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The long-term properties of mineral-cement-emulsion mixtures

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highlights

- Nine different mineral-cement-emulsion mixtures were tested acc. to AASHTO TP79.
- The long term changes of dynamic moduli and phase angles were investigate.
- Mixtures were tested in periods of 28 days and 1.5 year.
- The mean change (increase) of dynamic moduli ranged from 9% to 14%.
- The mean change (decrease) of phase angles ranged from 4% to 8%.

abstract

This publication presents evaluation of long-term behavior of mineral-cement-emulsion (MCE) mixtures. MCE mixtures are among the major products of cold recycling of old asphalt pavements. They are com-posed by binding of the old materials reclaimed from the pavement and new mineral aggregate using two different binding agents – cement and bituminous emulsion. While bituminous emulsion dissolutes and binds materials quite fast, it does not increase the stiffness modulus of the whole mixture. Opposite behavior occurs for cement. Its effects appear slowly and all construction materials that contain cement present the increase of strength and stiffness modulus with time. Usually the increase of strength or mod-ulus is similar for all tested materials for the same curing periods.

This article investigates the impact of combination of two binding agents and their different amounts on the increase in strength and stiffness modulus of mineral-cement-emulsion mixtures with curing time. Conducted literature and laboratory studies showed that regarding the short term changes of mod-ulus and phase angle, mineral-cement-emulsion mixtures present similar behavior to other cement-bound materials, such as cement concrete or cement-bound mixtures. In the case of long-term behavior similarities to the cement-treated materials were found as well: an increase in moduli and a decrease in phase angles were observed for longer curing times. This kind of behavior illustrates that hydraulic bonds affect both mechanical and rheological long-term properties of mineral-cement-emulsion mixtures.

Keywords: Cold recycling of asphalt pavement, Mineral-cement-emulsion mixtures, Dynamic modulus, Phase angle, Long-term behavior

1. Introduction

Presently full-depth cold in-place recycling of old asphalt pavements using cement and bituminous emulsion is one of the most popular recycling technologies, as it is quite fast and relatively inexpensive, with strongly reduced usage of new materials [1–6]. A mineral-cement-emulsion mixture base layer provides very good mechanical performance, even when the base material obtained from old deteriorated asphalt pavements is of inferior quality.

Mineral-cement-emulsion mixtures include two different binding agents – cement and bituminous emulsion – that allow to obtain properties stated in the requirements. Each of the binding agents is responsible for meeting specific requirements [7–10]. Bituminous emulsion is responsible for creation of bituminous bonds, and – as a result of increase in cohesion of the layer – resistance to moisture-induced damage. It also provides sufficient flexibility of the layer, which should lead to minimization of the risk of shrinkage cracking. Cement is responsible for creation of hydraulic bonds, and, as a result, a further increase in resistance to moisture-induced damage. It also provides sufficient preliminary strength and bearing capacity of an MCE base layer for the purpose of construction traffic. As a side effect – in contrast to the bituminous emulsion – it is also responsible for an increased risk of shrinkage cracking. The overall behavior of an MCE base layer is the result of interaction between both binding agents. Special effort should be taken during MCE mixture design to obtain more bituminous

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bonds, as the risk of reflective cracking on the surface of the pavement is quite high, due to high amount of cement in the mixture. Mechanical properties stated in the requirements [11] should be achieved for the lowest possible cement content.

Indirect tensile strength is used as a basic parameter for evaluation and description of materials treated with cement and bituminous emulsion, due to simple specimen preparation, simple laboratory procedure and repeatability of results. Recently for mineral-cement-emulsion mixtures and other materials treated with either cement or bituminous emulsion more emphasis is being put on stiffness moduli, both short- and long term. The main purpose of stiffness moduli research is to obtain the optimum combination of binding agents that would result in the maximum bearing capacity of constructed layer with simultaneous reduction of possible extensive shrinkage. As most studies described in the literature review were conducted for shorter times of curing, it is hard to predict the final increase in stiffness modulus with curing time, as the addition of cement will be still responsible for creation of hydraulic bonds.

Literature review shows that in most requirements and regulations [7,8,11–15] mineral-cement-emulsion mixtures are evaluated similarly to the hydraulically treated materials. Typical requirements – especially in terms of strength – are given for periods of 7 and 28 days, as cement is one of the two main binding agents, even if its amount is strongly reduced. There is only a single case of requirements based on the elastic or even viscoelastic properties of the mix.

The development of strength of mineral-cement-emulsion mixtures with time of curing has been quite extensively investigated in recent years: especially the early-stage strength – up to 7 days [16], and short-term strength – up to 60 days of curing [17–23]. The case of long-term performance of mineral-cement-emulsion mixtures has not been thoroughly described and up to now only a single study has been conducted.

