CRABforOERE

Report detailing mix design methodology improving and the test results on CMA formula studied

Deliverable D4

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1 Introduction

Cold recycling is widely applied worldwide as well as within Europe, and practical as well as theoretical experience is available. However, these experiences are mostly focussed on selected types of cold recycled materials and are based on local application conditions, and therefore the understanding of these materials, their actual composition as well as applied mix design vary considerably within Europe.

In section 2, this deliverable summarises the mixture design procedures applied in selected European countries. After a short summary of general data regarding applied national specifications, some limit values will be gathered for different phases of mix design methodology.

Then the next objective of this deliverable report as the main output of CRABforOERE-work package 4 (WP4) is to propose a set of relevant parameters for an experimental program based on incorporation of RA aggregate into CRM mixtures. Thus, effects of mix composition (bitumen and cement dosages), curing conditions and testing conditions on mechanical performances using indirect tensile strength (ITS), triaxal and Duriez tests have been studied for the same mix designs. Therefore, section 3 and 4 describe the applied methodology, whereas the results are summarised in section 5 and discussed in section 6.

2 National Mix design procedures

2.1 German mix design

The German specification documents M KRC (FGSV, 2005) and M VB-K (FGSV, 2007) prescribes a mix design procedure for cold recycled materials produced in-situ and in-plant respectively. In general, reclaimed asphalt (RA) and added natural aggregates are mixed with bitumen emulsion or foamed bitumen as well as water and cement.

The mix design procedure can be divided in six steps:

Step 1: Analysis of reclaimed road materials for suitability as mix granulate: aggregate grading, bitumen content, natural water content;

Step 2: Choice of binders (bitumen emulsion/foamed bitumen, mineral binder type) and optimisation of foamed bitumen, if required;

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

Step 4: Mix preparation and specimen compaction;

Step 5: Curing of specimens;

Step 6: Mechanical tests.

2.1.1 Analysis of reclaimed road materials

The new cold recycled mix usually contains of more than 90 % of the recovered material from existing roads. Therefore, the evaluation of the characteristics of the reclaimed material is a fundamental issue for the whole mix design process. Firstly, it is important to recover a suitable sample representing the actual properties of the reclaimed road material during the recycling project. For in-plant recycling, suitable samples from a stockpile may be feasible for conducting the mix-design, if a stockpile management for reclaimed asphalt materials guarantees homogeneous properties of the stockpile. However, more usual for cold recycling project is the application of single-source reclaimed material which is milled just before the cold recycling pro-

cess from the actual construction site. This is true for in-situ recycling but also for plant recycling where a mobile cold mixing plant is set at the construction site. In these cases of single-source reclaimed road material, the sample for conducting the mix design should be milled from the actual site in advance representing the real material conditions during recycling.

From the reclaimed road material sample, the bitumen content as well the grading is evaluated. The binder content is needed for checking the added residual bitumen content after construction.

Regarding the grading of the reclaimed road material, the specifications demand for a minimum fines content (< 0.063 mm) and specific range proportions of fine aggregates (≤ 2 mm) as summarised in Table 1. Usually, pure milled reclaimed asphalt shows a shortage of fine particles. In order to achieve the required grading, additional crushed aggregates (max. 15 %) can be added to the mix granulate material during the recycling process.

Table 1: Specifications for particle grading of mix granulate according to German mix design procedure

In-situ (M KRC, FGSV, 2005)	Emulsion cold mix		Foamed bitumen cold mix	
	Fines (< 0,063 mm)	Fine aggregates (> 0,063; < 2 mm)	Fines (< 0,063 mm)	Fine aggregates (> 0,063; < 2 mm)
	2 - 10 %	≥ 20 %	3 - 12 %	≥ 25 %
In-plant M VB-K (FGSV, 2007)	Emulsion/foamed bitumen cold mix			
	Fines (< 0,063 mm)	Fine aggregates (> 0,063; < 2 mm)	Coarse aggregates (> 45 mm)	
	4 - 9 %	20 - 30%	≤ 10 %	

2.1.2 Choice of binders (bitumen emulsion / foamed bitumen, mineral binder type) and optimisation of foamed bitumen

The following binders should bei applied in the cold recycling mixture:

- Bitumen emulsion: Slow setting bitumen emulsions produced with at least 60 % of paving grade bitumen.
- Foamed bitumen: Paving grade binders according to EN 12591 (but only 50/70 and 70/100) should be used to produce foamed bitumen. The bitumen foam properties are defined by expansion ratio (initial foam volume / pure bitumen volume) and half-time (duration until the foam degenerates to half of its initial volume). The foaming water content, foaming temperature and air pressure shall be optimised to reach an expansion ratio ≥ 10 as well as a half-time ≥ 10 s.
- Cement: Portland cement or Portland slag cement are required. 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, the use of Class 32.5 cement is usually recommended. However, as Class 32.5 CEM I is not marketed or even produced recently, it is usually replaced by Class 42.5 CEM I or alternative Portland-limestone cements/Portland slag cement or hydraulic road binders (HRB) according to EN 13282.

The range of specified binder contents are summarised in Table 2.

Table 2: Limit values for different types of binder in cold recycled mixtures according to German mix design procedure

Binder mix	Bitumen emulsion + Cement		Foamed bitumen + Cement	
Specification	Bitumen emulsion	Cement	Foamed bitumen	Cement
M KRC	2,0 - 6,0 %	3,0 - 6,0 %	2,5 - 5,0 %	1,0 - 3,0 %
M VB-K	3,0 - 5,0 %	1,5 - 2,5 %	2,5 - 5,0 %	1,0 - 2,0 %

2.1.3 Evaluation of optimum compaction water content and reference density

During construction, cold recycled materials more or less have similar properties as unbound road materials. Because of ambient paving and compaction temperature, the friction between the grains is not reduced by low-viscosity bitumen; thus, water is required for improving the compactibility. Therefore, the optimum water content is usually evaluated by the modified Proctor method according to EN 13286-2. However, other compaction procedures (e.g. gyratory or static compaction) are also applied for identifying the optimum water content.

The added water content is dependent on the natural moisture of the recycled asphalt material (RAP) as well as the water content of the bitumen emulsion (if used). The following equation can be used for determinating the added water content (w_{zu}):

$$w_{zu} = w_{erf} - w_{eig} - w_{em} - 0{,}5 * B \qquad (1)$$

With:

w_{erf} = required water content (according to modified Proctor test) [%]

w_{eig} = water content of the reclaimed asphalt [%]

w_{em} = water content of the bitumen emulsion [%]

B = residual bitumen of the emulsion [%]

If foamed bitumen is in used, w_{em} and B are neglected.

2.1.4 Mix preparation and specimen compaction

For mix preparation, it is recommended to use a laboratory mixer which is capable of thoroughly mixing the cold recycled mixtures in short time. The standard is the usage of a double-horizontal-shaft-mixer for 120 seconds up to 300 seconds. After mixing, the specimens for mechanical tests need to be compacted. The typical compaction method is the static compaction (Duriez) with a pressure of 2,8 MPa. The dimension of the specimens is 150 mm in diameter and 125 mm height.

2.1.5 Curing of specimen

Cold recycled materials show a significant time-dependent strength development after laying and compaction. For materials with bitumen 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. The free emulsion water has to drain or evaporate from the mix. The addition of mineral binder (cement, lime or HRB) can accelerate the curing process (by providing a reduction on the water content, which is used on 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. For foamed bitumen mixtures, curing also is required for added water evaporation

and drainage. In the case of mixtures conmtaining hydraulical binders, e. g. cment, the hydration process results in a time-dependent increase of strength in the cold recycled mix. The standard curing laboratory curing procedure for cold recycled materials is shown in Table 3.

Table 3: Laboratory curing procedure according to German mix design procedure

Stage of curing	Duration	Temperature	Humidity
1.	2 d	20° C	≥ 95 % (specimen in sealde plastic bag)
2.	26 d	20° C	40 % - 70 %

2.1.6 Mechanical tests

For mix design usually mechanical tests are applied on cured specimen. The main properties checked in specifications are:

- void content,
- ITS according to EN 12697-23 with added measurement of horizontal deformation, which allows to assess the stiffness modulus,
- water sensitivity tests by comparing the ITS of dry-cured and water-conditioned specimens (ITSR).

The test conditions applied are summarised in Table 4.

Table 4: Test parameters and specfication values for mechanical test methods according to German mix design procedure

Specification	Void content	ITS test temperature	ITS	Water conditioning	ITSR	Stiffness*
M KRC	8 – 15 %	5 °C	0,75 – 1,2 MPa	14 d at 20° C	≥ 70 %	3.000 – 7.000 MPa
M VB-K	5 - 15 %		0,7 – 1,0 MPa			

* for bituminous bound CRM-layers

2.2 Italian mix design

Cold recycled bituminous mixtures can be produced in-plant (stationary or mobile) or in-place (using a recycling machine also called Recycler). If necessary, Reclaimed asphalt (RA) is mixed with virgin aggregates to obtain a grading curve complying with the specifications. Bitumen emulsion, cement and water are added to obtain the required volumetric and mechanical properties.

The mix design procedure can be divided in 3 main phases:

1. Materials selection
2. Optimization of the water dosage
3. Optimization of the binders dosage (Bitumen emulsion and cement)

In the first phase, the key points are selecting the RA and adjusting the mixture gradation by adding virgin aggregate. This is usually necessary for having an adequate content of fines (filler and fine sand) and thus a good “mortar phase” in the mixture. Commercially available bitumen emulsion and cement are employed and there is no explicit criterium for their selection.

In the second phase, the procedure focuses on workability of fresh mixtures. Similar to unbounded mixtures, the addition of water will enhance compactability and optimize dry density. Additionally, specimen saturation needs to be avoided.

In the last phase, the optimal dosage bitumen emulsion and cement is found based on ITS and water resistance, after a standardised curing protocol. Further, the stiffness of the design mixtures is verified and controlled.

2.2.1 Materials selection

RA is the main component of the mixtures and, during mix design. It is considered as a “black rock”. Its grading distribution must be determined using the wet sieving method (EN 933-1).

- For in-plant production, RA shall be classified in accordance to EN 13108-8 and the grading distribution shall be determined on the stockpiled RA (“black curve”). Before stockpiling, site-won asphalt must be processed (e. g. crushed and screend) in order to remove asphalt lumps and foreign material.
- For in-place production, the grading distribution (“black curve”) and the bitumen content must be checked on the RA material sampled after one pass (or more passes) of the Recycler (without the addition of binders).

Virgin coarse and fine aggregate shall conform to EN 13043. Their properties shall conform to the required values specified in Table 5.

Table 5: Specifications for aggregate properties according to Italian mix design

Property	Norm	Symbol	Unit	Required value
COARSE AGGREGATE				
Los Angeles coefficient	EN 1097-2	LA	%	≤30
Crushed particles	EN 933-5	C	%	100
Maximum dimension	EN 933-1	D	mm	31,5
Fines content (passing to 0.063 mm)	EN 933-1	f	%	≤1
Freeze-thaw resistance	EN 1367-1	F	%	≤1
Flakiness index	EN 933-3	FI	%	≤30
Water absorption	EN 1097-6	WA_{24}	%	≤1,5
FINE AGGREGATE				
Sand Equivalent	EN 933-8	ES	%	≥60
Crushed particles			%	100
Fines content (passing to 0.063 mm)	EN 933-1	f	%	≤2

The target composition of the design aggregate blend (RA + virgin aggregate) shall be within the grading envelope specified in Table 6.

Bitumen emulsion shall conform to EN 13808, shall be cationic, slow setting, with high stability when mixed with cement. For base courses, modified emulsion is required (C60BP10), and the properties of the recovered binder (by evaporation) shall conform to the required values specified in Table 7.

Table 6: Grading envelope for mix granulate according to Italian mix design procedure

Sieve size	In-plant Passing (%)	In-place Passing (%)
80	100	100
63	100	95-100
40	85-100	85-100
22,5	70-100	70-95
10	50-75	50-75
4	60-42	60-42
2	20-35	20-35
0,5	10-18	10-18
0,063	4-8	4-8

Table 7: Specified bitumen emulsion properties according to Italian mix design procedure

Property	Norm	Unit	Required value
Penetration	EN 1426	0,1 mm	50-70
Softening point	EN 1427	°C	> 60
Fraas breaking point	EN 12593	°C	< -13
Elastic recovery at 25°C	EN 13398	%	≤2

Cement shall conform to EN 197-1. There are no a-priori limitations on type and strength class.

2.2.2 Optimization of the water dosage

The optimization of the water content shall be carried out on the design aggregate blend plus 2% of cement, by dry aggregate mass (no emulsion). Samples shall be prepared at different water dosages, as specified in Table 8.

Table 8: Assessed water contents for identification of optimum water content

Cement dosage	2%					
Water dosage	3,0	4,0	5,0	6,0	7,0	8,0
Number of specimens	3					

Specimens shall be compacted using the gyratory compactor according to the protocol specified in Table 9. To produce homogeneous specimens, aggregate particles retained on the 20 mm sieve may be removed before compaction.

Table 9: Gyratory compaction parameters in experiments for identification of optimum water content

Mould diameter	150 mm
Mass of specimen	2800 g
Number of gyrations	100
Angle of inclination	1,25°
Revolution speed	30 rpm
Vertical pressure	600 kPa

Each specimen must be weighted immediately before and immediately after compaction to check for material loss (normally water extruded during compaction).

The dry density of the specimens shall be calculated based on the mixture composition and the final volume of the specimen in the mould. The mass is to be assessed after oven drying.

The optimum water content is the value giving the highest dry density. However, the mass loss during compaction has to be less than 0,5 %.

2.2.3 Optimization of the binders dosage

To determine the optimum dosage by dry mix granulate mass of bitumen emulsion and cement, mixture samples shall be prepared at different binder dosages, as specified in Table 10.

Table 10: Binder dosage to assess in Italian mix design procedure

Cement dosage	1,5			2,0			2,5		
Emulsion dosage	3,0	3,5	4,0	3,0	3,5	4,0	3,0	3,5	4,0
Number of specimens	4								

The added water content of the samples will be determined as follows:

$$W_{add} = W_{tot} - W_{em}$$

Where W_{tot} (%) is the optimal value determined abobe, W_{add} (%) is the dosage of added water (pre-wetting water) and W_{em} is the water content of the bitumen emulsion.

Specimens shall be compacted using the gyratory compactor according to the protocol specified in Table 9. After compaction, specimens shall be oven-cured at 40°C for 72 h (3 days) and then conditioned at 25 °C for 4 hours. The ITS of the specimens shall be determined according to EN 12697-23.

Mixtures with ITS > 0,35 MPa will be considered for further testing of stiffness and loss of ITS due to water sensitivity:

- indirect tensile stiffness modulus (ITSM) shall be determined according to EN 12697-26, annex E on specimens prepared and cured with the same procedure described for ITS and conditioned at 20 °C for 4 hours.
- the loss of ITS will be determined measuring the ITS on specimens prepared and cured with the same procedure described above, and than conditioned in water for 1h (under vacuum).

The optimal mixture will be the one with the lowest stiffness and complying the following criteria:

- ITS > 0,35 MPa
- ITSM > 3000 MPa
- Loss of ITS < 30% (ITSR > 70 %)

The dry density of the optimal mixture shall be determined using the procedure described above. This value will be used as reference for quality assurance testing.

2.3 Swedish mix design

2.3.1 Tests of the source material

Representative samples of the reclaimed asphalt (RA) shall be taken from stockpiles according to TDOK 2014:0146. Each taken sample shall be analyzed with respect to:

- Binder content
- Softening point for the recycled material. In those cases when the softening point cannot be tested, the kinematic viscosity shall be analyzed at 60°C.
- Geometrical properties (particle size distribution) for the dry RA according to EN 933-1.
- The reclaimed material shall be dried at 50°C to constant weight and thereafter tempered to room temperature before being sieved.
- Particle size distribution of the extracted aggregates.
- Water content of the RA.

Determination of binder type, binder additive and added ballast shall be based on the results from the preliminary tests.

2.3.2 Recipe for CRM

The recipe shall include suitable parts of the following information:

- Employed mixing plant
- Type of CRM
- Employed material quarry for added natural aggregates
- Amount and type of added
    - Bitumen emulsion
    - RA including bitumen content of that reclaimed asphalt and softening point, alternatively kinematic viscosity at 60°C for reclaimed bitumen
    - Aggregates including relevant properties
    - Additives including mixing process
- Particle size distribution according to EN 933-1 for dry reclaimed asphalt including natural aggregates shall be presented graphically together with maximum and minimum values according to specification according to Table 11.
- Residual bitumen content (% by weight)
- Laboratory packing method and compaction temperature
- Bulk density (EN 12697-6, procedure D)
- Maximum density (EN 12697-5)

- Air void content (EN 12697-8)
- Indirect tensile strength ratio (ITSR) (TDOK 2014:0147)
- Stability at +25°C (EN 12697-34)
- ITS at +10°C (EN 12697-23)
- Stiffness modulus (EN 12697-26)

2.3.3 Specifications for CRM mix composition

The specified mix granulate grading is given in Table 11. The requirement is for the distribution at the feed to the asphalt plant. The distribution curve can cross one of the inner limits. Table 12 gives the requirements on bitumen contents, Table 13 the aggregate specifications.

Some specifications depend on the traffic loading of the road, where the CRM is applied. Therefore, the average daily traffic (AADT) and the average daily traffic of heavy vehicles (> 3,5 t) $AADT_h$ are considered.

Table 11: Particle size distribution including added ballast according to Swedish specification

Sieve size (mm)	Percent passing by weight, min-max	
	Outer limit	Inner limit
45	-	-
31,5	100	-
22,4	85 - 100	-
16	67 - 100	-
11,2	48 - 95	60 - 83
8	30 - 80	40 - 70
5,6	15 - 67	25 - 58
4	10 - 55	17 - 48
2	5 - 35	10 - 30
1	2 - 17	5 - 14
0,5	1 - 10	3 - 8
0,25	0,5 - 6	1 - 6
0,125	0 - 4	1 - 3
0,063	0 - 2	0,5 - 1,5

Table 12: Water ratio, added bitumen emulsion, residual bitumen according to Swedish specification

	Base layer	Surface layer
Water ratio for ingoing RA including added ballast (% by weight)	3,0 - 5,0	2,0 - 4,0
Added bitumen emulsion (% by weight) Based on bitumen emulsion with 60% bitumen; when using bitumen emulsions with lower or higher bitumen content, recalculation must be done to ensure corresponding residual bitumen content.	1,3 - 2,8	2,3 - 4,4
Total bitumen content for CRM (% by weight) (RA bitumen + residual bitumen from emulsion) When recycling oil gravel or softened oil gravel the requirement on binder content is lowered to 3,4 - 5,5% for base layers and to 4,0 - 6,5% for surface layers.	4,4 - 6,5	5,0 - 7,5

Table 13: Specifications for added aggregates according to Swedish specification

Properties	AADT (in both directions)	
	<500	501-1500
Flakiness index, FI	≤20	≤20
Crushed faces, C, category	$C_{50/30}$	$C_{50/30}$
Ball mill value, A_N	≤19	≤14
Los Angeles value, LA	≤30	≤30

2.3.4 Performance requirements on CRM

A merged sample of reclaimed asphalt shall be the base of the mix design process. Mixing, compaction of specimens and curing shall be executed according to TDOK 2014:0147.

Within mix design, comparative tests shall be executed at three different binder contents within the range for the present specification Table 12.

All tests shall be executed on two specimens except for ITSR where three test replicates are prescribed.

- Bulk density shall be determined according to EN 12697-6, procedure D.
- Maximum density shall be determined from the same samples according to EN 12697-5.
- Air void content shall be calculated according to EN 12697-8.

For base courses, see Table 14, the mix with the highest stability/stiffness shall be selected, provided that all other requirements are fulfilled.

For surface courses, see **Fehler! Verweisquelle konnte nicht gefunden werden.**, the mix with the highest ITSR shall be selected, provided that all other requirements are fulfilled.

Table 14. Performance requirements for CRM.

Properties	Method	Specification	
		base course	surface course
Air void content (% by volume)	EN 12697-8	6,0 - 14,0	4,0 – 12,0
Stability according Marshall at +25°C[1)]	EN 12697-34	>7	> 5
Stiffness modulus, MPa[1)]	EN 12697-26	>2000	-
Indirect tensile strength at +10°C Dry samples, 7 days	EN 12697-23	-	>300
Water sensitivity, ITSR in %	TDOK 2014:0147	>50	>60

[1)] Marshall stability or stiffness modulus shall be chosen.

2.4 United Kingdom mix design

In the United Kingdom, specifications for the production of CRM has been covered in the *Specification for Highway Works* (MCHW1) Series 900, TRL 386 and 611 report. These specifications have covered the production of bitumen bound recycled material using an *in-situ* stabilisation process. In recent years, *ex-situ* or plant mixed cold recycled material technology has evolved and there is increasing demand for a wider range of materials to be covered by a publicly available material specification. Two specifications have been developed: one for the *ex-situ* process and one for the *in-situ* process. The division of the specifications has been done for means of clarity only. The structure and performance requirements for the materials in each specification are identical. *Ex-situ and In-situ* CRM shall be designed to achieve the specified level of the appropriate end performance property.

