chevalier, E; Pouteau, B (2008). Stiffness of Cold Recycled Materials. Proceedings of the "4th Euroasphalt & Eurobitume Congress", Copenhagen, Denmark, Paper 403-008, 10p.

Eckmann, B., Soliman, S. (2008). Performance assessment of cold recycling in place. Proceedings of the "16th IRF World Meeting - "Sharing the Road", Lisbon, Portugal.

Eckmann, B., Delfosse, F., Chevalier, E. (2012). Reducing emissions and consumption of virgin aggregates through cold in-place recycling. Proceedings of the "5th Euroasphalt & Eurobitume Congress", Istanbul, Turkey, USB Pen Disk, 11p.

EA NEN 7375:2004 (2004). Leaching characteristics of granular building and waste materials. The Determination of the Availability of Inorganic Components for Leaching: the Tank Test. Environment Agency, UK

EN 12697-26 (2012). Bituminous mixtures. Test methods for hot mix asphalt. Stiffness

Evotherm [™] 3G M1 Safety Data Sheet. North Charleston - South Carolina: MeadWestvaco Corporation, 2011.

Faltus, M. (2009). New types of hydraulic binders based on waste materials, Ecology and new building materials and products, ISBN 978-80-254-4447-4.

Fernández del Campo (1998). Tratado de estabilización y reciclado de capas de firmes con emulsión asfáltica.

Formanová, Z. (2011). Recyklace prováděná za studena na místě, poznatky z laboratorních měření. JUNIORSTAV conference. Brno.

www.fce.vutbr.cz/veda/JUNIORSTAV2011/pdf/2.3/Formanova_Zuzana_CL.pdf



Forde, M. (editor), (2009). ICE manual of construction materials. London: Thomas Telford Limited, ISBN 978-072 736-420.

Fordyce, D., Khweir, K. (2002). The use of a designed foamed bitumen stabilized RAP in an urban high street. Proceedings of the "9th International Conference on Asphalt Pavements", Copenhagen, Denmark.

Forschungsgesellschaft für Straßen- und Verkehrswesen (2005). Merkblatt für Kaltrecycling in situ im Straßenoberbau - M KRC.

Guisado, F., Santiago, J.L., Páez, A., Ayala, M. (2011). Influencia de la temperatura de mezcla en las propiedades mecánicas de los reciclados en frío con emulsión. Journal "Asfalto & Pavimentación" No. 2, pp.31-39.

Grilli, A.; Graziani, A.; Bocci, M. (2012). Compactability and Thermal Sensitivity of Cement-Bitumen Treated Materials. Road Materials and Pavement Design, Vol. 13(4), pp. 599-617.

He, G., Wong, W.: Laboratory Study on Permanent Deformation of Foamed Asphalt Mix Incorporating Reclaimed Asphalt Pavement Materials." Construction and Building Materials, Volume 21, Issue 8, pp. 1809-1819, Elsevier, 2007.

Hela, R., Orsáková, D. (2013). The Mechanical Activation of Fly Ash, Procedia Engineering, Vol. 65, pp. 87-93, ISSN 1877-7058.

Iwanski, M., Chomicz-Kowalska, A. (2013). Laboratory Study on Mechanical Parameters of Foamed Bitumen Mixtures in the Cold Recycling Technology, Procedia Engineering, Volume 57, p. 433-442, ISSN 1877-7058

Jenkins, K.J., de Ven, M.F.C., de Groot, J.L.A. (1999). Characterisation of Foamed Bitumen. 7th Conference on Asphalt Pavements for Southern Africa (CAPSA).

Jenkins K.J. (2000). Mix Design Considerations for Cold and Half-warm Bituminous Mixes with emphasis on Foamed Bitumen, PhD Dissertation, University of Stellenbosch, South Africa.

Khweir, K., Fordyce, D., McCabe, G. (2001). Aspects Influencing the Performance of Foamed Bitumen Stabilized Aggregate Mixtures. The Asphalt Year Book 2001, Institute of Asphalt Technology, pp. 27-34.

Lesueur, D., Clech, H., Brosseaud, A., Such, C., Cazacliu, B., Koenders, B., Cerino, P., Bonvallet, J. (2004). Foamability and Foam Stability. Road materials and pavement design, ISSN 1468-0629, vol.5, no. 3, pp. 277-302, Paris.

Maccarone, S., Holleran, G., Leonard, D.J., Hey, S. (1994). Pavement Recycling Using Foamed Bitumen. Proceeding 17th ARRB Conference, Part 3.

Martínez, A., Miró, R., Pérez, F. (2007). Spanish experience with the application of gyratory compactor and indirect tensile test in design and control of cold recycled asphalt pavement. Annual Meeting of the Transportation Research Board, Transportation Research Record, no. 2001, pp. 163 168.

McAsphalt. http://www.mcasphalt.com/en/files/pdf/products/2-Evotherm_20Contractor.pdf (accessed May 11, 2013).

Merrill, D., Nunn, M., Carswell, I. (2004). A Guide to the Use and Specification of Cold Recycled Materials for The Maintenance of Road Pavements. Prepared for County Surveyors' Society, Highways Agency, Hanson Envoronmental Fund (Viridis), Scottish Executive, Tarmac, UKQAA, WS Atkins, Colas, RBA. TRL Report TRL 611. First published 2004, ISSN 0968- 4107, TRL Limited.



Milton, L.J., Earland, M. G. (1999). Design Guide and Specification for Structural Maintenance of Highway Pavements by Cold In-Situ Recycling, TRL Report TRL 386, Transport Research Laboratory, Crowthorne.

Czech Ministry of Transportation (2009). Technical Specifications TP 208 – Cold recycling of structural pavements layers of flexible pavements, Prague (in Czech).