On the other hand, not much interest was given to the investigation of elastic modulus of mineral-cement-emulsion mixtures. Bocci et al. [17] investigated the influence of different curing conditions on the development of stiffness modulus. It was also found that curing in 40 °C strongly increases the development of stiffness modulus, probably due to increased hydration of cement. Meocci et al. [18] compared laboratory-prepared specimens with cores obtained from existing pavements. Stimilli et al. [24] investigated ITSM and complex modulus of both laboratory and field specimens. Kavussi & Modarres [20] investigated ITSM at three different temperatures (−10, 5 and 25 °C) for three curing periods (7, 28, 120 days). Valentin et al. [21] tested the stiffness modulus of both mineral-cement-emulsion mixtures and foamed bitumen mixtures. The test included influence of several factors on the value of stiffness modulus, e.g. proportions of the binding agents and curing time. Graziani et al. [22,23] tested the stiffness modulus of two different cold-recycled mixtures – mineral-emulsion mixture, with small addition of cement, and mineral-cement-emulsion mixture, for curing times of up to 120 days. Lin et al. [10] tested viscoelastic properties of mineral-cement-emulsion mixtures in the temperature range from −10 °C to 50 °C, but only for curing times of up to 28 days.

The assessment of change in the stiffness modulus of mineral-cement-emulsion mixtures is important for the pavement design process, as it influences the mechanical and fatigue behavior of the whole structure. An increase in stiffness modulus will result in an improvement of fatigue properties and bearing capacity of the pavement structure [25]. On the other hand, as the mixture contains cement as one of the binding agents, an increase in modulus can lead to shrinkage cracking of the mineral-cement-emulsion mixture base, and, consequently, to reflective cracking at the surface of the asphalt courses. The assessment of change in stiffness modulus and phase angle can indicate whether the impact of the increase in stiffness modulus of mineral-cement-emulsion mixtures is relevant to the comprehensive assessment of pavement fatigue life and failure mechanisms.

Fig. 1 presents comparison of the development of strength of mineral-cement-emulsion mixtures and hydraulically treated materials, such as hydraulically treated mixtures and concrete cement. Fig. 2 presents comparison of development of stiffness or elastic modulus over time for the aforementioned materials. To compare the processes, the values of strength and stiffness or elastic modulus were normalized. The period of 28 days of curing for each material was set as reference and the results for other curing periods were presented as a fraction or multiplication of the reference value. This method of analysis was chosen in order to compare the behavior of cement-treated mixtures in a very wide range of strength or modulus. Results for mineral-cement-emulsion mixtures were compared with those obtained for cement concrete [26]. A brief summary of the literature review is presented in Table 1.

2. Experimental program

2.1. Materials

The MCE mixture for research was designed according to the Polish requirements [11] and its mineral skeleton comprised of reclaimed asphalt, unbound crushed aggregate 0/31.5 and fine aggregate 0/2. CEM I 32.5R and cationic bituminous emulsion C60B5R were used as binding agents. The amounts of each binding agent used were set to 2, 4 and 6%, to create a matrix of 9 different combinations of binding agents in MCE mixtures (from 2% of cement and 2% of bituminous emulsion up to 6% of cement and 6% of bituminous emulsion). Grading curves of the designed MCE mixtures are presented in Fig. 3. Basic data regarding composition and mechanical properties of the selected mixtures is presented in Table 2. According to the requirements [11], samples for assessment of basic properties of MCE mixtures were compacted in a Marshall compactor with 75 blows per side. The amount of water added to the mixture was assumed on the basis of the following equation:

$$W_{dod} = W_{opt} - W_{nat} - W_{em} - 0.5 \times B \tag{1}$$

where: W_{dod} – the amount of added water [%], W_{opt} – optimum moisture content, determined from Proctor method [%], W_{nat} – natural moisture of MCE mixture (both reclaimed asphalt and additional aggregate) [%], W_{em} – water included in the bituminous emulsion [%], B – the amount of bitumen in the bituminous emulsion [%].

MCE mixtures were mixed using a standard laboratory mixer. First cement and water were added to the reclaimed asphalt and aggregate in the form of suspension with w/c ratio of 1. Next the bituminous emulsion was added and all components were mixed for 2 min at an ambient temperature of +25 °C.

Samples for the dynamic modulus test were compacted in a Superpave Gyratory Compactor to achieve 99% of the Marshall samples compaction. Vertical stress of 600 kPa, angle 1.25° and gyration rate of 30 gyrations per minute were used.