2.4.1 General requirements for aggregate

The aggregate for CRM can come from the milled material from existing roads; alternatively, other approved aggregate types may be used from other sources. The source and stock of the aggregate for recycling is a key factor in the decision about material composition and the construction process. A fine-grained aggregate may be more suitable to hydraulic binders whilst certain types of aggregate may prove incompatible with certain bituminous binders. If it is possible to obtain an adequate volume of aggregate from the road to be maintained by milling and/or crushing of the existing material, then an *in-situ* stabilisation process could be favoured to an *ex situ* process.

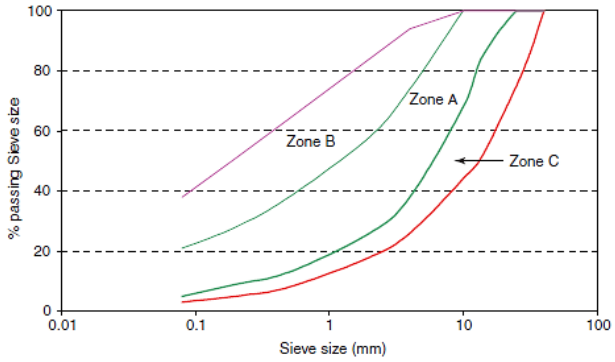
Natural, recycled unbound and manufactured (artificial) aggregates shall be clean, hard and durable and shall comply with BS EN 13043:2002 and be CE marked and have a declared performance which demonstrates that the aggregate meets the requirements of the specification. Where recycled coarse aggregate or recycled concrete aggregate is used in bituminous mixtures, it shall have been tested in accordance with Clause 710. The constituents of a sample of recycled aggregate shall be classified by hand-sorting the coarse aggregate particles in accordance with BS EN 933-11.

Grading

Many of the materials that could be cold recycled materials are described under the umbrella of European (CEN) standards. It is, therefore, prudent to ensure that the grading used for the specification to cover CRM is consistent with all the materials in the European standards that are likely to be required to be covered by this guide. In particular, the sieve sizes shall be consistent with European standards (which have replaced previous BS sieve sizes in the UK). Milton and Earland (1999) defined two grading zones for in situ stabilisation: Zone A and Zone B. Zone A is a well graded aggregate of nominal size 50 mm.

Zone B is a finer grading than Zone A; materials graded to within Zone B were only permitted to be used if mix design testing proved that this aggregate could be used to produce a consistent CRM. The development of the grading curves has attempted to maintain the curves defined by Milton and Earland whilst incorporating the CEN approach to grading. Three grading envelopes are shown in Figure 1: Zone A (suitable for all cold recycled materials), Zone B (finely graded aggregate only suitable in certain circumstances) and Zone C (a coarse grading that is only suitable for in situ stabilisation). The pulverised pavement material and any supplementary aggregate and/or filler shall normally be granular material with not less than 5% and not more than 20% passing the BS 75-micron sieve Zone A graded material]. Approval for use of pulverised granular material containing up to 35% passing the BS 75-micron sieve Zone B graded material shall require confirmation by the overseeing organisation, subject to the results of the mixture design procedures below-described.

The detail of the grading requirements for these zones is given in Figure 1.



	Percentage by mass passing		
Sieve (mm)	*Zone A*	*Zone B*	*Zone C*
40	100	100	100
31,5	100	100	86-100
20	100	100	75-100
14	85-100	85-100	52-100
10	68-100	68-100	44-100
4	38-74	38-94	26-74
2	26-58	26-84	18-58
0,5	13-38	13-64	8-38
0,250	9-28	9-51	5-28
0,063	5-21	5-38	3-21

Figure 1. Grading curves for cold recycled mixtures and particle size distribution of mixture for cold recycling adapted from UK mix design specifications

2.4.2 Primary binder agent

The primary binder shall be foamed bitumen or bitumen emulsion. The base bitumen shall comply with BS 3690: Part 1 and shall normally have a penetration grade 100. Subject to the results of the mixture design procedures and approval of the Overseeing Organisation, the base bitumen may have a penetration grade up to 200.

Other constituents

The constituents and required quality standards of hydraulic cement, filler and lime delivered to site shall be certified by the supplier, whose manufacturing and delivery processes shall be implemented using quality management systems in accordance with the ISO 9000 series of standards and certified by an accredited body. Hydraulic cement as a filler or adhesion agent shall be Portland cement, Portland blast furnace cement or Portland pfa cement, in accordance with sub-Clause 3 of Clause 1001 in the Specification for Highway Works. Pulverised Fuel Ash

(PFA), used as a filler, shall be in accordance with BS 3892: Part 1. Lime for lime stabilisation (or as a modifier for plastic fines) shall be either quicklime or hydrated lime, complying with sub-Clause 3 of Clause 615 in the Specification for Highway Work.

2.4.3 Moisture content

The moisture content of the mix granulate prior to stabilisation is equally important as grading, since it is of primary importance in controlling the workability of the mixture and therefore, governs the degree of compaction that may be achieved. It shall be measured in accordance with BS 812: Part 109 using the high temperature method. The optimum moisture content would normally be considered as the target moisture content, but in practice for recycled mixtures, the specified moisture content depends to a certain extent, on the binder used and whether a filler has been added in any great quantity. For the pulverised aggregate stabilised using foamed bitumen binder, the 'fluid' component of the binder will enhance the workability of the mixture and aid the thick lift compaction of the material. Therefore, a target moisture content in the range ±2% of optimum moisture content, determined in accordance with BS 1924: Part 2: 1990, is considered appropriate to achieve adequate thick/lift, full-depth compaction. Also, where more than 4 % by mass of adhesion agent and/or filler are added to the pulverised aggregate, the optimum moisture content should be determined using the modified aggregate. Where the moisture content prior to stabilisation fails to meet the target range, the moisture content should be adjusted either by aeration to reduce the moisture content or by controlled addition of water, if the moisture content is lower than required.

Test	Standard
Moisture content	BS 1377: Part 2
Bulk Density	BS 598: Part 104
Air Voids Content	BS 598: Part 104 & sub-Clause 45 of Clause 970AR
Grading and Binder Content	BS 598: Part 102
Indirect Tensile Stiffness Modulus [ITSM]	BS DD 213: 1996

Figure 2. Testing standards for the design and characterisation of recycled bitumen bound material

2.4.4 Mixture design and characterisation

The approach proposed in UK for CRM allows the contractor more freedom in which to conduct the mix design; it is, however, a requirement of the specification that the mix design stage is documented and used as part of the compliance procedure in the permanent works. Therefore, it does not form part of the contract requirements but the end product requirements (which state that the work carried out in the mix design stage must be followed) will form part of the contract. The contractor has some freedom to choose the method and detail of the mix design stage as agreed with the client. Nevertheless, prior to commencing the pulverisation and stabilisation works, the Contractor shall demonstrate to the satisfaction of the Overseeing Organisation, using the results of mix design procedures described here introduced. The testing standards used for the mixture design of the recycled bitumen bound mixture shall be those listed in Figure 2.

The laboratory prepared aggregate shall be thoroughly mixed with measured proportions of the bitumen binder and adhesion agent(s), to produce at least three trial mixtures with different added bitumen contents. The type and grade of the bitumen and adhesion agent(s) used in the trial mixtures shall be the same as those used in the finished works. The different added residual bitumen contents of the trial mixtures shall be set at increments of between 0.5% and

1.0% in the range 3.0% to 6.0%, with appropriate allowance made for residual binder in any crushed asphalt component. From each trial mixture, four 150 mm diameter x 75mm to 100mm high, cylinder test specimens (briquette specimens) shall be manufactured, compacted to refusal by vibratory compaction in a cylindrical metal mould, using the compaction mould assembly and vibrating hammer described in BS598: Part 104. The bulk density of each cylinder shall be determined. The briquette specimens shall be cured for a period of 72 hours at a nominal temperature of 60°C. Following this, the briquette specimens shall be conditioned in air for a minimum period 12 hours at 20°C and then immediately tested in accordance with BS DD 213 to determine the ITSM.

After further conditioning of the specimens, immersed in water at 24°C for a minimum period of 24 hours, the ITSM tests shall be repeated on each specimen. The characteristics of the mixture to be used in the works, including any added water, shall be determined using the optimum ITSM (dry) values. If peak conditions are not clearly displayed then plateau characteristics shall be accepted and the lowest added bitumen content for which all the criteria defined in Figure 3 are met, shall be used in the works subject to a minimum of 4.0% for mixtures containing only pulverised unbound or cement bound aggregate and 3.0% for mixtures containing only pulverised bitumen bound materials.

Property or characteristic	Individual specimens	Mean from test set
Moisture content	N/A	Optimum ±2%
Indirect Tensile Stiffness Modulus [dry]	2000MPa	2500MPa
Indirect Tensile Stiffness Modulus [wet]	1500MPa	2000Mpa
Bitumen content	N/A	Target ±0.5%
Particle size distribution	N/A	Zone A or Zone B
Air voids content	Maximum 9%	Maximum 7%

Figure 3. Acceptable design and characteristic requirements for recycled bitumen bound material

2.5 French mix design procedure

The method of study of hot mixes can not be transposed to mixes with the bitumen emulsion because it does not take into account their peculiarities and especially their evolutionary character. The formulation and the evaluation of the performances of the mixes with the emulsion call upon a series of adapted tests, carried out according to the methodology hereafter from French IDRRIM Guide.

The methodology has 4 phases, schematized by the following Figure 4.

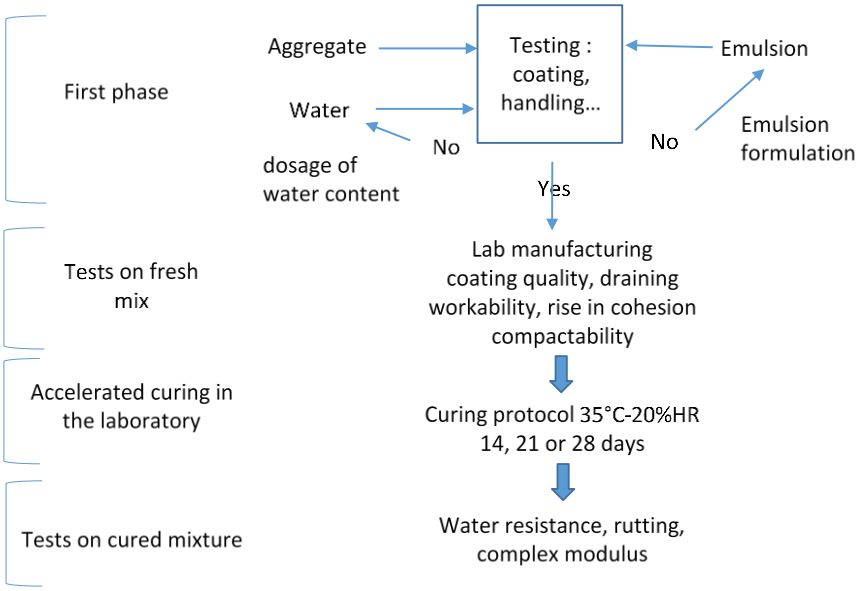


Figure 4. French mix design methodology

2.5.1 First phase

This phase begins with the selection of constituents. The choice obviously begins with the aggregates, whose source is the closest possible to the site. This prior choice of aggregates then dictates the selection of a suitable coating emulsion and the operator:

- checks the affinity of the aggregates and the emulsion,
- if it is suitable, hold the starting formulation,
- otherwise, reformulate the emulsion or choose another, etc.

Depending on the intended use of the asphalt mix and the observations regarding the affinity between the constituents, the formulator makes a choice of manufacturing process (order of introduction of the constituents, fractionation and / or pre-treatment).

Laboratory work consists of performing test sprays and visually evaluating coating, emulsion breaking, draining and workability by hand according to EN 12697-55. Once the emulsion has been chosen, this step makes it possible to set the dosage by binding and the water content.

2.5.2 Tests on fresh mix

Various types of mixer can be used (vertical axis or two horizontal shafts). The mixing procedure replicates as closely as possible the manufacturing procedure, planned or used, of the construction site: order of introduction of the constituents, distribution of the water and the emulsion within the granular fraction, possible precoating, etc. The temperature of the mixture

at the time of mixing must not defined. In the absence of more specific instructions related to site conditions, a temperature between 18 and 25 °C is recommended.

Feedback usually shows a good match between laboratory observations and those made in the field. On site, the quality of coating can vary considerably from one material to another. To evaluate and compare objectively, it must be quantified by the percentage of mineral surface covered by the bituminous binder. The ideal method is image analysis, on the material or digital photo. Most often, we are satisfied with visual observation (NF P 98-257-1 standard of August 2004). Imperfect coating is acceptable for the base course materials. On the other hand, for surface mixes, an incomplete coating can cause a sensitivity to water and, above all, induces the risk of degradation under traffic.

Observations on site show that some asphalt mixes coming out well coated with the mixer then partially disengage during the various handling operations they undergo, this phenomenon especially affecting the large grains. If we fear this tendency to stripping, it is appropriate to subject the fresh material to solicitations that induce debonding effect. Various possibilities exist, the simplest being to extend the mixing well after obtaining the maximum coating. The mix can also be left to rest for 24 hours and then reevaluate the coating quality. A strong tendency to stripping will lead the operator to reformulate.

For stripping with water, the approach is similar, by submitting the fresh mix to immersion or watering. The case is however extremely rare because of the surfactants conventionally used to be promoters of adhesion.

Depending on the petrographic nature of aggregates, the type of emulsion and their reactivity, the amount of water likely to drain in the mix from its manufacture to its implementation is very variable. It is important to know, on the one hand to adjust the water content in the manufacturing, on the other hand to define the precautions to take during transport. The drip test consists of measuring the amount of water that can drip from a fresh mix placed on an incline, simulating a storage area or a truck body, for 24 hours, under standard laboratory conditions. (18 to 28 ° C) or at the place of manufacture.

The ability to implement a mix with the emulsion (flow from the arrival of the truck, passage under the finisher table) depends on the degree of emulsion breaking, the viscosity of the binder, so the cohesion of the mixture. Two tests make it possible to evaluate the cohesion of the mixture at a given age. The repetition of one or the other of these tests after increasing time characterizes the appearance of the rise in cohesion. The workability test as described in EN 12697-53, consists of measuring the resistance force to a shear force exerted by advancing a plate pushed by a piston into a mold previously filled with a mix. The torque measurement test consists in measuring the torque necessary for the decohesion of a mix with the emulsion using a "blade-anchor" driven by a motor equipped with a torque measuring sensor. These tests can be used as aids to the formulation or simply to check that the mixture will not pose a workability problem on site.

The compaction ability of the mixes with the emulsion is studied in the gyratory Shearing compactor (GSC). Various models are usable. Some may be equipped with a dedicated device allowing the aspiration, collection and weighing of water extruded during densification. The feedback has shown that the tests at the GSC:

- make it possible to differentiate the compactability of a mix with the emulsion compared to another,
- do not provide a prediction of compactness at the end of the construction site, this in because of the great variability of the supports.

Overall, it appears that the compactness at 200 gyrations is an approximation of a mean void content corresponding to the plateau that the asphalt will reach in medium or long term depending on local conditions.

The curing protocol (14 days at 18° C and 50% relative humidity (RH)) does not really influence the asphalt behavior. It corresponds to the "fresh" state, reached in place after a few days or weeks, depending on weather conditions.

The standard compaction procedure (force 60 kN or 120 kN depending on the size maximum grain size and mold dimensions) leads to a level of compaction very high, even unrealistic. Indeed, in the majority of cases, this level is never achieved in place, even for a long time.

On the other hand, the "arranged" procedure (molding force divided by 3) gives a level of compaction which is an approximation of the compactions that will be obtained at the end of implementation, the exact value largely depends on the support.

The different modalities of the Duriez test also make it possible to evaluate the sensitivity to asphalt water at various levels of compaction and curing.

2.5.3 Accelerated curing in the laboratory

This step is a key point of the method, in the perspective of a performantial approach. For the protocol of curing of the test specimen, the parameters selected are: 14 days at 35° C and 20% relative humidity. These values were chosen to cause intense curing without causing thermal shock, cracking, reworking of the binder film, or premature aging.

The relevant accelerated cure time depends on the volume and geometry of the specimen. For "small" test bodies, a duration of 14 days is appropriate to simulate the "stability" state that the asphalt will reach in situ. The time required to achieve this stabilization depends on the reactivity of the asphalt and local weather conditions. In practice, this corresponds to 1 or 2 summers (exceptionally 3).

An example of a "small" test specimen is that made with a small gyratory device: diameter 80 mm, height 100 mm). A longer curing time is required for "large" test bodies, including GSC cylinders, Duriez cylinders, rut test plates, complex modulus and fatigue. Research is under way to specify the appropriate protocols.

2.5.4 Tests on cured mixture

It may be useful to measure the compressive strength of Duriez specimens having cured at 35 °C - 20% RH. These resistances are evaluated on dry specimens on the one hand, and on specuimens after 7 days of immersion at 18° C on the other hand. This procedure makes it possible to determine an immersion / compression ratio on asphalt. It is thus possible to determine the water resistance gain following curing.

Behavior monitoring in place shows almost no rutting. Two reasons explain this observation: on the one hand, cold mixes generally have a limited binder content; on the other hand, the traffic supported is weak or medium, so not or little channeled. Nevertheless, it is necessary to check the resistance to rutting as soon as the expected traffic exceeds a limit that can be located at class T3 (50 to 85 heavy vehicles per day). The tests with the rutter are carried out at 60° C on plates whose thickness depends on the type of asphalt. The tests with the rutter must be carried out on cured mixture brought to a void content close to that it will have in "medium term" (2 or 3 years after implementation). This implies to sufficiently compact the specimen, which is not always easy with a very “rubby” material. Various compaction protocols were tested but results are insufficient for the moment to standardize one or more protocol(s). Moreover, the appropriate curing of the test body, recommended at 35 ° C-20% RH, needs to be carried out for a fairly long period of time (at least 21 days for 5 cm plates, more for 10 cm plates).

The determination of the stiffness is carried out according to one or the other of the test methods described in standard NF EN 12697-26. For laboratory-made mixes, specimens must be compacted to a density close to that expected on the job site. They must be cured at 35° C-20% RH, for a time that is a function of the volume and the developed surface of the specimen, as well as the possible presence of fluxing agent.

The tests most often performed are those of indirect traction on a cylindrical specimen (IT-CY) and those of traction-compression on cylindrical specimens (TC-CY). Two-point bending on trapezoidal (2PB-TR) or prismatic (2PB-PR) specimens can also be used.

The behavior monitoring in place has not, to date, demonstrated fatigue failure similar to that observed with hot mixes. On the other hand, cases of degradation due to loss of cohesion have sometimes been noted with cold mixes. In the current state of knowledge, the use of the fatigue degradation model of hot mixes is therefore not applicable to cold mixes. The same is true of the corresponding flexural fatigue tests. It should also be noted that the evolutionary character of emulsion mixes must be taken into account in the behavior model.

2.6 Summary on mix design procedures

The summarised mix design procedures applied in five European countries follow a similar procedure. During mix design following stages are conducted:

a) Sampling of RA:

   For obtaining a representative sample material for the mix design, the reclaimed asphalt sample should comply with the material actually applied during the construction process. If a stockpiled RA in a mixing plant or other source is applied, the sampling procedure according to EN 13108-8 can be applied. In case of a mix design for a specific road maintenance project, sample material shall be obtained by trial milling of the real structure.

b) Analysis of RA characteristics:

   The reclaimed asphalt materials shall be thoroughly assessed according to EN 13043 and EN 13108-8. Most important information is the grading of the RA particles ("black rock"). The binder content and bitumen characteristics of the RA as well as its aggregate grading is required for checking the mix composition during the construction works but is of marginal importance of the actual mix design process.

c) Composition of mix granulate

   In all countries, the mix granulate shall meet a dense mix composition. As freshly milled reclaimed asphalt usually has low contents of fines (< 0,063 mm) and fine particles (< 2 mm), usually fine natural aggregate and/or fillers are added to the mixture in order to achieve a favourable gradation.

   In Figure 5, the grading envelopes applied in four of the five national specifications are compared. Whereas in Germany and Italy, similar grading of the mix granulate is specified, the Swedish requirements prescribe a lower fines content. In the UK, comparably high contents of fine grains are accepted, while the maximum grain size is limited to 20 mm. Especially in Italy and Germany, grain diameter up to 45 mm are accepted.