Mofreh, F., Saleh (2007). Effect of rheology on the bitumen foamability and mechanical properties of foam bitumen stabilised mixes, International Journal of Pavement Engineering, Vol. 8, Iss. 2.

Mollenhauer, K., Ipavec, A., Gaspar, L., Marsac, P., Mirski, K., Batista, F., Antunes, M.L., McNally, C.and Karlsson, R. (2011a). Synthesis of national and international documents on existing knowledge regarding the recycling of reclaimed road materials in asphalt. DIRECT MAT DIsmantling and RECycling Techniques for road MATerials - Sharing knowledge and practices, Deliverable D5. FP7/2007 2013 EC no. 218656.

Mollenhauer, K., Ipavec, A., Gaspar, L., Marsac, P., Mirski, K., Batista, F., Antunes, M.L., McNally, C.and Karlsson, R. (2011b). Best Practice guide for the dismantling of asphalt roads and use of recycled materials in asphalt layers. DIRECT MAT DIsmantling and RECycling Techniques for road MATerials - Sharing knowledge and practices, Deliverable D19. FP7/2007 2013 EC no. 218656.

Olard, F., Beduneau, E., Seignez, N., Dupriet, S., Bonneau, D. (2009). Laboratory performance based assessment of half warm mix asphalts with high recycling rate by means of the factorial experiment design approach. Proceedings Advanced Testing and Characterization of Bituminous Materials, Taylor & Francis Group, London, ISBN 978-0-415-55854-9, Volume I, pp. 651-660.

PG4 (2001). Pliego de Prescripciones Técnicas Generales para Obras de Conservación de Carreteras. Ministerio del Fomento, Madrid, Spain.

Piau, J.-M., et al. (2006): SAMARIS - Sustainable and Advanced MAterials for Road InfraStructure, Final Report, LCPC and FEHRL.

Reznik, B. (2011). The effect of aggressive conditions on mechanical properties of alkali-activated of fly ash, pp. 312. ISBN: 978-80-214-4232-0.

Ruckel, P.J., Acott, S.M., Bowering, R.H. (1982). Foamed- Asphalt Paving Mixtures: Preparation of Design Mixes and Treatment of Test Specimens. In: Asphalt materials, mixtures, construction, moisture effects and sulfur. Washington, DC: Transportation Research Board. (Transportation Research Record; 911), pp 88-95.

Ruenkrairergsa, T., Phromsorn, C., Silarom, P. and Ketnoot, W. (2004). Engineering Properties of Foamed Bitumen Mixtures in Thailand. Proceedings of the 8th Conference on Asphalt Pavement for Southern Africa (CAPSA '04), ISBN No. 1-920-01718-6, Sun City, South Africa.

Sear, L.K.A. (2001). Properties and Use of Coal Fly Ash: A Valuable Industrial By-product, Thomas Telford Ltd, London, United Kingdom, pp 261, ISBN: 0727730150.

Sekulic, Z., Popov, S., Duricic, M., Rosic, A. (1999). Mechanical activation of cement with addition of fly ash, Materials Letters, Vol. 39, Issue 2, pp. 115-121, ISSN 0167-577X.

Serfass, J.P.; Carbonneu, X.; Eckmann, B.; Triquigneaux, J.P. (2009). Design method for cold and warm emulsion mixtures based on links between laboratory and field. Advanced Testing and Characterization of Bituminous Materials, Loizos, Partl, Scarpas & Al Qadi (eds), © 2009, Taylor & Francis Group, London, ISBN 978-0-415-55854-9, Volume I, pp. 609-618.



Serfass, J.P., Poirier, J.E., Henrat, J.P., Carbonneau, X. (2004). Influence of curing on cold mix mechanical performance. Materials and Structures/Matériaux et Constructions, Vol.37, June 2004, pp. 365-368.

Sunarjono, S. (2009). Laboratory Mixture Design for Foamed Asphalt, Dinamika TEKNIK SIPIL, Akreditasi BAN DIKTI No: 110/DIKTI/Kep/.

Sunarjono, S. (2008). The Influence Foamed Bitumen Characteristics on Cold Mix Asphalt Properties. 32 PhD thesis, University of Nottingham.

Tebaldi, G., Dave, E., Marsac, P., Muraya, P., Hugener, M., Pasetto, M., Graziani, A., Grilli, A., Marradi, A., Wendling, L., Gaudefroy, V., Jenkins, K., Loizos, A., Bocci, M. (2012). Synthesis of Specimen Preparation and Curing Processes for Cold Recycled Asphalt Mixes. ISAP Conference.

Townsend, T.G. (1999). Leaching characteristics of asphalt road waste. Final report No. 98-2, State University System of Florida, Florida center for solid and hazardous waste management, Gainesville, Florida.

Tijeda, T. (1999). Investigación sobre el comportamiento en laboaratorio de las mezclas bituminosas recicladas con emulsión. Bases para un nuevo método de formulación. PhD thesis, Madrid.

Valentin, J. (2009). Problems of selected performance characteristics of cold recycling mixes. Ph.D. thesis, CTU in Prague.

Valentin, J., Rohovec, J. (2009). Alternative Leaching Test for Selected Secondary Materials Used in Road Construction. In ENVIROAD. Warszawa: Road and Bridge Research Institute, 2009, p. 1-11. ISBN 83-89252-02-3.

Vanicek, I., Vanicek, M. (2008). Earth Structures - In Transport, Water and Environmental Engineering, Springer, pp 370-376, ISBN: 978-1-4020-3963-8

Wirtgen GmbH (2012). Cold Recycling - Wirtgen Cold Recycling Technology. Manual, 1st edition, Germany.