Indirect tensile strength (ITS) was tested according to the EN 12697-23 standard, indirect tensile stiffness modulus (ITSM) was tested according to the EN 12697-26 standard (IT-CY, method C). In the case of C6E2, C6E4 and C6E6 MCE mixtures, basic mechanical parameters were not assessed, as specified mixtures do not comply with the Polish requirements, and were not planned in the preliminary part of the research.

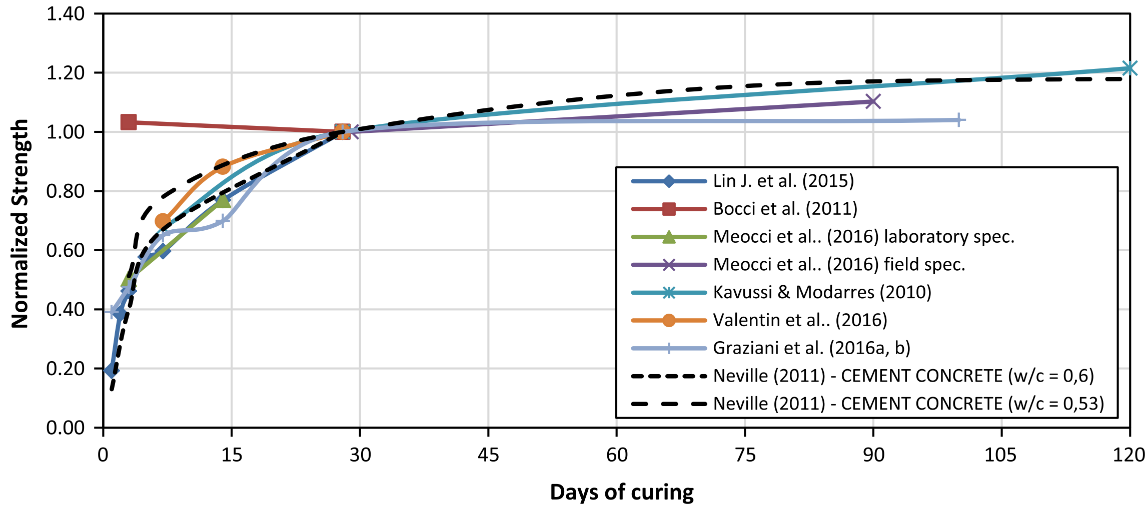


Fig. 1. Normalized strength on the basis of literature review.

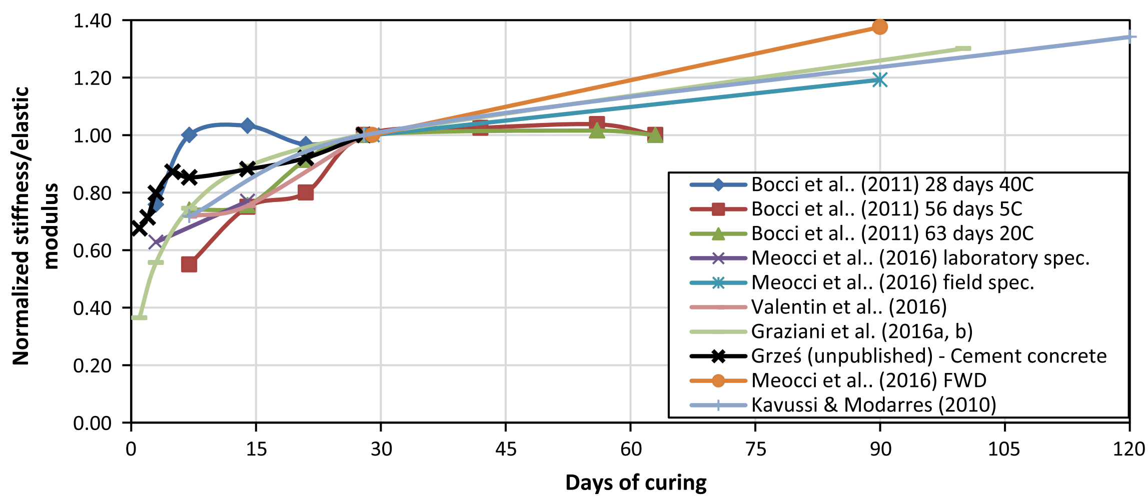


Fig. 2. Normalized stiffness or elastic modulus on the basis of literature review.

Cement concrete C8/10 and cement-treated aggregate C1.5/2 were used for comparison. Their basic parameters are presented in Table 3.