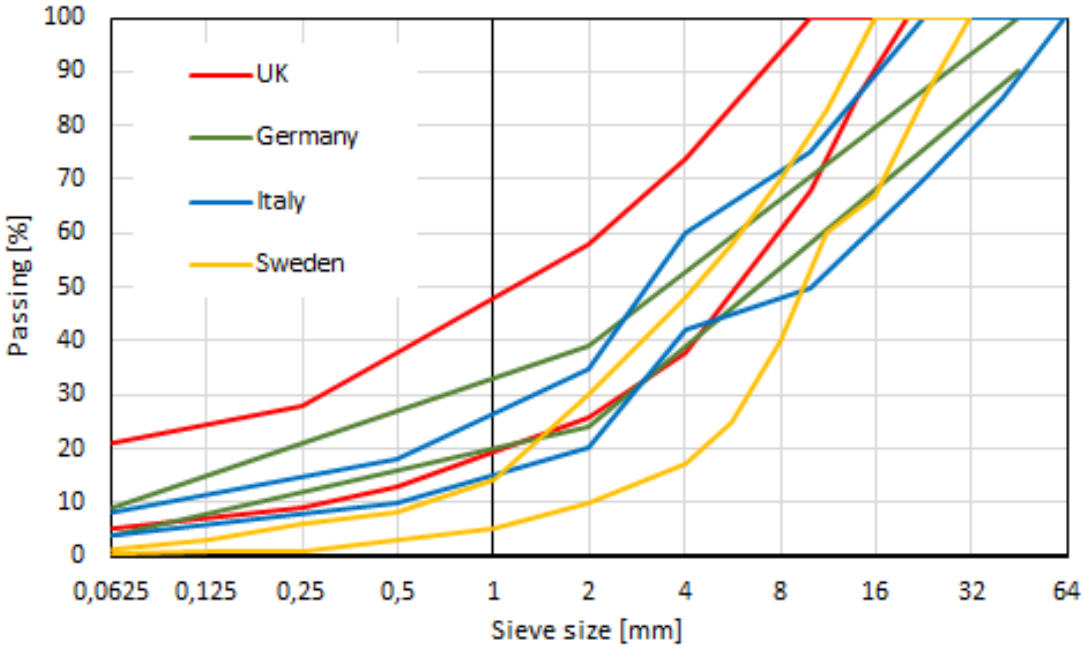


Figure 5. Grading envelopes for mix granulates for cold recycled materials according to four national mix design specifications

d) Assessment of compaction water content

For reaching water content enabling a good workability and compactability, modified Proctor tests according to EN 13286-2 or similar tests applying other compaction procedures (static compaction/Duriez according to EN 12697-12 with varied compaction loads, gyratory or vibratory) are applied. As results, the reference dry density and an optimal water content are derived. For evaluating the added water content for the mix preparation, the moisture of aggregate and the bitumen emulsion water content are subtracted from the optimum water content.

e) Selection of binders and binder content

Most mix design methodologies prescribe minimum contents for bituminous emulsion and cement addition according to Table 15. As bitumen emulsion, slow-breaking emulsions with unmodified bitumen are commonly applied. The emulsified bitumen usually applied in base layers is a 50/70, 70/100 or 160/220.

Table 15: Binder content specifications in national mix design procedures

Country	Content of binders [%]			
	bitumen emulsion	residual added bitumen	total bitumen (incl. RA)	cement
France	≥ 4,0	≥ 2,4	-	-
Germany	3,0 – 5,0	1,8 – 3,0	-	1,5 – 2,5
Italy	3,0 – 4,0	1,8 – 2,4	-	1,5 – 2,5
Sweden	1,3 – 2,8	0,8 – 1,7	4,4 – 6,5	-
UK	3	1,8	3,0 – 6,0	1,0*
*additionally fly ash can be added as filler to the mix granulate				

f) Laboratory mixing trials: mix preparation, compaction and curing

For finding suitable mix compositions, trial mixtures are usually prepared in laboratory with varied binder contents. After mix preparation at ambient temperatures, specimens are compacted using various compaction procedures. After compaction, the specimens

are cured in order to allow the breaking of the bitumen emulsion as well as to simulate medium-term and long-term field conditions of the material properties considering drying and hydration processes.

In Table 16, the applied compaction and curing procedures are summarised.

Table 16: Applied compaction and curing procedures for mix design

Country	Compaction procedure	Curing procedure		
		temperature [°C]	moisture [%]	Duration [d]
France	Duriez (static) EN 12697-56: σ_{comp} = 15 MPa	35	20	14
Germany	Duriez (static) EN 12697-56: σ_{comp} = 2,8 MPa	20	~95 (sealed) 40 - 70	2 28
Italy	Gyratory EN 12697-31 10 gyr.; 0,6 MPa	40	-	3
Sweden	Gyratory EN 12697-31 ≤200 gyr.; 0,6 MPa or Duriez (static) EN 12697-56: σ_{comp} = 7,0 MPa	40	-	7
UK	Vibratory EN 12697-32	30	-	3

g) After curing, the void content as well as mechanical properties of the specimen are tested and checked if required values are obtained. Besides the strength of the mixtures, derived by indirect tensile tests or uniaxial compression test (Duriez), the stiffness modulus as well as the water sensitivity are controlled.

The specification limits as well as test conditions are summarised in Table 17.

Table 17: Specification limits and test conditions for assessment of mechanical properties during mix design

Specification		France****	Germany	Italy	Sweden	UK
Void content		≤ 15% (≤ 12%) @ 100 gyrations	8 – 15 %	-	6 – 14 %	≤ 7 %
Strength [MPa]	procedure*	UCS, 18 °C	ITS, 5°C	ITS, 25 °C	MS, 25°C	-
	specification	≥ 2,5 (≥ 3,5)	0,7 – 1,0	≥ 0,35	≥ 7	-
Stiffness [MPa]	procedure	2PB, 15°C ITSM, 10°C	ITS**, 5 °C	ITSM, 20°C	ITSM	ITSM, 20°C
	specification	≥ 1.500 (≥ 2.500)	3.000–7.000	≥ 3.000	≥ 2.000	≥ 2.500
Water sensitiv-ity*	water cond.	7d @ 18°C	14d @ 20°C	1h, vac.sat.	1h, vac.sat + 23 h @ 25°C	
	specification	≥ 55 %	≥ 70 %	≥ 70 %	> 50 %	≥ 2000***

UCS: Uniaxial Compression Strength (Duriez), EN 12697-12
ITS: Indirect tensile strength (EN 12697-23)
2PB: 2-pont-bending (EN 12697-26, method A
MS: Marshall stability (EN 12697-34)
ITSM: Indirect tensile stiffness (EN 12697-26, method C)
*Water sensitivity: remaining strength/stiffness [%]
** stiffness measured in monotonic indirect tensile test
*** stiffness after water conditioning
**** Class 1 (Class 2)

3 Materials in mix design study

In sections 3 to 6, variations of CRM mixtures are assessed by experimental methods applied according to selected national mix design procedures. In section 3, the source materials and assessed binder contents are presented. The applied test procedures are presented in section 4, whereas the obtained results are summarised in section 5 and discussed in section 6.

3.1 Source materials

Aggregates employed for producing CRM mixtures were RA, basalt sand and limestone filler. The aggregates were supplied by University of Kassel. The RA were sampled from a stockpile of an asphalt mixing plant and had a nominal maximum grain size of 16 mm. The RA binder content was measured to 4,7 %, the softening point of the binder, recovered from RA was 62,4 °C. Table 18 resumes the main properties of the aggregates. Figure 6 displays the grading distribution of the RA grains.

Table 18. Main properties of aggregates

Aggregate	density (g/cm³)	WA_{24} (%)
Reclaimed asphalt 16 RA 0/16	2,737	1,5
crushed sand (0/2)	3,138	1,8
limestone filler (< 0,063 mm)	2,800	0,07

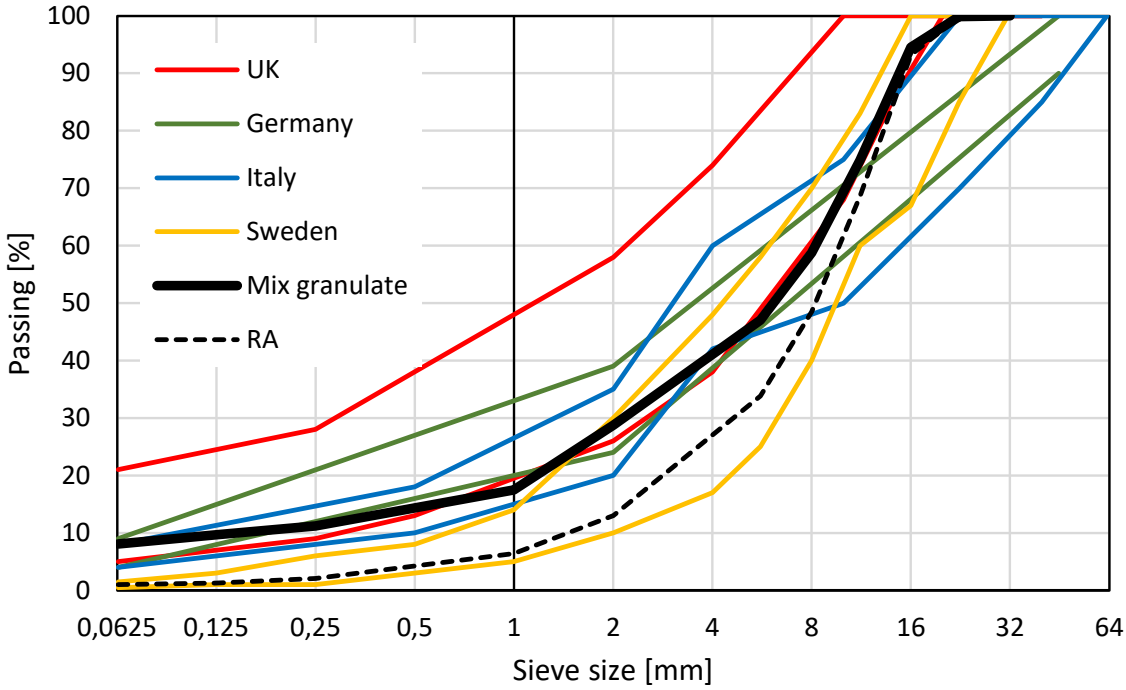


Figure 6. Grading of RA grains and designed mix granulate with added filler and 0/2 aggregates compared to the national specification envelopes

The mixture aggregate blend consisted of 80% RAP, 14% basalt sand and 6% limestone filler. Figure 6 reports its grading distribution.

The bituminous binder was a cationic slow-setting bitumen emulsion, supplied by Valli Zabban S.p.A. (Bologna - Italy), with a residual bitumen content of 60%. The emulsion base bitumen was 70/100. Furthermore, a second emulsion supplied by Esha Strasse GmbH (Germany) was

used within the laboratory programme. Both emulsions were specifically designed for cold recycling applications and coded C60B10 (EN 13808). The technical specifications are given in Table 19.

The cementitious binder was a Portland cement CEM I 42.5 R (EN 197-1).

Table 19: Technical specification of the used bitumen emulsions

Emulsion		
Binder content (%)	EN 1428	60
Viscosity at 40 °C / efflux time at 40 °C (s)	EN 12846-1	15/70
Breaking behaviour (with mineral filler) (%)	EN 13075-1	110–195
Mixing stability with cement (%)	EN 12848	< 2
Residual binder		
Penetration at 25 °C (mm/10)	EN 1426	100
Softening point (°C)	EN 1427	43

3.2 Mix variations

Within the experimental study, four mix designs were prepared and tested. The applied binder contents of bitumen emulsion and cement were selected according to the specification ranges identified within the national mix design study. This resulted in four combinations of bitumen and cement addition, see Table 20.

The residual bitumen dosage was 2%, corresponding to 3,3% of emulsion, by dry aggregate mass (mixtures "A" and "B") as well as 3,5% bitumen, corresponding to 5,8% of emulsion (mixture code “C” and “D”).

Two cement dosages were investigated, 1,5% (mixture coded as “A” respectively “D”) and 3% (mixture coded as “B”) and by dry aggregate mass. The total water content is controlled by the aggregate composition and therefore was equal to 4,5% by dry aggregate mass, which comprises aggregate absorption water, water brought by the emulsion and additional water.

According to the definitions of CRM materials types, the assessed mixtures can be classified as followed:

- A: bitumen stabilised CRM: BSM (as applied for example in Italy)
- B: bitumen-cement stabilised CRM: CBTM (as applied for example in Germany)
- C: GE-type CRM: CRA (Grave emulsion) (as applied in France)
- D: sealing CRM: CRA (as applied in UK and Germany)

These mix compositions further allow to assess the effect of bitumen and cement content to the mechanical properties of the CRM.

Table 20. Composition of CRM mixtures investigated

Material		**A**	**B**	**C**	**D**
Cement dosage	%	1,5	3,0	0,0	1,5
Emulsion dosage (Residual bitumen)	%	3,3 (2,0)	3,3 (2,0)	5,8 (3,5)	5,8 (3,5)
Total water	%	4,5	4,5	4,5	4,5

4 Experimental Methods

The four mixtures were prepared and tested in the three collaborating laboratories of Polytechnic University of Marche (UPdM), Uni Kassel (UKa) and Uni Gustave Eiffel (UGE). Therefore, each laboratory applied the individually available mixing, compaction, curing and test facilities.

4.1 Mixing procedure

Aggregates were first dried until reaching constant mass, at (105 ± 2) °C for sand and at (40 ± 2) °C for RA. The aggregate absorption water was added to the dry aggregate blend, and the wet samples were stored in a sealed plastic bag at least for 12 h at room temperature. After that, cement, water and bitumen emulsion were gradually added and mixed in this sequence to the aggregate blend. Mixing was carried out alternating mechanical and hand mixing to guarantee a good particle coating, checked by visual examination. In Figure 7 to Figure 9, f the applied mixers are shown. The whole mixing process required about 15 min. Three specimens were obtained from each batch of mixed material (about 9 kg for mixtures).

Figure 7. Mixer UPdM (ITA)

Figure 8. Mixer UKa (GER)


Figure 9. Mixer Uni Gustave Eiffel (FRA)

4.2 Compaction procedures

4.2.1 Gyratory compaction (UPdM)

Specimens were compacted with a gyratory compactor, adopting the following protocol: constant pressure of 600 kPa, gyration speed of 30 rpm and angle of inclination of 1,25 °. Compaction was carried out in fixed height mode. Final specimens were 60 mm height. To obtain the target content of voids, the final weight of the specimens was fixed. The mould diameter was 150 mm.

During compaction, the specimen height was recorded at each gyration, which allowed monitoring the voids (V_m) and the voids filled with liquids (VFL) (Graziani *et al.* 2016, Grilli *et al.* 2016):

$$V_m = \frac{v_A + v_W}{v} = \frac{v - (v_S + v_B)}{v} \quad (1)$$

$$VFL = \frac{v_B + v_W}{v_A + v_B + v_W} = \frac{v_B + v_W}{v - v_S} \quad (2)$$

where v is the total volume of the specimen, v_A is the volume of the air voids, v_S is the bulk volume of solids (aggregates, filler and unhydrated cementitious binder), $v_B$is the volume of residual bitumen from the emulsion and v_W is the volume of intergranular water (the volume of absorbed water is comprised in the bulk volume of the aggregate). Because v_W cannot be measured, from a practical point of view, V_m is calculated considering v, v_S and v_B.

Equations (1) and (2) are accurate only if material loss during compaction is negligible. To check any material loss during compaction, the mould was weighed before compaction and right after its end, before extruding the specimen.

Immediately after the end of compaction, specimens were extruded (Figure 10).

Figure 10. Extrusion of the gyratory compactor specimen

4.2.2 Vibratory compaction (Uni Kassel)

One part of the specimens within the experimental programme of the University Kassel were compacted with vibratory compactor (Figure 11), Mollenhauer, 2017. The following protocol was adopted for the vibratory compacted specimens for triaxial and indirect tensile strength tests: 30 seconds compacting time with an individual impact force of 16,8 Joule per blow. The mould diameter was 150 mm. For Triaxial-specimens the compaction was carried out in five layers.

In general, the ITS-specimens were extruded 1 - 3 hours after the end of compaction. Because of the higher mass and therefore the quite high amount of water within the specimen of the same diameter, the Triaxial-specimens were extruded after 24 hours from their moulds.

4.2.3 Static compaction (UGE, Uni Kassel)

For another set of specimens at Uni Kassel and Uni Gustave Eiffel, a static compaction was applied according to EN 12697-56, adopting the following protocols.

- Uni Kassel: axial compaction pressure: 49 kN for 5 minutes for a specimen diameter of 150 mm.
- UGE: axial compaction pressure: 60 kN for 5 minutes for a specimen diameter of 120 mm.

In general, 1 - 3 hours after the end of compaction, the specimens were extruded from their moulds.

Figure 11. Used vibratory compactor

Figure 12. Set up for static compaction at Uni Kassel (left) and University Gustave Eiffel (right)

4.3 Curing

Curing was carried out considering national boundary conditions for three different temperatures (20° C, 35° C and 40° C). Table 21 shows a compilation of the various procedures. Two curing conditions were considered:

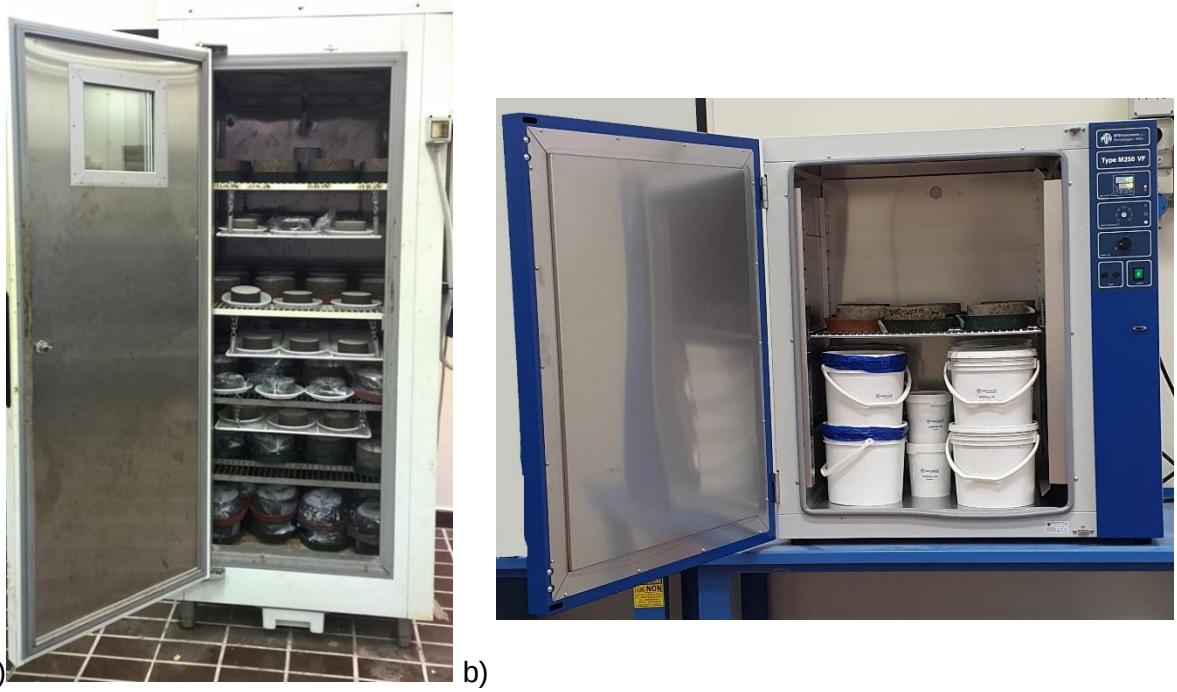
- Temperature=20°C and RH=(55 ± 15)%
- Temperature=35°C and RH=(20± 5)%
- Temperature=40°C and RH=(20 ± 5)%

All specimens were cured in unsealed conditions, allowing the free water evaporation (Figure 13).

Several curing times were adopted with the aim of monitoring the development of physical properties evolution, i.e. water loss by evaporation: 0.5, 1, 2, 4 hours, 1, 2, 3, 7 and 28 days. The CRM specimens' mechanical properties were measured after 3, 14 and 28 days of curing.

Table 21. Compilation of curing condition according to national standards

UPdM (ITA)	UK (GER)	UGE (FRA)
20° C: - climate chamber - unsealed conditions - (70 ± 5) % RH	20° C: - climate chamber - sealed conditions for 48 hours after compaction - unsealed conditions after 48 hours; (55 ± 15) % RH	35°C: - climate chamber - unsealed conditions - (20 ± 5) % RH
40° C: - oven - unsealed conditions	40° C: - oven - sealed conditions for 48 hours after compaction (immediately) - unsealed conditions after 48 hours	



a) b)

Figure 13. Curing of CRM specimens (ITA) a) climate chamber, b) oven

4.4 Volumetric analysis

The analysis of the volumetric properties of CRM specimens was carried out both at the fresh state (i.e. during the compaction process) and cured state.

The voids in the mixture at the fresh state, $V_{m,fresh}$, i.e. volume occupied by the intergranular water and air $V_{m,fresh}$ were obtained as (Grilli *et al.* 2012):

$$V_{m,fresh} = \frac{\rho_m - \rho_d}{\rho_m} \tag{3}$$

where ρ_m is the maximum density of the mixture and ρ_d is the specimen dry density.

$$\rho_d = \frac{p_{aggregate} + p_{abs,water} + p_{filler} + p_{bitumen} + p_{cement}}{v} \tag{4}$$

where p_i is the mass of the CRM constituent.

With curing, part of the water is bonded by the cement in its hydration process. The remaining part gradually evaporates, and its volume is occupied by air voids. A small amount may remain trapped in not-interconnected voids.

At the cured state $V_{m,cured}$ were evaluated according to EN 12697-8:

$$V_m = \frac{\rho_m - \rho_b}{\rho_m} \tag{5}$$

where ρ_b is the specimen bulk density.

However, in CRM mixtures, differently to traditional asphalt concrete, ρ_m and ρ_b depend on the cement hydration degree and consequently can change with curing time. Indeed, the hydrated cement consists of reacted cement, and the non-evaporable water. The non-evaporable water is the water that is bound chemically with the cement. Also, the physically bound water is adsorbed at the surfaces of gel particles and occupies the gel pores. Free unbound water occupies the larger capillary pores. Both physically bound water and free water constitute the evaporable water (Brouwers 2004). According to Powers' measurement, chemically bound water is about 0,23 g per gram of cement reacted during hydration, whereas the physically bound water is about 0,19 g per gram of cement reacted. The volume of the reaction products developed during the hydration is lower than the volume of cement and water reacted. This volume reduction, called chemical shrinkage, is about 6,4 ml/100 g cement reacted (Jensen and Hansen 2001). The volumetric composition of the cement paste depends thus on the degree of the hydration reaction, α. Its value ranges from 0 for the unhydrated cement, to 1 if the cement is fully hydrated. As only the capillary water is freely accessible for cement hydration, the complete cement hydration is possible only if the water to cement ratio is above 0,42 (= 0,23 + 0,19). If W/C ratio is lower, part of the cement will not hydrate (α < 1) (Powers 1958, Jensen and Hansen 2001). Therefore, the water bonded by Portland cement is $0{,}42 \cdot \alpha \cdot p_{cement}$.

To consider this effect, ρ_m was obtained according to EN 12697-5, Procedure C.

$$\rho_m = \frac{100}{\frac{p_{aggregate}}{\rho_{aggregate}} + \frac{p_{abs,water}}{\rho_{water}} + \frac{p_{filler}}{\rho_{filler}} + \frac{p_{bitumen}}{\rho_{bitumen}} + \frac{p_{cement}}{\rho_{cement}} + \frac{0{,}42 \cdot \alpha \cdot p_{cement}}{\rho_{water}}} \quad (6)$$

where ρ_i is the density of the CRM constituent. $\frac{p_{abs,water}}{\rho_{water}}$ is the volume of the aggregate pores.

ρ_b was measured according to EN 12697-6, procedure D (bulk density by dimension). However, the equation reported in the standard must be corrected for taking into account the presence of residual water, i.e. non evaporated, and the cement hydration degree as follows:

$$\rho_b = \frac{p_j - p_{water,residual} + 0{,}42\,\alpha\,p_{cement}}{\frac{\pi}{4}\,h\,d^2} \quad (7)$$

where p_j is the mass of the specimen at the curing time j, $p_{water,residual}$ is the mass of the residual water in the specimen (obtained as the difference between the total water and the evaporated water). h and d are the specimen height and diameter.

4.5 Testing

4.5.1 Water loss by evaporation

The water loss by evaporation (DW) of each specimen was measured by weighing the specimens (Figure 14):

$$DW = \frac{M_0 - M_i}{M_W} \cdot 100 \quad (8)$$

where M_0 is the specimen mass right after compaction, M_i is the specimen mass after *i* curing days and M_W is the total mass of water in the specimen (derived from its gravimetric composition). Specimens were stored on a plastic plate to avoid any material loss.

Figure 14. Weighing of the CRM specimen before the start of the curing (ITA)

4.5.2 Indirect tensile strength test

The indirect tensile strength (ITS) was measured using a servo-hydraulic testing machine following the procedure described by the standard EN 12697-23. The test was performed applying a constant rate of deformation of (50 ± 2) mm/min until specimen failure.

$$ITS = \frac{2 \cdot P}{\pi \cdot D \cdot h} \tag{9}$$

where P is the maximum load, D is the specimen diameter and h is its mean thickness.

During the tests at UPdM, the horizontal deformation u of the specimens was measured as well using two transducers set at the horizontal diameter. ITS tests were carried out at two different temperatures: 5 °C and 25 °C. Prior to testing, the spoecimens were conditioned at the test temperature for at least 4 hours.

4.5.3 Duriez Test

According to French norm NF P98-251-4, Duriez test evaluates the compression strength of cylindrical specimens of bituminous mixtures. A set of cylindrical test specimens is maintained at 18°C and 50 % humidity during 7 days. Then the set is divided into two equally sized subsets and conditioned. One subset is maintained at 18°C and 50 % humidity for 7 days while the other subset is saturated and stored in water. After conditioning, the compression strength of each of the two subsets is determined at 18 °C. The ratio of the compression strength of the water conditioned subset compared to that of the dry subset is determined and expressed in percent. The speed of the press is set at 1 mm / s. The simple compressive strength is determined at from the maximum breaking load of the test specimen. The compressive strength ratio, i/C, is calculated by the ratio Cw/Cd * 100 (where Cw is the average compression strength of the wet group and Cd is the average compression strength of the dry group).

4.5.4 Triaxial test

According to the Technical Guideline of the Southern African Bitumen Association (Sabita, 2020) the triaxial shear parameters (cohesion and internal friction angle) was identify by using the monotonic triaxial test. The test was performed by measuring the resistance to failure

(monotonic) of a cylindrical 150 mm diameter and 300 ± 2 mm height specimen prepared according to vibratory hammer compaction procedure as described previously. The confining pressure within this test programme was 0, 100 and 200 kPa. The test was carried out at 25° C.

$$\sigma_1 = \frac{P}{A} \cdot 10^{-3} \quad (10)$$

Where P is the maximum load and A the area of cylindrical specimen at beginning of the test. According to Jenkins et al. 2007 the relationship between major principle stress $\sigma_{1,f}$ and confinement stress σ_3 is described by:

$$\sigma_{1,f} = A \cdot \sigma_3 + B \quad (11)$$

Where

$$A = \frac{1 + sin\varphi}{1 - sin\varphi} \quad (12)$$

And

$$B = \frac{2 \cdot C \cdot cos\varphi}{1 - sin\varphi} \quad (13)$$

The values of A and B can be determined by linear regression analysis. The cohesion (c) and the internal friction angle (φ) can be calculated as follows:

$$\varphi = sin^{-1}\left(\frac{A-1}{A+1}\right) \quad (14)$$

$$c = \frac{B(1 - sin\varphi)}{2 \cdot cos\varphi} \quad (15)$$

4.5.5 Stiffness by cyclic indirect tensile stress test

For the assessment of the stiffness of CRM mixtures, the cyclic indirect tensile stress test (CIT-CY) according to EN 12697-26, Annex F, is applied. The deformation measurement system includes two LVDTs which are placed centrically on the cross-sectional area of the specimen. The measurements obtained during the tests are applied vertical force F and horizontal displacement u. From these the horizontal tensile stress, horizontal strain and stiffness are computed.

For the determination of the stiffness S(T), multistage tests are conducted at four temperatures (-10 °C to 20 °C) and varied loading frequencies between 0,1 Hz and 10 Hz. The stiffness modulus is calculated from vertical force and horizontal deformation measurements as the average value of three tested specimens. By time-temperature superposition principle, the stiffness master curve is determined. This allows the interpolation of the asphalt stiffness for any temperature condition as needed for the mechanistic-empirical pavement design procedure for the reference frequency of 10 Hz.

$$S(T) = \frac{E_\infty}{e^{(z_1 °C \cdot f^* + z_2)}} \quad (16)$$

where S(T) is the stiffness at a given Temperature and frequency, E_∞, z_1, z_2 are regression factors and

$$f^* = \log(f \cdot \alpha_T) = \log\left(f \cdot e^{\left(\phi\left(\frac{1}{T}-\frac{1}{T_0}\right)\right)}\right) \quad (17)$$

Is the reduced frequency after application of time-temperature-superposition, controlled by the parameter ϕ and the reference temperature T_0 (displayed in [K]).

The cyclic indirect tensile stress tests were conducted on cylindrical specimen obtained from vibratory compaction in a diameter of 150 mm and a height of 60 mm.

4.6 Summary of test program

Table 22 summarises the complete experimental programme identifying the applied compaction, curing and test procedures.

Table 22.Summary of the experimental programme

		UPdM (ITA)	UK (GER) (a)	UK (GER) (b)	UGE (FRA)
Material assessed		A, B	B	A, B, C, D	C, D
Specimen compaction		Gyratory	Static	Vibratory	Static
Curing temperature	°C	20, 40	20, 40	20	35
Moisture: water loss by evaporation					
Curing time	days	3,28	3, 28	-	-
ITS					
Curing time	days	3, 28	3, 28	28	1, 14, 28
Test temperature	°C	5, 25	5, 25	25	25
Number of replicates		48	24	12	8
Duriez test					
Curing time					1, 14, 28
Test temperature					18
Number of replicates					8
Triaxial					
Curing time	days			28	
Test temperature	°C			20	
Number of replicates				24	
Stiffness CIDT					
Curing time	Days			28	
Test temperature	°C			-10, 0, 10, 20	
Number of replicates				16	

5 Results

5.1 Water loss by evaporation

Figure 15 and 14 depicts DW evolution from 30 minutes to 28 days of curing for mixtures A to D cured at 20 and 40°C.

All the mixtures showed similar behaviour and showed a higher DW evaporation rate in the first curing days, then it slowed down tending to an asymptotic value. Specifically, DW was highly influenced by the curing temperature. DW evolution rate was higher for specimens cured at 40°C which lost about 60% of the total water in the first curing day. DW of specimens cured at 20°C was about half those of specimens cured at 40°C. After 28 days DW was on average 65% and 87% for mixtures cured at 20°C and 40°C, respectively. This measurments highlight that the curing process was not yet completed after 28 days of curing at the lower temperature of 20 °C.

The cement dosage also influenced DW. Mixtures B, characterised by the higher cement dosage, always had lower DW than specimens of mixtures A in the long-term. The different DW is directly linked to the water bonded by the cement and consequently not-evaporable. On the first curing day at 20 °C, DW of specimens of mixtures A and B were comparable, suggesting a slower hydration rate of cement than those occurring at the higher curing temperature.

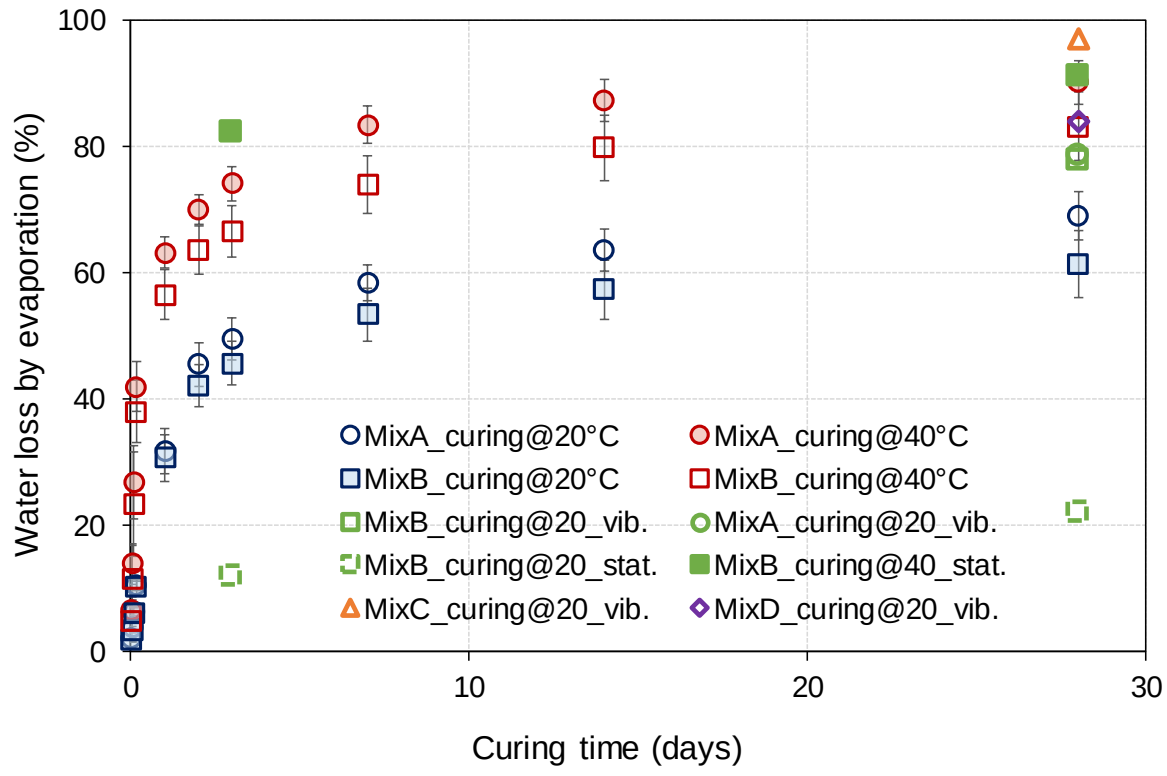


Figure 15. Evolution of water loss by evaporation with curing time

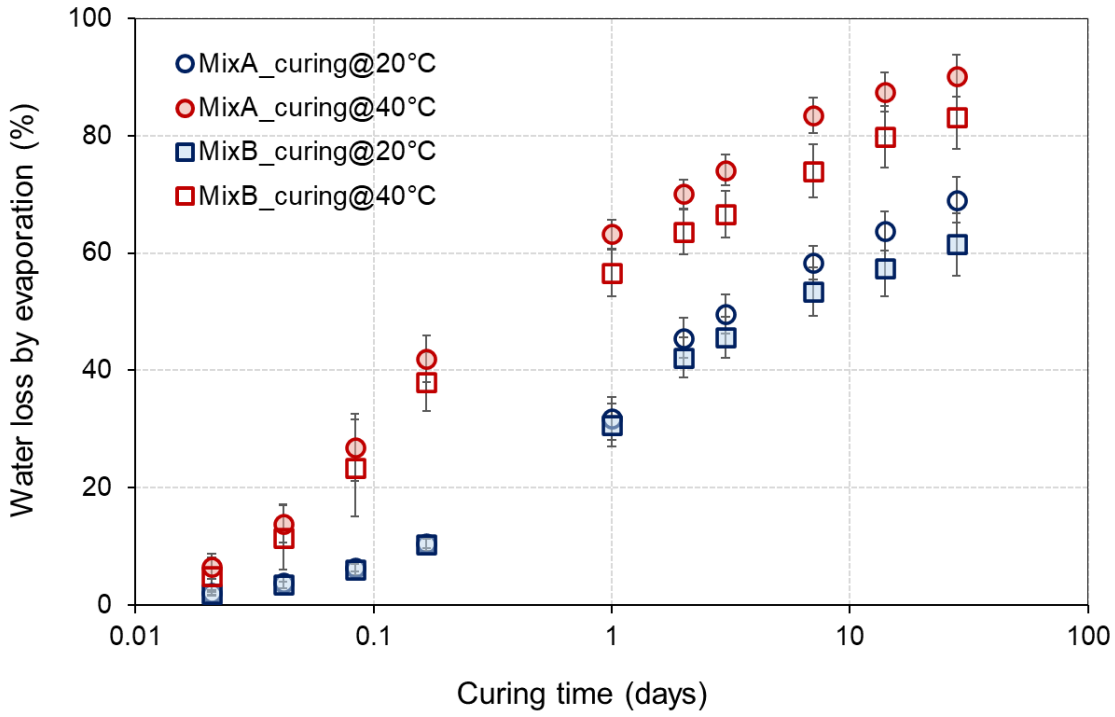
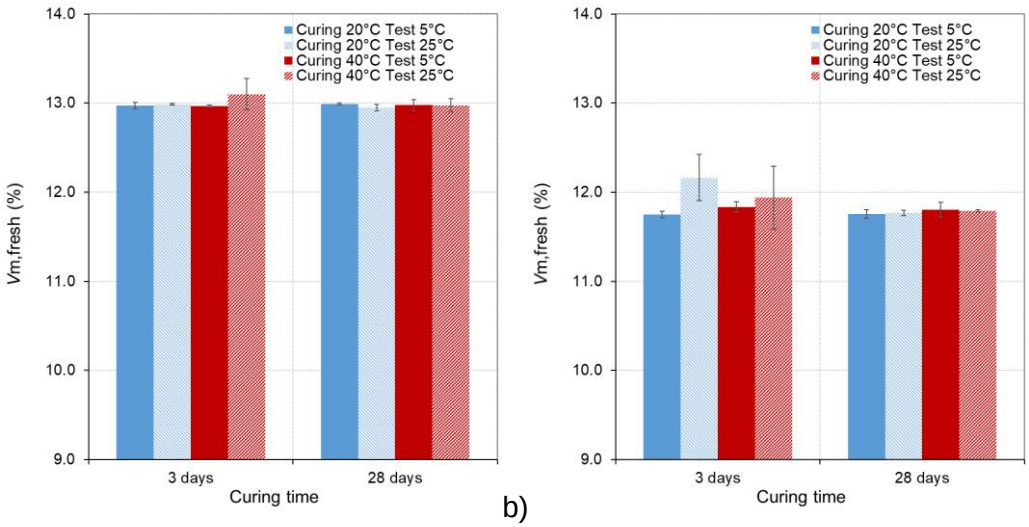


Figure 16. Evolution of water loss by evaporation with curing time (log scale)

5.2 Volumetric properties

Figure 17 depicts the average V_{mfresh} values of the CRM mixtures investigated. V_{mfresh} was about 13% and 11.9% for mixtures A and B, respectively. The compaction procedure adopted allowed obtaining a good specimens homogeneity in terms of volumetric properties.



a) b)

Figure 17. Voids in the mixture at the fresh state a) mixture A, b) mixture B

Table 23 reports the specimen composition derived from the binder extraction and the calculated maximum density.

Figure 18 depicts the average $V_{m,cured}$ values (Equation 5) obtained using the bulk density calculated using Equation 7 and the maximum density values reported in Table 24. ρ_m and ρ_b

were calculated considering different hydration degrees as a function of the curing temperature and time. In particular, it was assumed α:

- 0,5 after 3 curing days at 20 °C
- 0,8 after 3 curing days at 40 °C and 28 curing days at 20 °C
- 0,9 after 28 curing days at 40 °C.

Specimens of mixture B had generally lower voids than those of mixture A. Besides, the higher curing temperature and the longer curing time led to the lower V_m . These results are directly related to the cement hydration degree and the volume occupied by its hydration products. As expected, the V_m of specimens tested at 5°C and 25°C were generally comparable at the variability can be mainly linked to some differences in the compaction process.

Table 23. Composition of CRM mixtures measured after the binder extraction

	Mix A		**Mix B**	
	Composition by mass (%)	**Density (Mg/m³)**	**Composition by mass (%)**	**Density (Mg/m³)**
Aggregate	80,6	2,896	81,6	2,896
Filler	14,6	2,800	13,5	2,800
Bitumen	4,8	1,015	5,0	1,015
WA_{24}*	1,2	1,000	1,2	1,000
Mixture		2,581⁺		2,574⁺

* by dry aggregate mass, ⁺ maximum density of the mixture

Table 24. Maximum density of CRM mixtures

	Maximum density (Mg/m³)	
Hydration degree	**Mix A**	**Mix B**
0	2,604	2,609
0,5	2,591	2,584
0,8	2,584	2,569
0,9	2,581	2,565
1	2,579	2,560

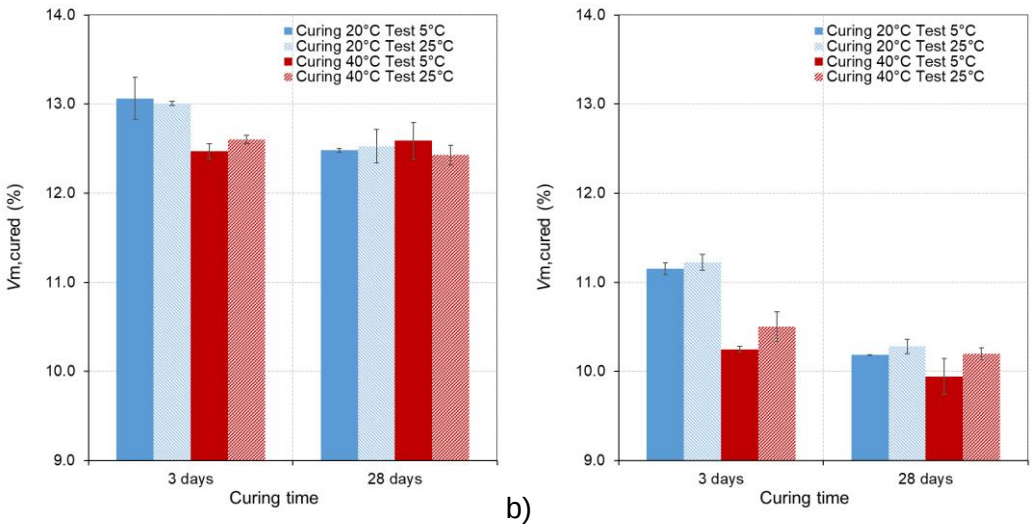


Figure 18. Voids in the mixture at the cured state as a function of curing time (and cement hydration degree) a) mixture A, b) mixture B

Within Figure 19 the average $V_{m,cured}$ of specimens of mixture B produced by static compaction are shown. In general, the voids content range between 14,8 % and 18,6 %. It becomes obvious that curing at 40° C results in higher void contents. This can be achieved by the higher water loss caused by higher curing temperatures (compare section 5.1). All specimens, which were compacted by vibratory compaction, result in void contents above 20 % (Figure 20). Highest $V_{m,cured}$ is observed by mixture C.

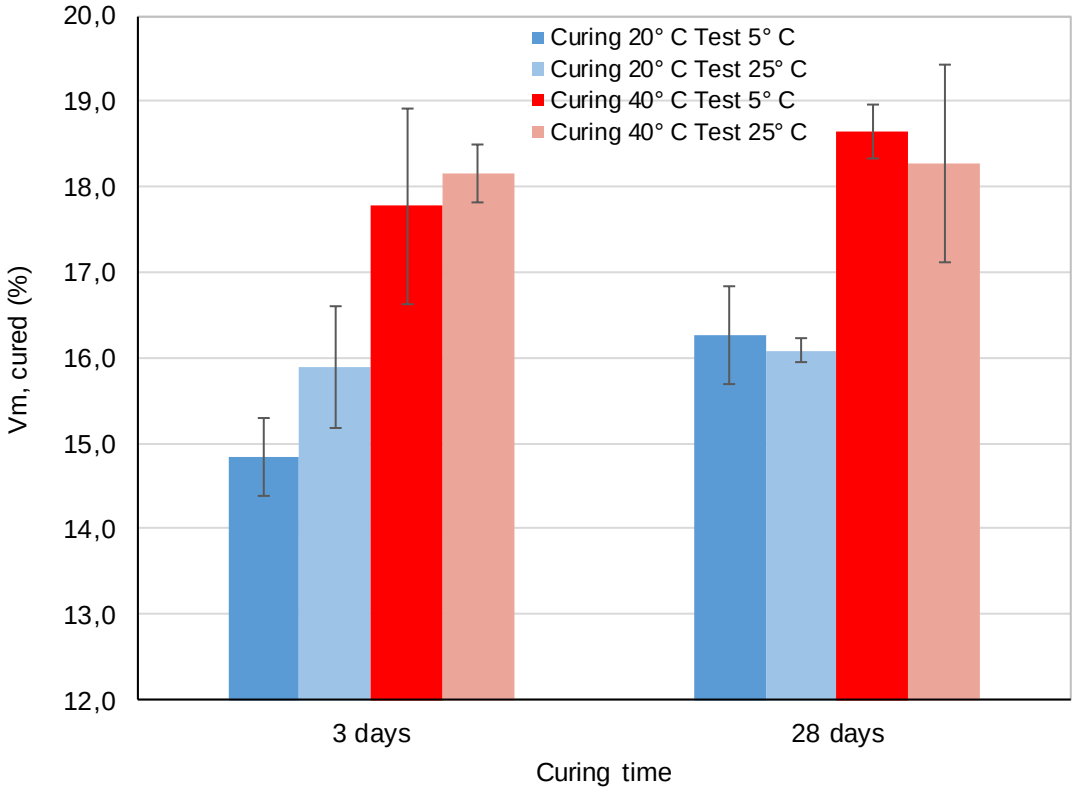


Figure 19. Voids in the mixture at the cured state as a function of curing time mixture B (static compaction)

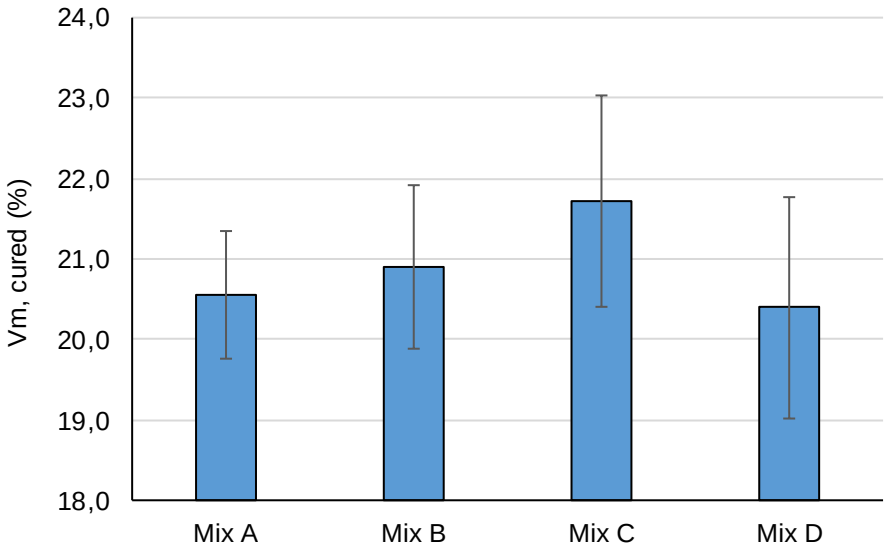


Figure 20. Voids in the mixture A to D (vibratory compaction) at the cured state after 28 days by 20° C

5.3 Indirect tensile strength

Figure 21 and Figure 22 report the measured values of the ITS as a function of curing conditions within the Italian laboratory. Specimens of mixture A had lower ITS than those of mixture B due to the lower cement dosage. The higher curing temperature and longer curing time led to higher strength.

The measured values of the indirect tensile strength within the German laboratory are given in Figure 23 and Figure 24. ITS measured at 5 °C was generally higher than those measured at 25 °C. The specimens compacted by the vibratory compactor resulted in lower ITS compared to the static compaction.

The application of a bitumen emulsion of the same type but produced by another supplier (in Germany) results in very similar ITS values compared to the originally applied bitumen emulsion from an Italian producer (Figure 25 and Figure 26).

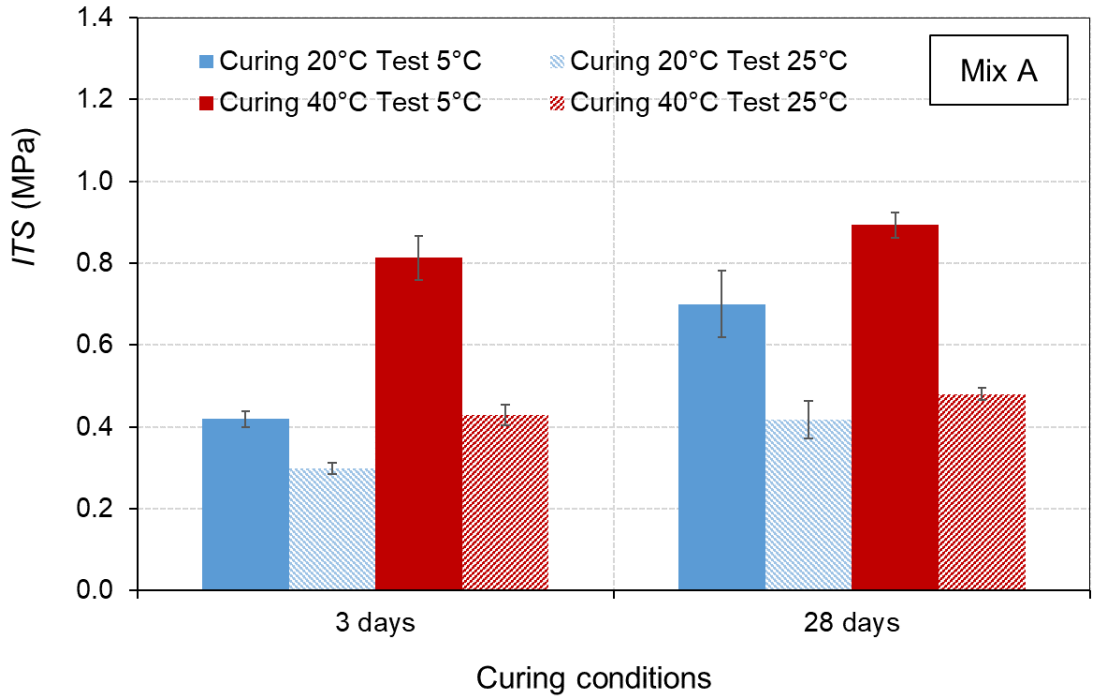


Figure 21. Indirect tensile strength test results obtained for mix A in Italy

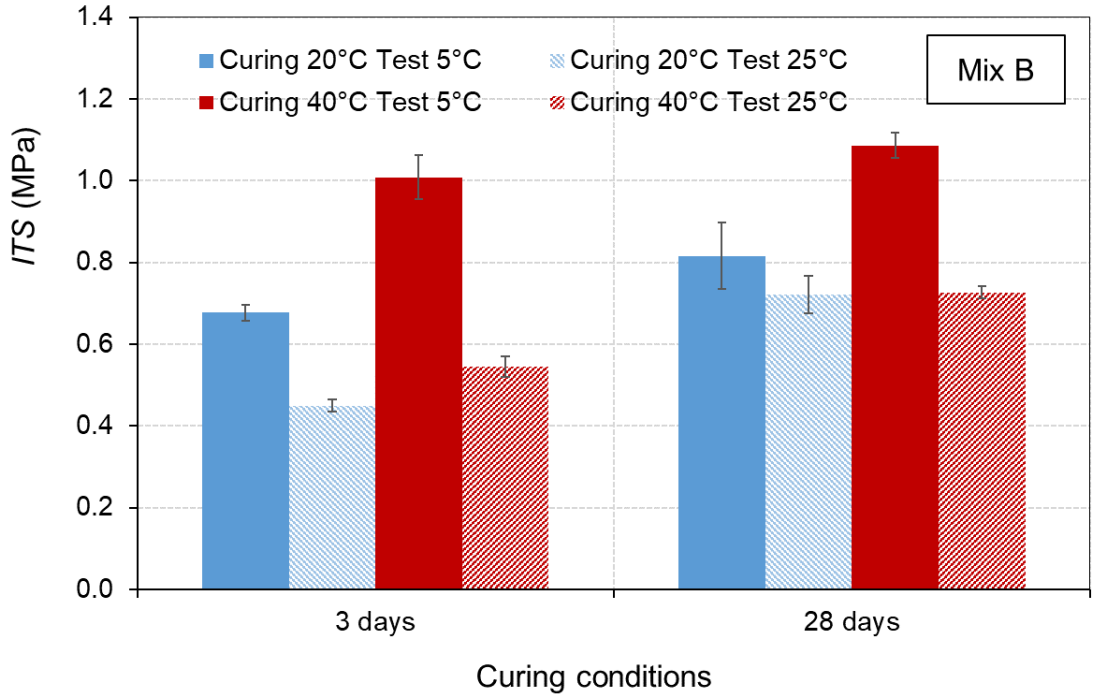


Figure 22. Indirect tensile strength test results obtained for mix B in Italy

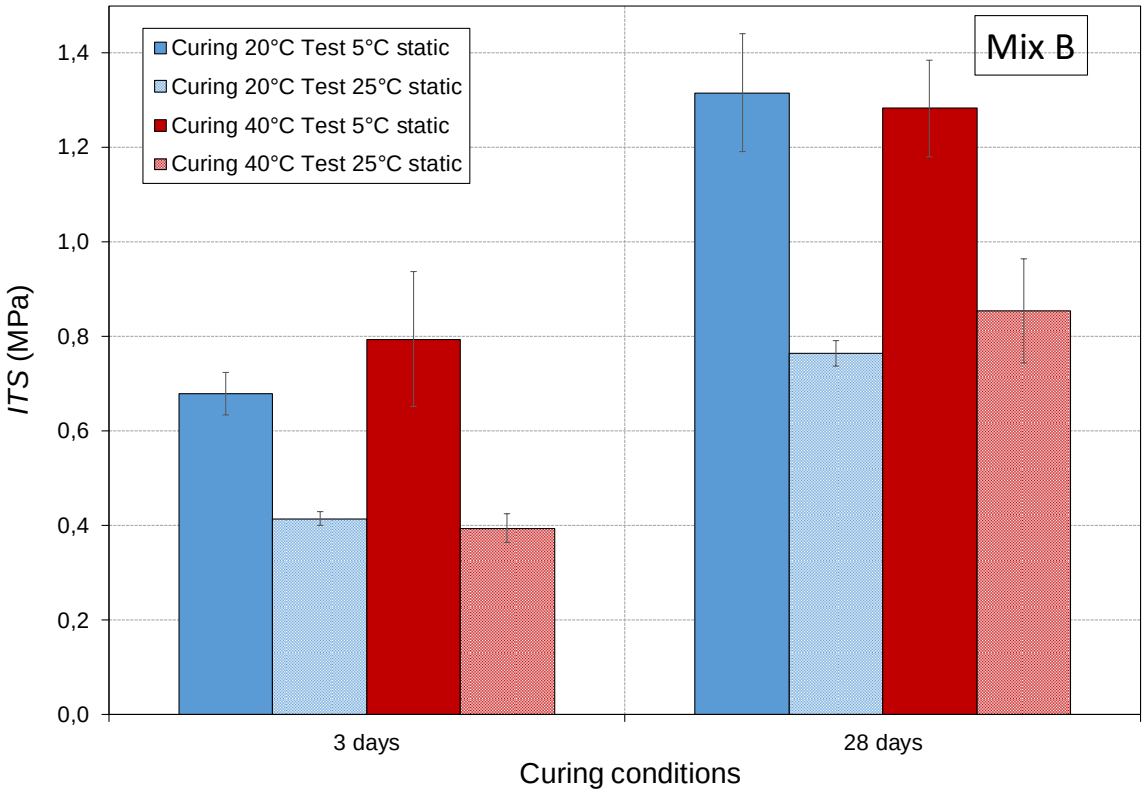


Figure 23. Indirect tensile strength test results obtained for mix B in Germany

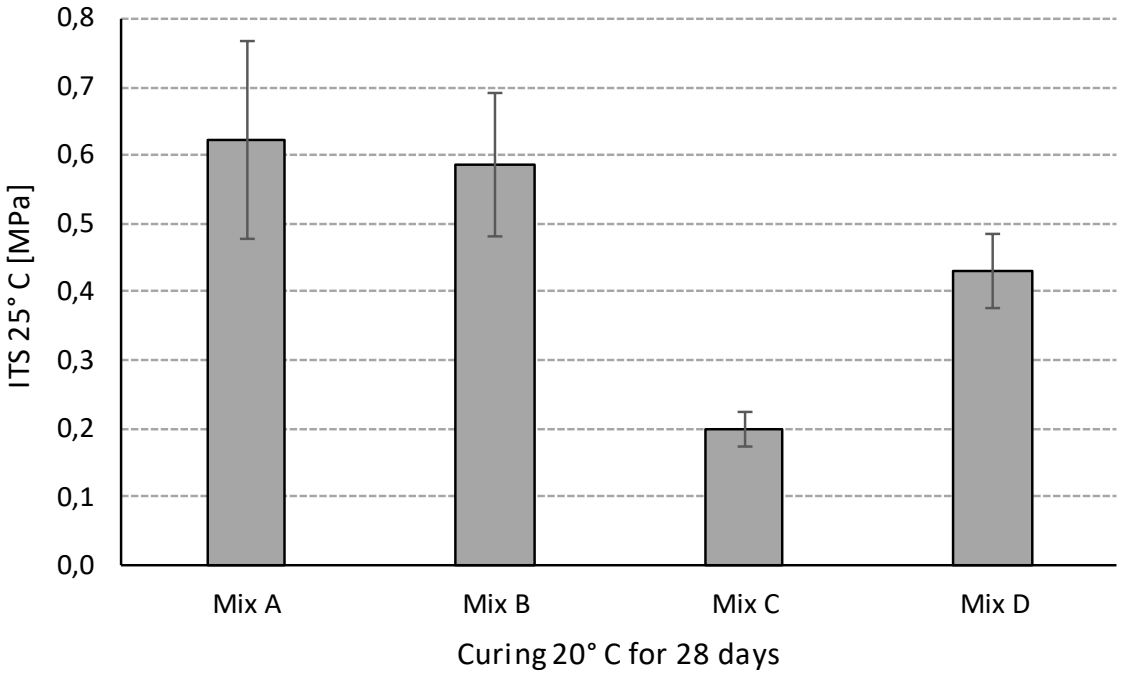


Figure 24. Indirect tensile strength test results obtained for all mixtures in Germany (vibratory compaction)

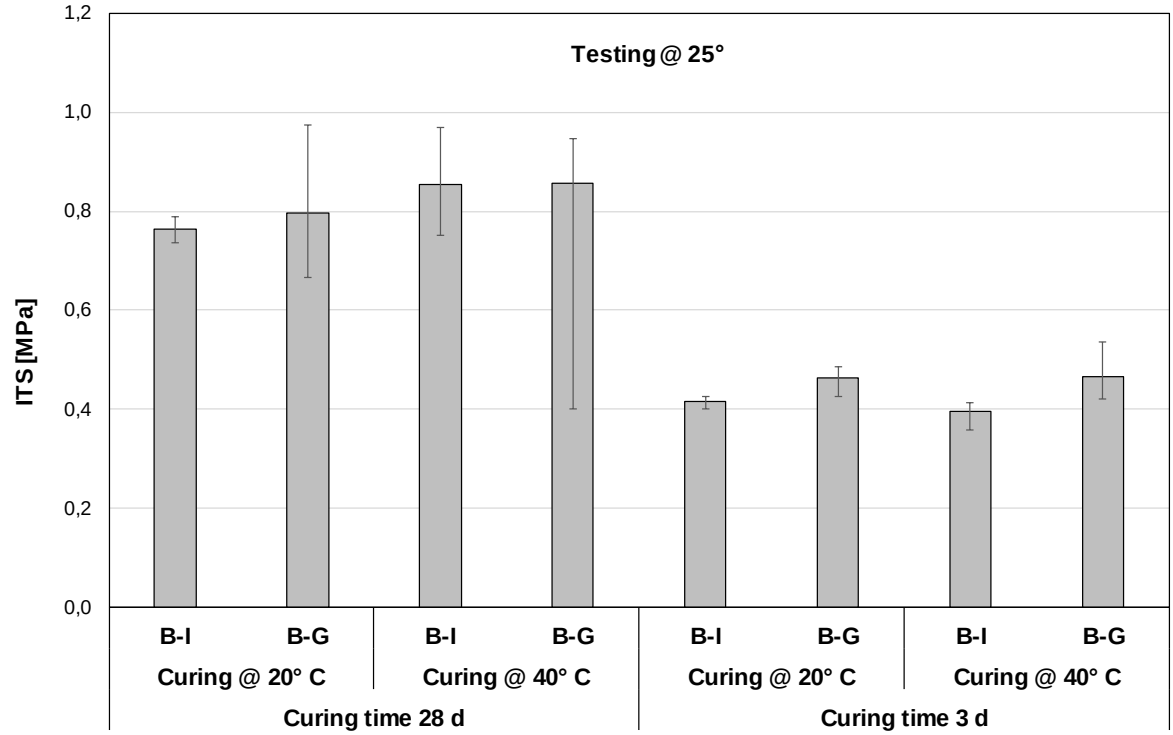


Figure 25. Indirect tensile strength test results obtained for mixture B in usage of Italian (B-I) and German (B-G) bitumen emulsion at test temperature of 25° C

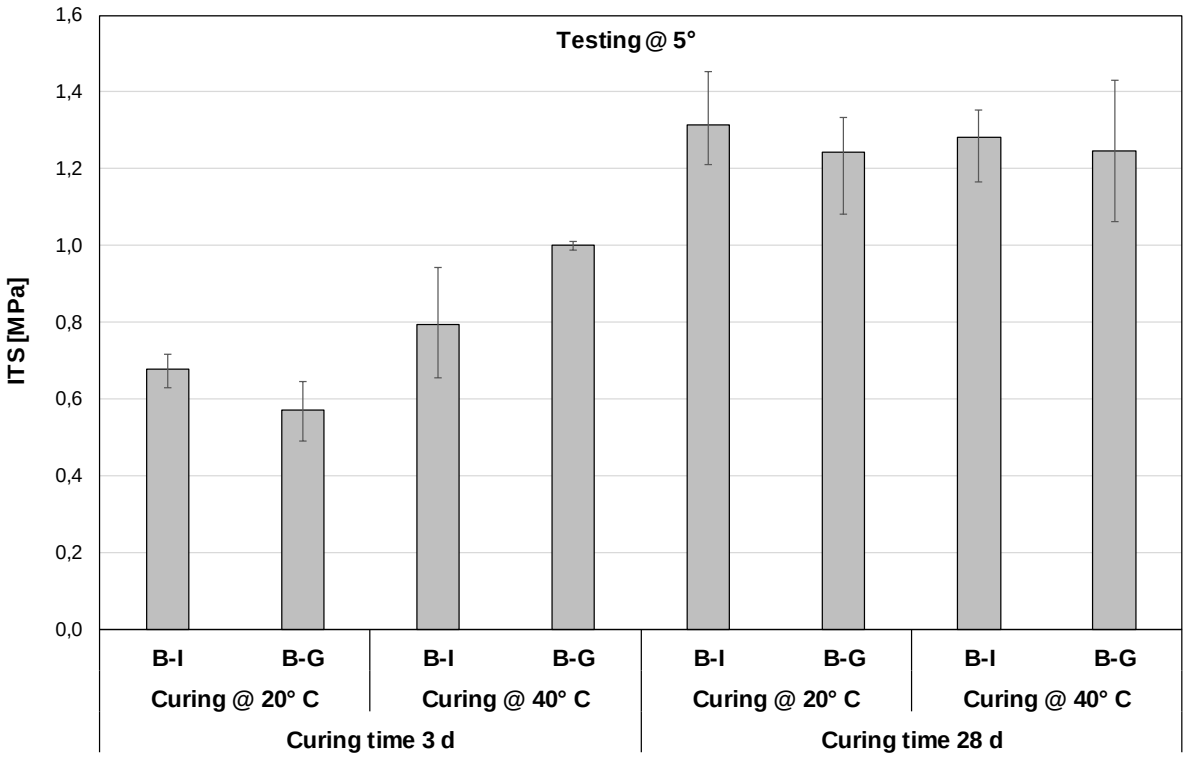


Figure 26. Indirect tensile strength test results obtained for mixture B in usage of Italian (B-I) and German (B-G) bitumen emulsion at test temperature of 5° C

5.4 Uniaxial compressive strength (Duriez)

Figure 27 reports the measured values of the dry compressive strength results as a function of curing conditions within the French laboratory. Specimens of mixture C had lower dry resistance than those of mixture D due to the lower cement dosage. The higher curing temperature and longer curing time led to higher strength and higher water loss.

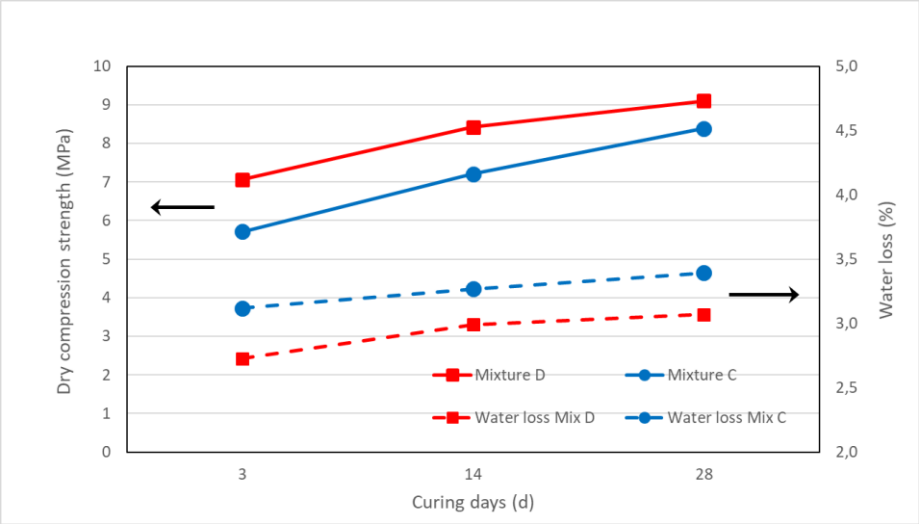


Figure 27. Dry compressive strength test results obtained for mixtures C and D in France at test temperature of 18° C

5.5 Shear strength

The shear strengths of all mixtures are given in Figure 28. Within Table 25 the function parameters of the linear shear functions as well as the shear strength are presented. All tests were performed at 20° C after 28 days of curing of the specimens.

The highest cohesion was obtained for mixture B with high cement dosage. Mixture C (no cement) has the lowest cohesion. The cohesion of mixture A and D is almost identical. The development of the friction angle shows an opposite dependence. Without any cement, mixture C has the highest value, whereas the friction angle for mixture B is the lowest. Even if the friction angle of mix A and D differs slightly, both mixtures shown similar behaviour regarding the shear strength.

Table 25. Compilation of function parameters and shear stength of each tested mixture

Mixture	Function parameter		Friction angle φ		Cohesion c
	A	B	rad	°	MPa
A	3,82	2,04	0,62	36	0,34
B	2,45	3,47	0,43	25	0,91
C	4,64	0,88	0,70	40	0,12
D	2,95	1,57	0,52	30	0,35

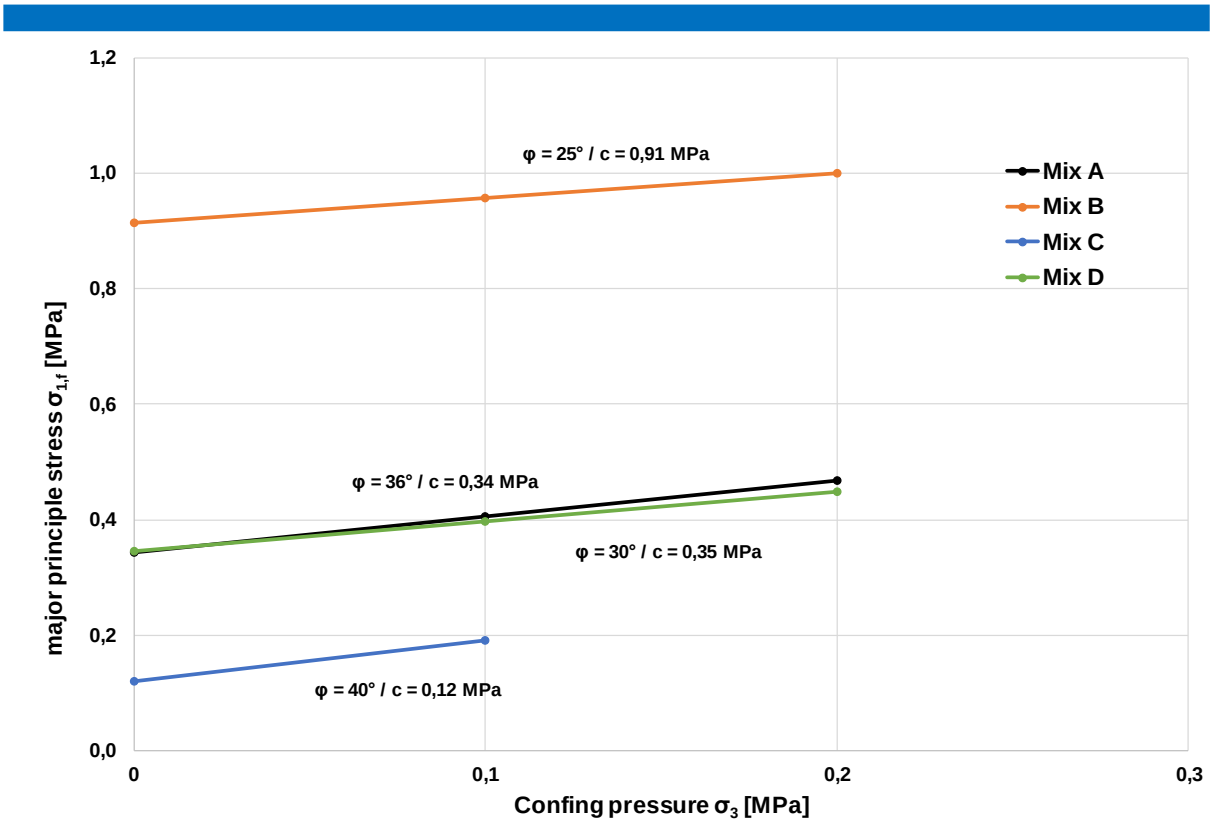


Figure 28. Shear strength test results obtained for all mixtures in Germany

5.6 Stiffness modulus by Cyclic indirect tensile stress tests

Within Table 26 and Table 27 the average of stiffness and strain values, measured at the tested temperatures and frequencys of all mixtures are listed. Testing of mixture C at 20 °C wasn't possible.

Because of a very low stiffness, it was not possible to reduce the stress to a level, at which the required strain would be lower than 100 µm/m.

Because of the high cement content within mixture B the specimens broke during the tests. For that reason, there is just single assignment of the test at 20° C. The stiffness values measured within the individual specimens are plotted versus the test frequency for the applied temperatures in Figure 29 to Figure 32. Except of mixture C, all tested materials are showing a high test scatter. In mixture A and D, one outlier with reducerd stiffness is vbisible, wherease the other two specimen show similar stiffness results. Still, the low stiffness of the third specimen is still considered within the mean value. As the test scatter seems to increase with cement content, it is assumed, that the hydration process during curing results in internal shrinkage cracks as well as brittle material properties, resulting in deteriorated sections within the specimen.

Within Figure 33 the stiffness-temperature-function calculated from the stiffness tests by application of time-temperatureequivalency is given. Table 28 identifies the parameters of each calculated stiffness-temperature-function. In general, each mixture shows a temperature dependency. But in consideration to the binder content significant differences within the function can be observed. Related to the cement content the temperature effect on mixture B is comparatively low. This goes along with a lower time-temperature-equivalency parameter ϕ. Whereas the function of mixture C shows a stronger effect of the temperature to the stiffness. The behaviour of mixture A and D seems to be almost similar. In comparison to mix A the temperature dependency is more distinctive for mix D at extrapolated temperature areas.

Table 26. Average of stiffness and strain for mixture A and B within CIT-CY

T [°C]	f [Hz]	Mixture A				Mixture B			
		|E*|[MPa]		Strain [‰]		|E*|[MPa]		Strain [‰]	
		Av.	Dev.	Av.	Dev.	Av.	Dev.	Av.	Dev.
20	10	5259	1345	0,063	0,019	7871	177	0,071	0,002
20	5	4637	1214	0,068	0,021	6839	75	0,078	0,001
20	3	4342	1187	0,063	0,021	6236	47	0,080	0,001
20	1	3771	1086	0,054	0,020	5445	31	0,082	0,000
20	0,3	3165	927	0,051	0,019	4626	6	0,088	0,000
20	0,1	2614	784	0,046	0,016	3968	29	0,093	0,001
10	10	6390	1097	0,056	0,011	9067	1108	0,073	0,009
10	5	5909	1024	0,055	0,011	8407	1204	0,076	0,011
10	3	5590	996	0,053	0,011	8020	1209	0,073	0,011
10	1	4958	898	0,052	0,011	7252	1190	0,077	0,013
10	0,3	4303	800	0,052	0,011	6504	1255	0,076	0,015
10	0,1	3704	675	0,051	0,011	5819	1242	0,079	0,017
0	10	7598	1496	0,051	0,012	9527	1374	0,075	0,011
0	5	7146	1368	0,050	0,011	9065	1406	0,076	0,012
0	3	6837	1300	0,047	0,010	8743	1406	0,076	0,012
0	1	6254	1223	0,047	0,011	8077	1414	0,076	0,013
0	0,3	5617	1102	0,046	0,010	7274	1516	0,079	0,016
0	0,1	5054	973	0,045	0,010	6670	1496	0,082	0,018
-10	10	8672	1343	0,054	0,009	10055	1615	0,077	0,012
-10	5	8459	1280	0,052	0,009	9664	1605	0,078	0,013
-10	3	8158	1264	0,051	0,009	9465	1576	0,074	0,012
-10	1	7700	1177	0,050	0,008	8949	1576	0,075	0,013
-10	0,3	7145	1110	0,049	0,008	8406	1515	0,074	0,013
-10	0,1	6563	964	0,049	0,008	7924	1514	0,075	0,014

Table 27. Average of stiffness and strain for mixture C and D within CIT-CY

T [°C]	f [Hz]	Mixture C				Mixture D			
		|E*|[MPa]		Strain [‰]		|E*|[MPa]		Strain [‰]	
		Av.	Dev.	Av.	Dev.	Av.	Dev.	Av.	Dev.
20	10	-	-	-	-	6086	465	0,051	0,004
20	5	-	-	-	-	5471	469	0,053	0,005
20	3	-	-	-	-	5101	425	0,052	0,004
20	1	-	-	-	-	4514	415	0,049	0,005
20	0,3	-	-	-	-	3921	348	0,047	0,004
20	0,1	-	-	-	-	3412	328	0,042	0,004
10	10	3834	109	0,046	0,001	6739	415	0,049	0,003
10	5	3244	85	0,045	0,001	6242	407	0,048	0,003
10	3	2886	106	0,045	0,002	5950	415	0,045	0,003
10	1	2296	47	0,041	0,001	5321	358	0,044	0,003
10	0,3	1752	73	0,034	0,002	4626	291	0,043	0,003
10	0,1	1436	254	0,020	0,005	3983	230	0,041	0,003
0	10	5667	268	0,046	0,002	7489	641	0,050	0,004
0	5	5084	216	0,045	0,002	7119	625	0,048	0,004
0	3	4653	164	0,046	0,002	6827	584	0,046	0,004
0	1	3950	180	0,047	0,002	6198	529	0,045	0,004
0	0,3	3176	202	0,043	0,003	5494	504	0,045	0,004
0	0,1	2464	246	0,043	0,004	4868	564	0,045	0,005
-10	10	7355	324	0,048	0,002	9373	730	0,046	0,004
-10	5	6966	261	0,047	0,002	9025	681	0,045	0,004
-10	3	6610	218	0,047	0,002	8664	632	0,043	0,003
-10	1	5934	194	0,048	0,002	8176	574	0,042	0,003
-10	0,3	5145	101	0,046	0,001	7544	498	0,042	0,003
-10	0,1	4332	151	0,048	0,002	6858	432	0,041	0,003

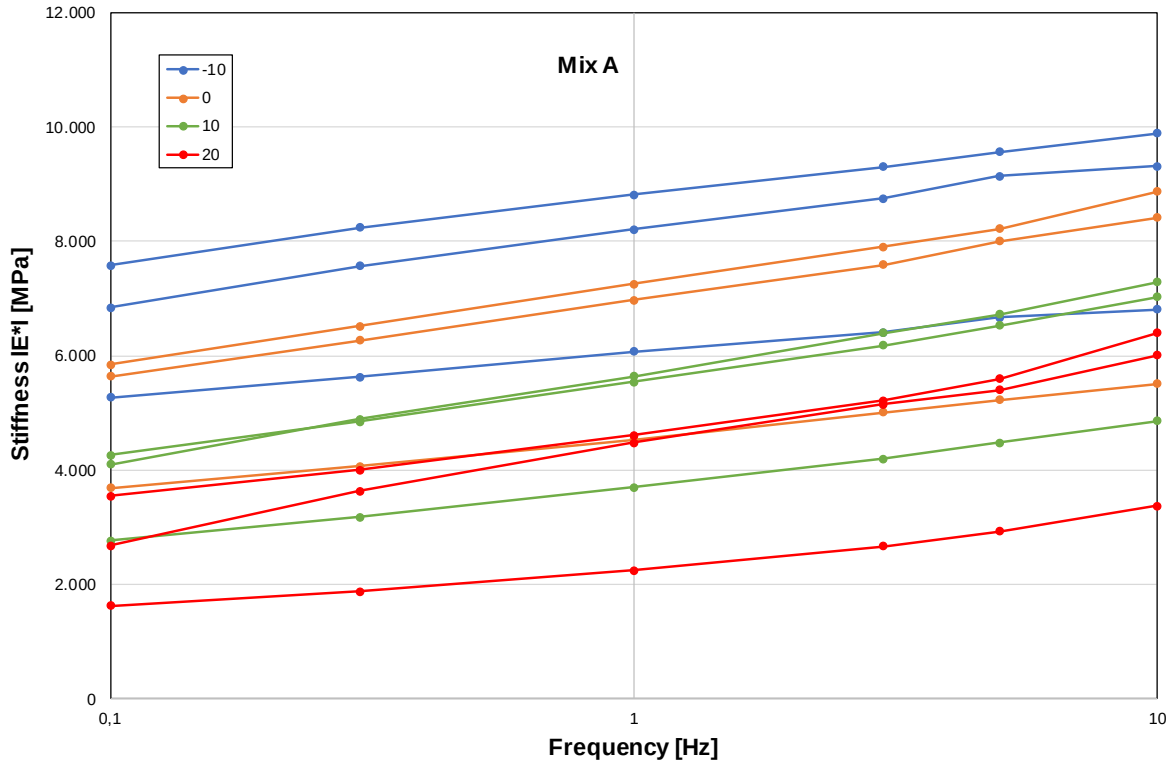


Figure 29. Stiffness of Mix A depending on frequency and temperature

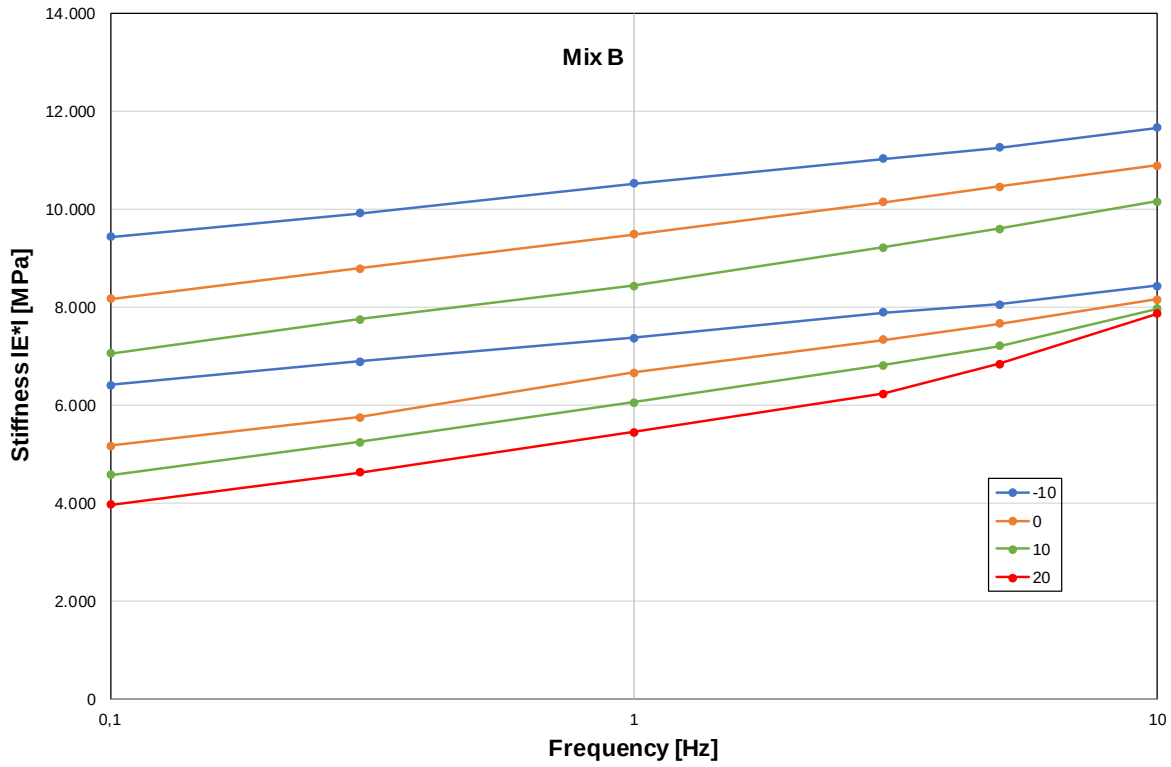


Figure 30. Stiffness of Mix B depending on frequency and temperature

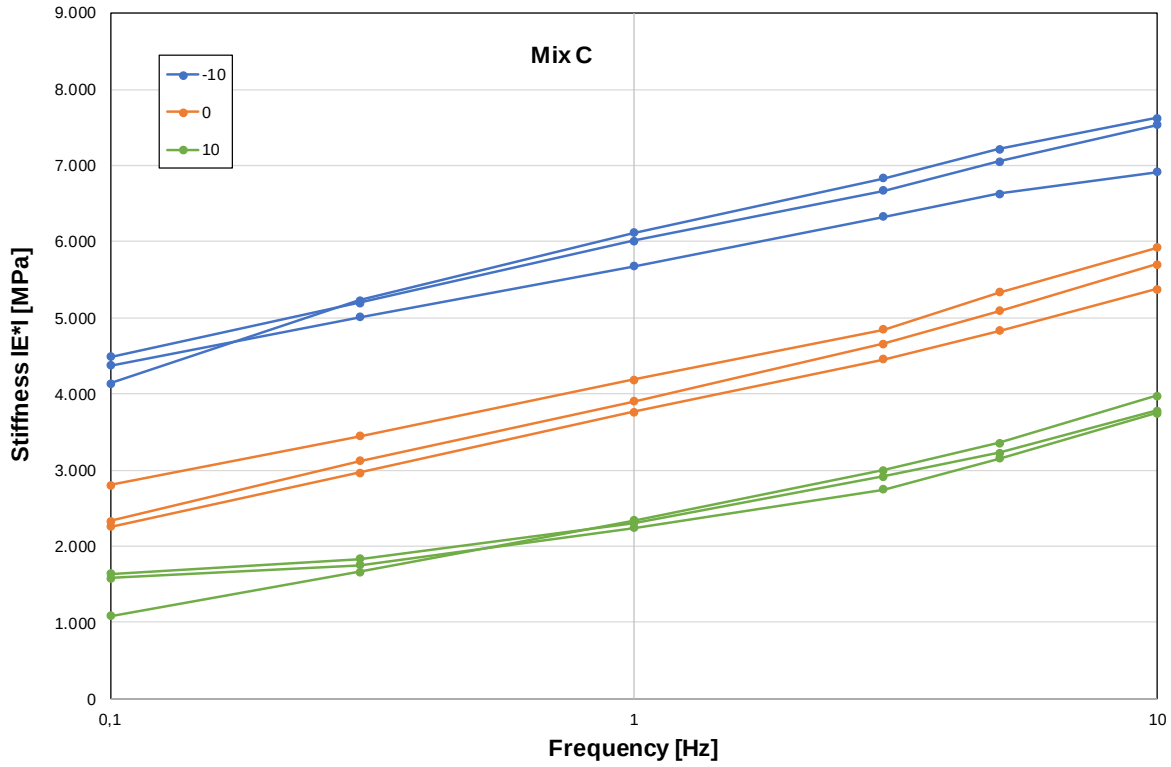


Figure 31. Stiffness of Mix C depending on frequency and temperature

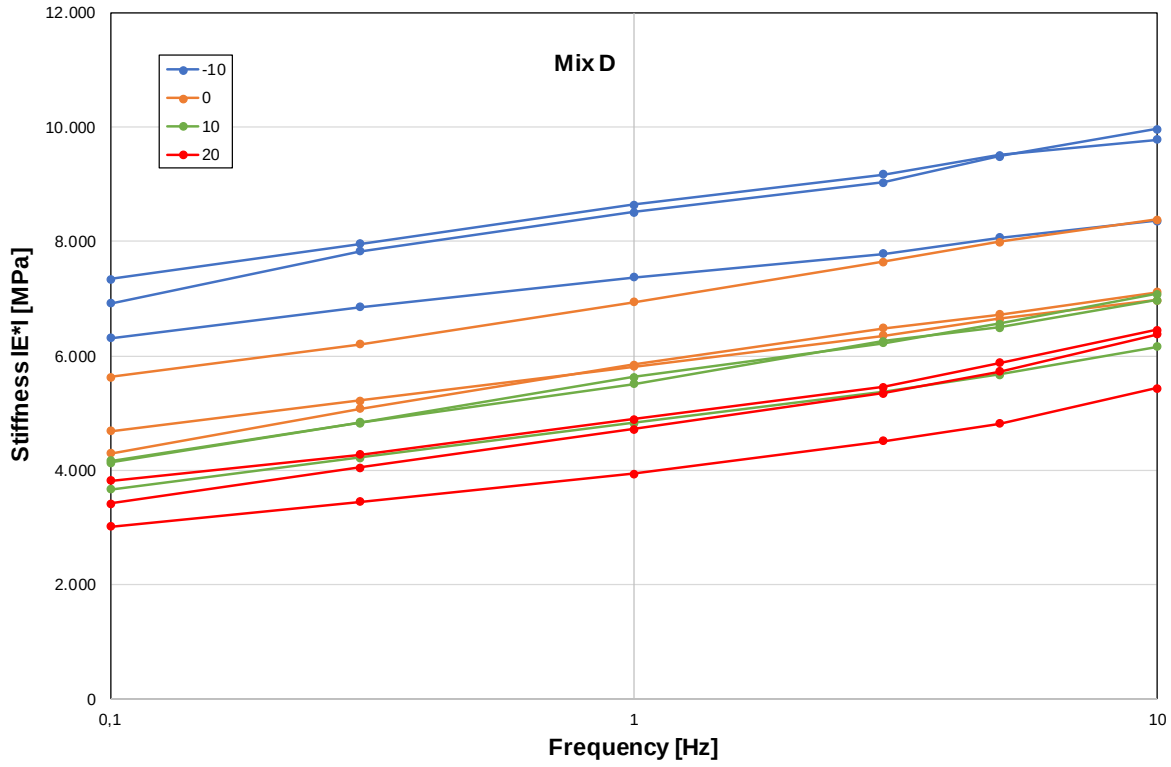


Figure 32. Stiffness of Mix D depending on frequency and temperature

Table 28. Parameters of stiffness-temperature-functions

Mixture	z_1	z_2	ϕ	$\lvert E^* \rvert_{+\infty}$	R^2
	[-]	[-]	[-]	[MPa]	[-]
A	0,6292	-0,4970	18.098	10.840	0,68
B	-0,1482	-0,5383	11.209	11.860	0,40
C	1,8698	-0,6822	20.259	9.680	0,98
D	1,2518	-0,3122	15.749	19.835	0,88

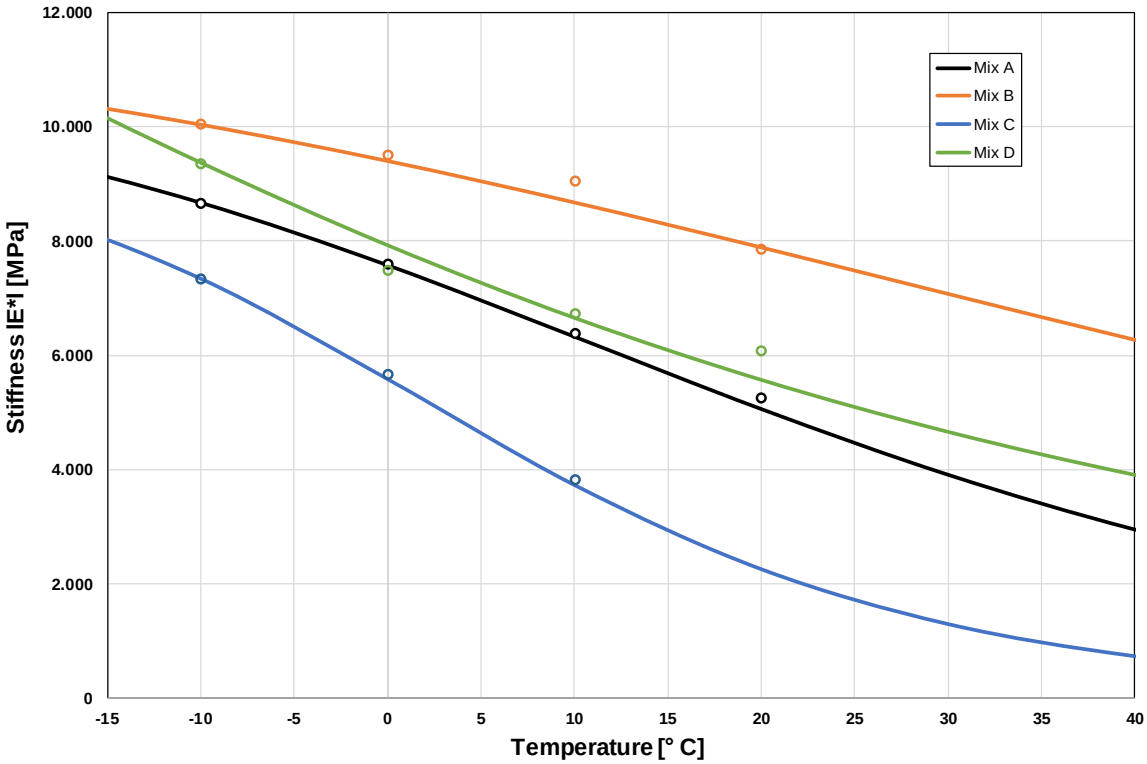


Figure 33. Stiffness-temperature-function calculated for Mix A to D

6 Discussion

In the following section, the results obtained within the interlaboratory experimental study are discussed. Therefore, the effect of the varied mix properties (binder contents), compaction and curing procedures are discussed seperately.

6.1 Effect of binder dosages

In Figure 34, the ITS obtained at the various mixtures after 3, 14 and 28 days of curing in different conditions are plotted versus the content of cement (left) and bitumen (right). According to A and D formulations with same cement content (1,5%), there is no influence of bitumen content on ITS measured at 25°C whatever the curing conditions from 20 to 40°C.

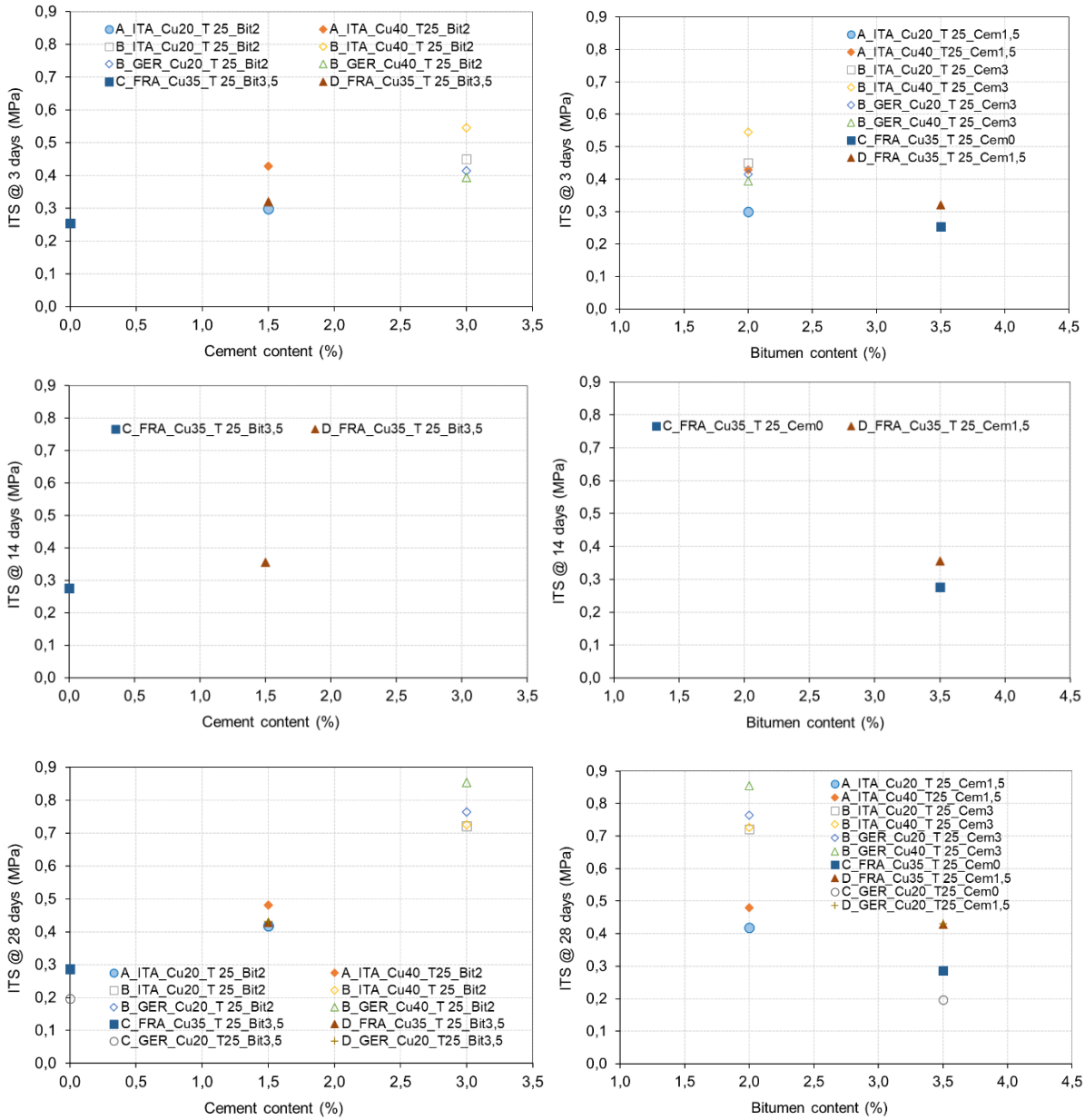


Figure 34. ITS tests results obtained at 25 °C on mixtures A to D, plotted versus cement content (left) and bitumen content (right).

However, when considering the results of the triaxial strength tests (Figure 28) as well as the stiffness modulus tests (Figure 33), an increase in bitumen content will result in lower shear strength but in a higher stiffness value.

Higher cement content (from 0 to 3%) increases the ITS measured at 25°C. After 3 days, ITA samples (A and B) cured at higher temperature (40 °C) increases ITS value. It seems different in the case of German samples with 3% cement (see B GER cured at 40°C vs 20°C). After 28 days, the increase in ITS value is stronger with 3% cement than with 1,5% (and 0%).

6.2 Effect of curing conditions

In this section, the effect of curing time and temperature are analysed. The influence of different curing RH is also evaluated.

6.2.1 Curing temperature

The effect of the curing temperature was analysed on mixtures:

- A – Gyratory-compacted (ITA)
- B – Gyratory-compacted (ITA) and static compacted (GER)

Both laboratories measured the ITS after 3 and 28 curing days at 20 °C and 40 °C. Test temperatures were 5 and 25 °C. The results are shown in Figure 35

In general, the curing temperature influences the evolution rate. It affects the kinetics of water evaporation, emulsion breaking and cement hydration, especially in the early curing stages. In the long-term, CRM mixtures tend to reach asymptotic properties regardless of curing temperature (Graziani *et al.* 2016, Du 2018, Raschia *et al.* 2019).

In Figure 35, the indirect tensile strength values obtained at specimens from mixture A and B after application of the two curing temperatures are plotted for the several curing times and test temperatzures. Figure 36 directly compares the average ITS values measured on CRM mixtures cured at 20 °C and 40 °C for the same time and tested under the same conditions. Each point is labelled using an alphanumeric code that indicates the mixture composition (A or B), the curing time (t3=3 days or t28=28 days) and the testing temperature (T5=5°C or T25=25°C). ITA mixtures showed a similar behaviour regardless of the binder dosage, curing time and testing temperature. Also, GER mixtures showed a similar trend. Therefore, the experimental data was fitted considering linear regressions:

$$y = ax + b \tag{16}$$

where a and b are fitting parameters obtained by applying the total least squares regression (or orthogonal linear regression). Table 29 lists the fitting parameters.

ITA and GER mixtures showed quite different behaviour. For ITA mixtures, decreasing the curing time or increasing the testing temperature led to greater detachment of the measured points from the equality line. This indicates that the effect of the higher curing temperature is more visible after 3 curing days because of the higher curing rate. Also, tests at 5°C allow a better understanding of the curing temperature influence as they highlight the contribution of the bitumen, which is thermo-dependent. The ITS of GER mixtures did not show a significant dependence from the curing temperature, and the linear regression is almost superposed to the equality line. Therefore, the effect of curing temperature is almost negligible for GER mixtures. This may be related to the curing RH. The first 48 sealed curing hours may favour the cement hydration as well as hindering the emulsion breaking. This may lead to the formation of a more cementitious structure and a reduced effect of the curing temperature.

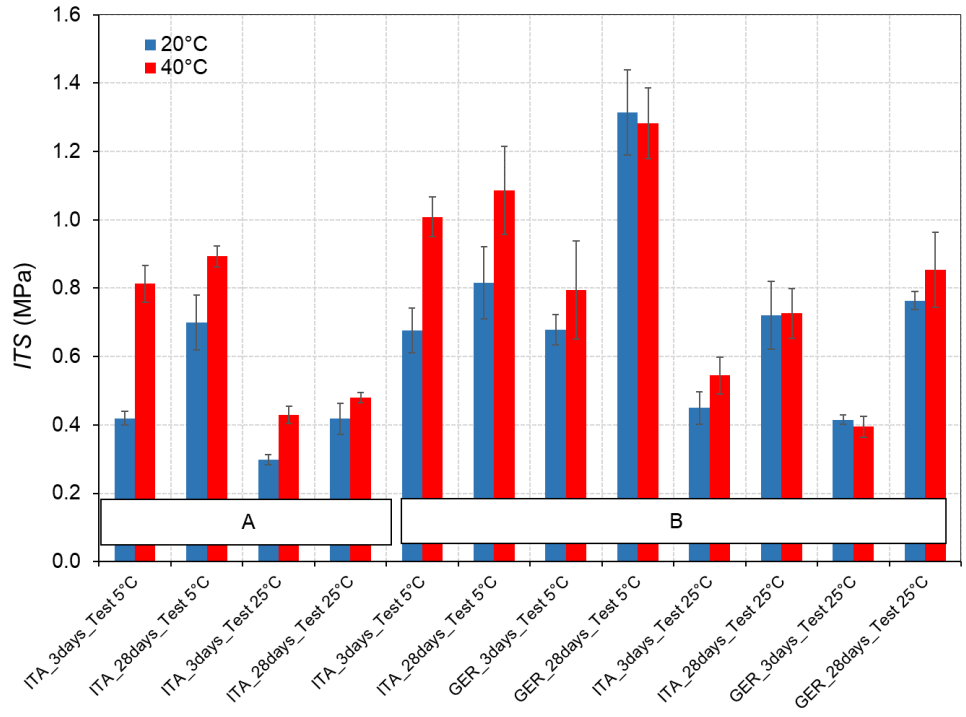


Figure 35. ITS tests results obtained on mixtures A and B changing the curing temperature.

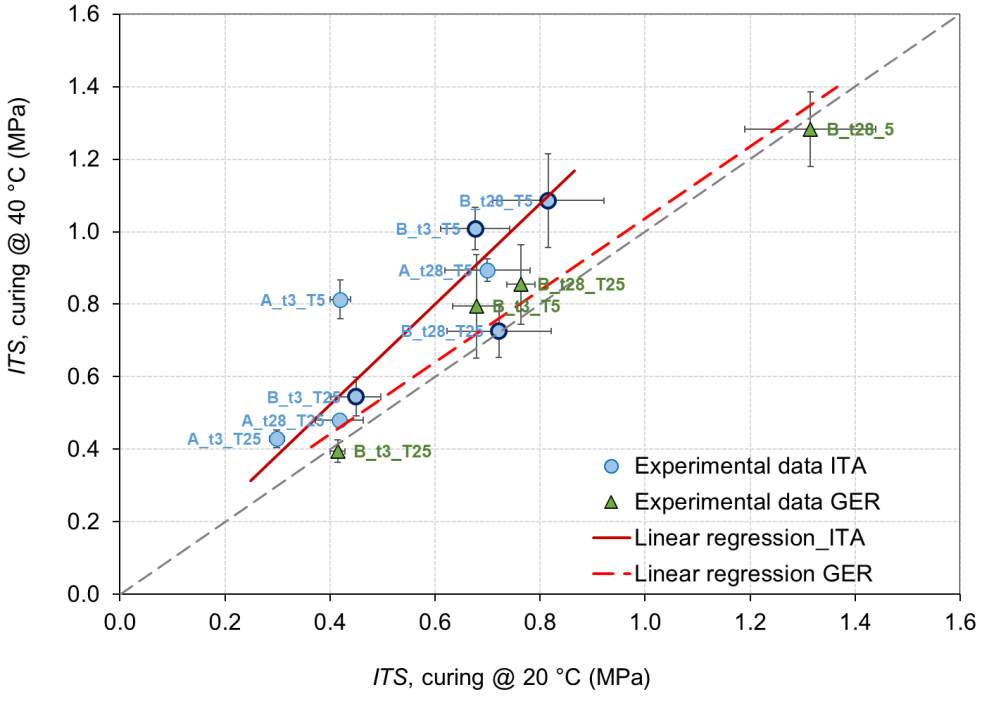


Figure 36. Effect of curing temperature

Table 29. Effect of curing temperature, linear regression fitting parameters (Equation 16)

CRM	a	b	R^2
ITA	1,38	-0,03	0,65
GER	0,99	0,04	0,96

6.2.2 Curing time

The effect of the curing time was analysed on mixtures: A – ITA, B – ITA (gyratory compaction) and GER ; C and D – FRA (static compaction)

All laboratories measured the ITS after 3 and 28 curing days. FRA laboratory measured the strength also after 14 days. Curing temperatures were 20°C and 40°C in ITA and GER, and 35°C in FRA. Test temperatures were 5 and 25 °C in ITA and GER and 25°C in FRA.

Figure 37 depicts the ITS values obtained at the different curing times. It can be observed that the increased curing time always led to an increase in the strength of CRM, as expected.

Figure 38 compares the average ITS values measured on CRM mixtures cured 3 and 28 days obtained considering the same curing and test temperatures. Each point is labelled using an alphanumeric code that indicates the mixture composition (A, B, C or D), the curing temperature (C20=20°C, C35=35°C or C40=40°C) and the testing temperature (T5=5°C or T25=25°C). The total least square linear regression (Equation 14) was applied for fitting the experimental data obtained in the various laboratories, i.e. ITA, GER and FRA. Table 30 lists the fitting parameters. ITA and FRA mixtures showed similar dependency with respect to time despite their different composition. Under the same testing conditions, the effect in the strength increase of the higher curing temperature is less visible due to the accelerated curing rate. Again, the two-days sealing conditions after compaction, which was applied in GER-specimen will result in a reduced breaking after one effective day of curing (3 days). Therefor, the time effect of curing is more visible than for the mixtures, which were unmoulded directly after compaction and cured in unsealed conditions (ITA, FRA).

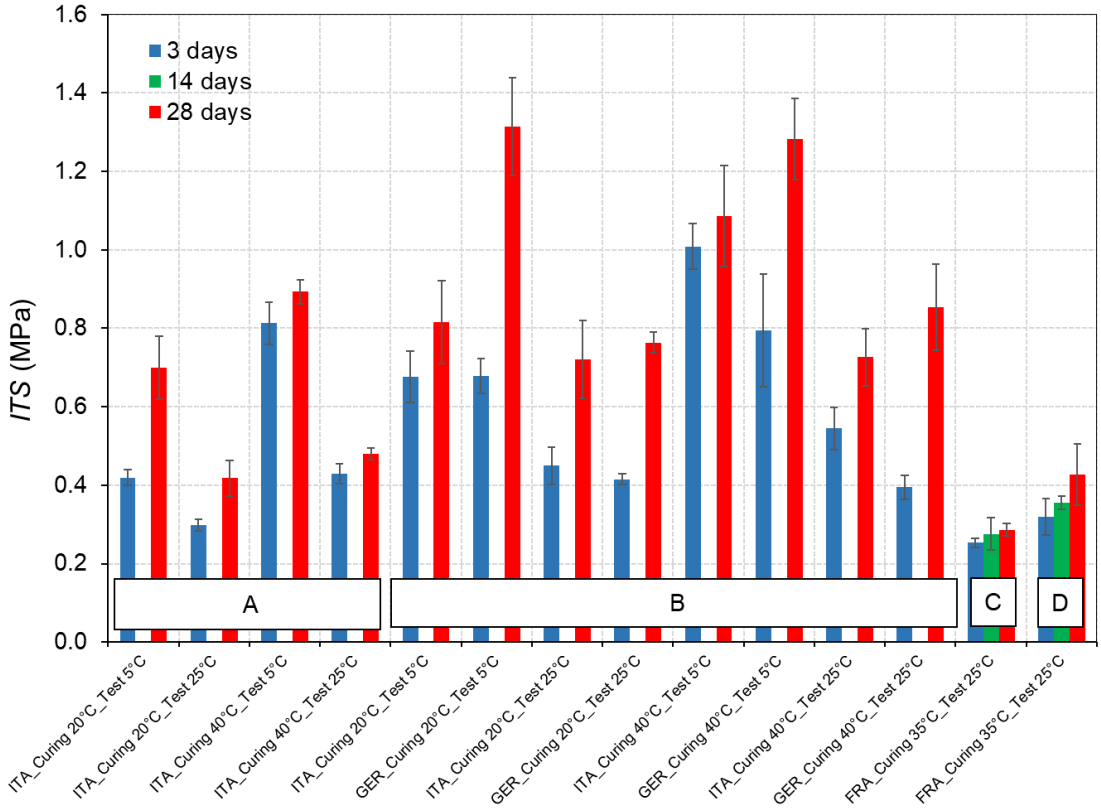


Figure 37. ITS tests results obtained at different curing times

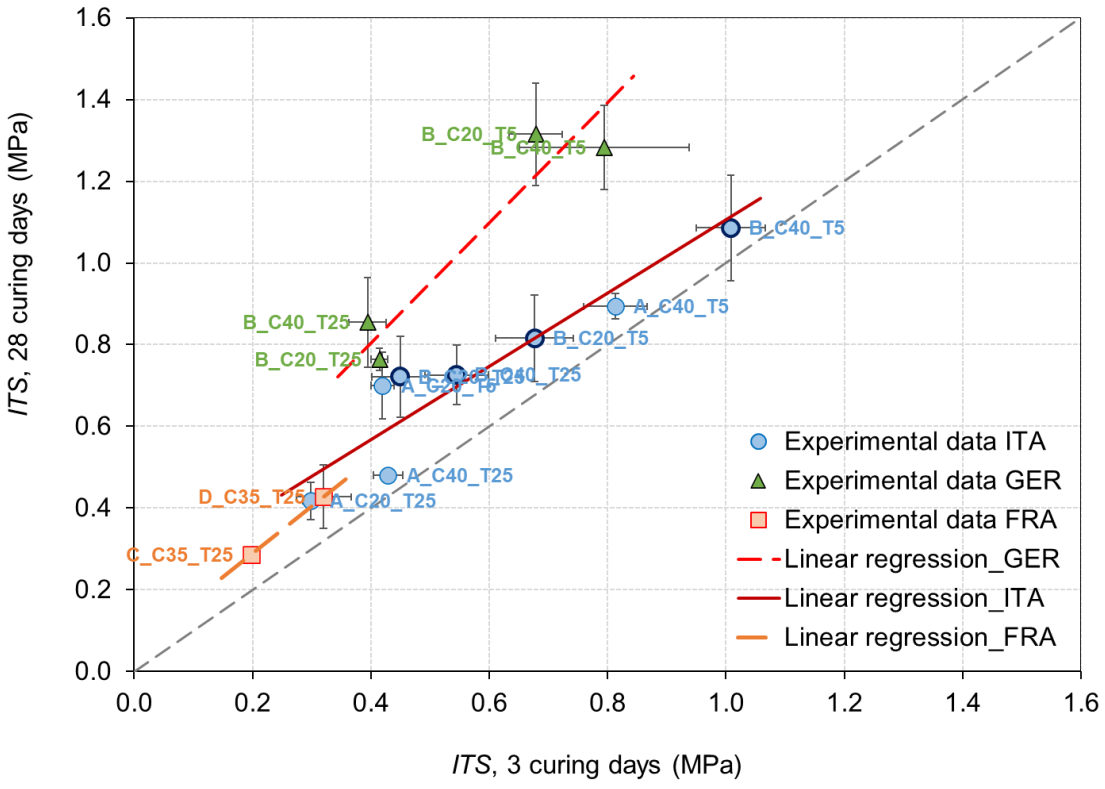


Figure 38. Effect of curing time

Table 30. Effect of curing time, linear regression fitting parameters (Equation 16)

CRM	a	b	R^2
ITA	0,90	0,21	0,86
GER	1,48	0,21	0,89
FRA	1,16	0,06	1,00

6.2.3 Recommendations about curing laboratory procedure

Curing temperature, time and relative humidity contribute to the development of the CRM microstructure and mechanical properties.

The specifications for the laboratory mix design of CRM mixtures should be related to the potential construction procedure, for example:

- The curing temperature shall be selected according to the average temperature of the construction period.
- The curing relative humidity shall be the same as the site. Therefore, the actual time of application of the prime/tack coat and superior courses, which translates into the sealed curing of the CRM, should be considered.

Usually, the actual conditions of the construction are not yet identified in the mix design phase. In this case, it would be recommended to carry out the laboratory tests considering a wide range of curing temperatures and RH conditions to provide a range of strength of the material which includes the actual properties of the material achievable in situ.

6.3 Effect of test temperature

Because of the temperature-dependent behaviour of bitumen, the indirect tensile strength of a hot mix asphalt specimen is higher at lower temperatures. Within cold recycled materials cement is an additional binder, which doesn't show this dependence. But cement have a distinctive time-dependent strength development. In Figure 39, the indirect tensile strength of mixture A as well as mixture B is plotted. Without any exception, the indirect tensile strength is higher for test temperature at 5° C. This suggests a bitumen dominance of both mixtures. Especially within mixture A with a higher ratio of bitumen to cement. Even if the cement content in mixture B is higher, the samples according to the German procedure show quite high temperature-dependent indirect tensile strengths. This is with high probability an indication of activities within the bituminous binder of the RAP.

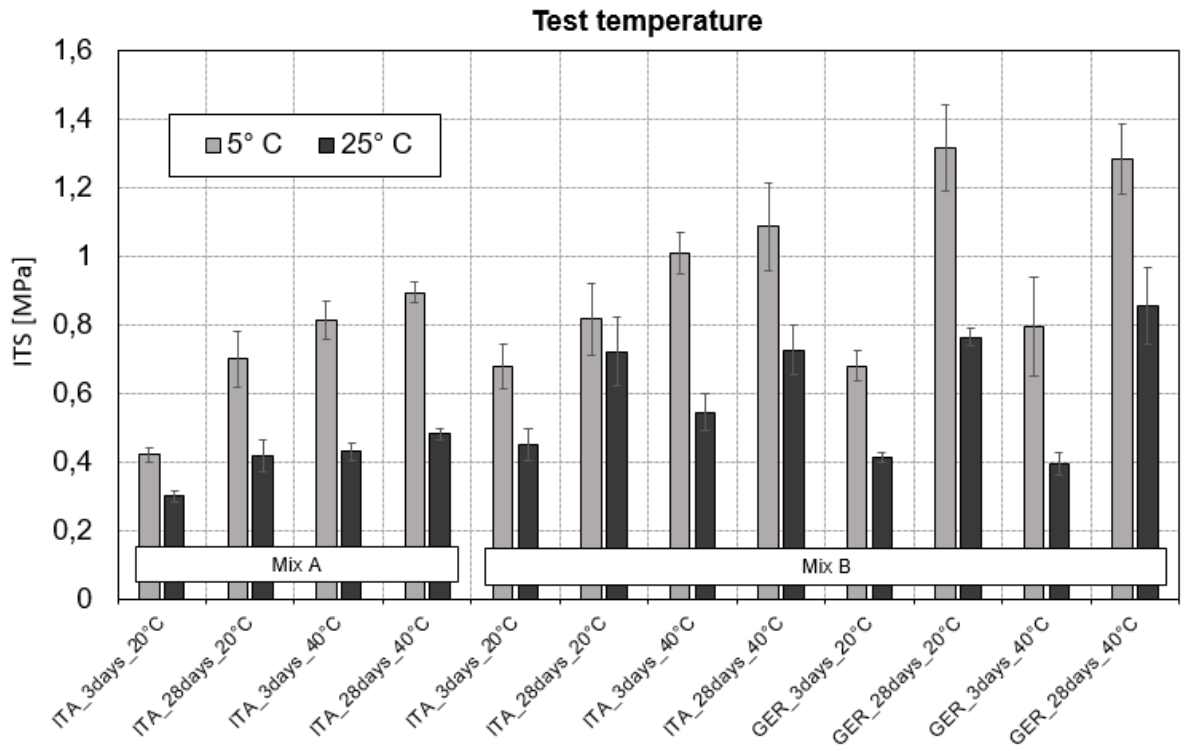


Figure 39. Indirect tensile strength test results obtained for Mix A and B

Within Figure 40 the ratio of indirect tensile strength at 5° C and 25° C is plotted. The ratio (ΔITS_{T25}) is calculated as follows:

$$\Delta ITS_{T25} = \frac{ITS_5 - ITS_{25}}{ITS_{25}} \cdot 100 \quad (18)$$

In General, a high ratio should also identify the effect of bitumen. Furthermore, decreasing of the ratio from 3 days to 28 days suggests a prior influence of the cement. Within mixture A of the Italian laboratory study, the ratio is increasing or almost similar for longer curing times. Especially the samples with a curing temperature of 20 degree suggest that the bitumen is more dominant. The ratio of mixture B indicates a significant decrease which is referred to the hydration of the higher amount of cement within this recipe. The curing temperature of 40 °C results in higher values of ΔITS_{T25} and therefore in an increased bituminous effect. This can be explained by a higher role of the RA binder for the overall strength properties of the specimen, which may be reactivated in a higher degree compared to lower curing temperature.

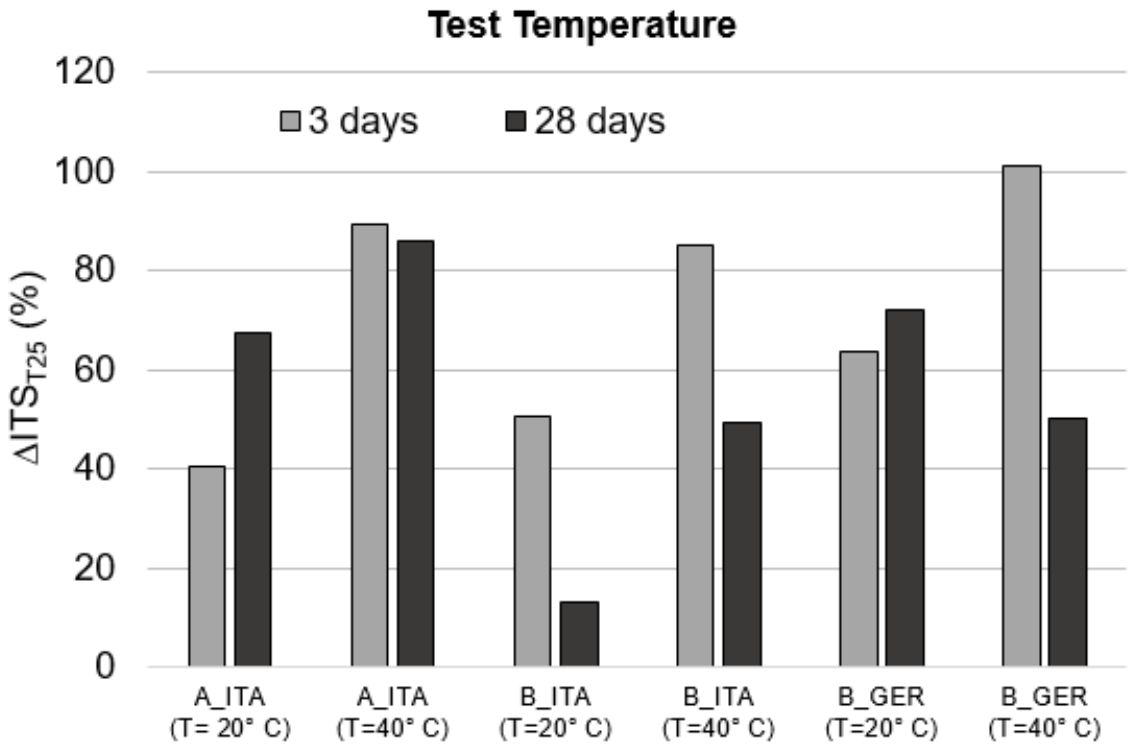


Figure 40. ITS-ratio (ΔITS_{T25}) of indirect tensile strength considering test temperature for Mix A and mix B

6.4 Comparison of mix design results with national specifications

The four CRM mixtures were designed in order to assess the whole range of practically applied bitumen emulsion and cement proportions. These mixtures were tested according to the nationally applied mix design specifications, which vary between compaction procedure, curing conditions, test procedure and test parameters. Besides the direct comparison between the results obtained, the study allows to indirectly compare the quality level applied in the national specification documents.

In Table 31, the required properties of the CRM bases according to the nationally applied specification documents in France, Germany and Italy are summarised. For these specifications, the prescribed compaction, curing and test methods were practically applied on the four selected mixtures. For these mixtures, it is indicated, if the mix properties comply with the individual specification limits.

The composition of the mix granulate fulfils the requirement on grading according to the German and Italian specifications. Regarding the applied binder contents, mixtures A and B doesn't comply with the minimum specified bitumen emulsion content according to French specifications, whereas mixture C doesn't comply with the minimum required cement content according to German standard.

Regarding the achieved void content after compaction, the specification limits applied in Germany are not reached.

Regarding the mechanical strength of the mixtures after curing, the three compared national specifications vary in terms of curing temperature, moisture condition and time, whereas the test conditions to assess the strength differ as well.

Mixtures A and B were prepared with a bitumen emulsion content of 3,3 % and were tested according the the German and Italian mix design specifications. Both mixtures comply with the

Italian minimum indirect tensile strength requirement of 0,35 MPa (tested at 25 °C). With an ITS of 0,43 MPa, the result obtained for mix A is near of the specification limit. This mixture doesn't comply with the minimum ITS (assessed at 5 °C) as specified in Germany, where a minimum strength of 0,7 MPa is specified. For mixture B, the ITS of 0,76 MPa meets the specification limits for in-plant and in-place produced CRM mixtures.

The minimum ITS-specification is about the double of the ITS-specification applied in Italy, which seems reasonable considering the applied test temperatures if 5 °C and 25 °C and the assessed ITS ratio values (compare Figure 39). The ITS values obtained according to German mix design specifications exceed the ITS results according to Italian procedures by factors of about 1,4 (both for mixture A (1,40) and B (1,38)). The difference in minimum ITS is higher than the difference between the actually tested mixtures. This can be an indication, that the mix design requirements applied in Germany are more conservative compared to the values applied in Italy. This may also explain the generally higher binder contents and especially the higher cement contents applied in Germany compared to Italy, as the specification requirements demands for a higher strength of the CRM material.

Mixtures C and D were tested according to the French and German mix design procedure. Here, the obtained ITS doesn't reach the specification limit according to German standard documents.

Table 31: Tests results on mixtures A to D in reference to the national mix design specifications applied in France, Germany and Italy

Country	Property	Specification	Mixtures			
			A	B	C	D
France[1)] Comp.: static (Duriez) Curing: 14 d at 35°C	bitumen emulsion content [%]	≥ 4	3,3	3,3	5,8	5,8
	void content [%]	≤ 15	-	-	8,2	7,6
	UCS (18 °C) [MPa]	≥ 2,5	-	-	7,7	8,4
	Water sensitivity	< 55 %	-	-	-	-
	Stiffness (ITSM, 10 °C) [MPa]	≥ 1.500	-	-	-	-
Germany[2),3)] Comp.: static, 50 kN Curing: 2d sealed, then unsealed 20 °C	< 0,063 mm [%]	4-9 (2-10)	6			
	< 2 mm [%]	24-39	26			
	bitumen emulsion content [%]	3-5 (2-6)	3,3	3,3	5,8	5,8
	cement content [%]	1,5-2,5 (3-6)	1,5	3	0	1,5
	void content [%]	5-15 (8-15)	20,6	15,9	21,7	21,5
	C: 7d: ITS (5 °C) [MPa]	0,6-0,8 (0,5-0,8)	-	-	-	-
	C: 28 d: ITS (5 °C) [MPa]	0,7-1,0 (0,75-1,2)	0,6	0,76	0,20	0,43
	C: 28 d: ITSR [%]	≥ 70 %	-	-	-	-
Italy[4)] Comp.: gyratory Curing: 3d at 40 °C	< 0,063 mm [%]	4-8	6			
	< 2 mm [%]	20-35	26			
	< 10 mm [%]	50-75	56			
	< 22,5 mm [%]	70-100	73			
	ITS (25 °C) [MPa]	≥ 0,35	0,43	0,55	-	-
	ITSR [%]	≥ 70 %	-	-	-	-
	stiffness, (ITSM, 10 °C) [MPa]	≥ 3.000	-	-	-	-

[1)] Guide IDRRIM [8]; [2)] M VB-K (FGSV 2005a) [2], [3)] M KRC (FGSV 2005b) [1], in brackets, [4)] Provincia Autonoma di Bolzano (2016) [9]

7 Conclusions and recommendations

The following conclusions can be drawn from the assessment discussed within this report:

1. Drying of mixtures is faster at higher curing temperatures and at lower cement dosages.
2. Concerning the mix formulation with a bitumen content of 3.3%, the addition of cement (3%) makes it possible to achieve higher strength performance than mixtures without cement or at lower dosage (1.5%).
3. For materials with moerate cement addition (here 1,5 %), the addition of a high bitumen content (5.8%) will reduce the indirect tensile strength.
4. Regarding the curing conditions in lab, it may be necessary to take into account the jobsite conditions and apply them in the laboratory (temperature and humidity). Regarding the curing period from 3 to 28 days, the results show, from a general point of view, an increase in mechanical performance will happened after 28 days while water content is not yet stabilized. Additional works are necessary to draw a finalized method.
5. The neglecting effect of another bitumen emulsion of the same type indicates, that the general mix design system as well as material performance is robust according to the individual bitumen emulsion product applied.
6. The compaction protocol and the mechanical tests carried out (geometry, stress, temperature) influence the mixtures performance. This indicates that a harmonization of practices at the European level is necessary to succeed in achieving Cold recycled materials mix design.
7. The nationally applied specification documents seem to specify the materials on a different quality level.

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