2.2. Modulus and phase angle test

The moduli and phase angles for all materials (mineral-cement-emulsion mixtures, cement concrete and cement-treated aggregate) were tested in the cyclic tensile test according to the AASHTO TP79 standard. The tests were conducted at three temperatures: 4 °C, 20 °C and 40 °C, for nine different frequencies – from 0.1 to 25 Hz. The test specimens compacted in the gyratory compactor were 100 mm in diameter and 150 mm in height. Specimens were tested after two periods of curing at 20 °C – 28 days and 1.5 year.

3. Test results and discussion

Test results for selected MCE mixtures (C4E4 for 28 days and 1.5 year) are presented in Table 4. Full data for all mixtures was presented in separate studies [27,28].

The appropriate master curve equation was chosen on the basis of literature review [29–35]. The equation used (2) assumed that shift factor was calculated using Arrhenius equation. In further analysis "psi" units were converted into "MPa" units.

$$\log|E*| = \delta + \frac{(Max - \delta)}{1 + e^{\beta+\gamma\left\{\log f + \frac{\Delta E_a}{19.14714}\left[\frac{1}{T} - \frac{1}{T_R}\right]\right\}}} \tag{2}$$

where: |E*| – dynamic modulus, psi (1 psi = 0.00689 MPa); Max – limiting maximum modulus, psi; f – frequency, Hz; T_R – reference temperature, K; T – test temperature, K; δ, β, γ – fitting parameters; ΔEa – activation energy (treated as a fitting parameter).

Examples of master curves developed using Eq. (2) for curing periods of 28 days and 1.5 year are presented in Fig. 4. Master curve parameters determined for all tested MCE mixtures are presented in Table 5.

Figs. 5 and 6 present master curves determined for tested mineral-cement-emulsion mixtures for the periods of curing equal to 28 days and 1.5 year. Obtained results were grouped to present the influence of selected binding agents on the values of dynamic modulus. Fig. 7 presents Black diagram (phase angle vs. modulus) of tested mineral-cement-emulsion mixtures and, for comparison – cement concrete and cement-treated aggregate as well. In the case of the MCE mixtures only one mixture is presented for better

Table 1
Summary of the literature review.

Author	Binding agents composition	Curing periods	Curing conditions	Test method of strength or modulus
Kavussi & Modarres [20]	Cement: 0.0–3.0% Emulsion: 4.0%	7, 28, 120 days	Temperature: 25 °C	ITS and M_R test according to ASTM D4123-04
Bocci et al. [17]	Cement: 2.0% Emulsion: 3.0%	3, 7, 14, 21, 28, 42, 49, 56, 63, 70 days[a]	Three different cases of curing conditions: (1) 28 days at 40 °C (2) 63 days at 20 °C (3) 56 days at 5 °C followed by 14 days at 40 °C	ITSM according to EN 12697-26
Stimilli et al. [24]	Cement: 2.0% Emulsion: 4.0%	1, 6, 18 months	Specimens cored out from existing pavement	Complex modulus according to AASHTO TP79-09 ITSM according to EN 12697-26
Graziani et al. [22]	Cement: 2.0% Emulsion: 3.0%	1, 3, 7, 28, 90 days	(1) Laboratory specimen: temperature of 20 ± 2 °C constant relative humidity of 50 ± 5%; with and without possibility of water evaporation (2) specimens cored out from existing pavement	ITS according to EN 12697-23 ITSM according to EN 12697-26
Graziani et al. [23]	Cement: 2.0% Emulsion: 3.0%	1, 3, 7, 14, 28, 100 days	Two different cases of curing conditions: (1) temperature of 25 ± 2 °C (2) temperature of 40 ± 2 °CIn both cases constant relative humidity of 70 ± 5%; possibility of water evaporation	ITS according to EN 12697-23 ITSM according to EN 12697-26
Meocci et al. [18]	Cement: 1.5% Emulsion: 3.5%	Laboratory: 72 h + 10 days Field: 1, 3 h, 1, 7, 14, 21, 30, 90 days	Laboratory: 72 h at 40 °C and 10 days at 20 °C Field: specimen cored out from existing trial section	ITS according to EN 12697-23 ITSM according to EN 12697-26 FWD according to ASTM 4694 LWD according to ASTM 2583
Valentin et al. [21]	Cement: 0.0–3.0% Emulsion: 2.5%, 3.5%[b]	7, 14, 28 days	Temperature of 20 ± 2 °C Relative humidity: 40–70%	ITS according to EN 12697-23 ITSM according to EN 12697-26
Lin et al. [10]	Cement: 2.0% Emulsion: 3.5%	7, 14, 21, 28 days	(1) temperature of 20 °C and relative humidity of 60% (2) temperature of 65 °C	Dynamic modulus according to AASHTO T342 and JTJ E20-2011

[a] Different test periods for different curing conditions.
[b] Paper also presents test results for mixtures with foamed bitumen and cement.

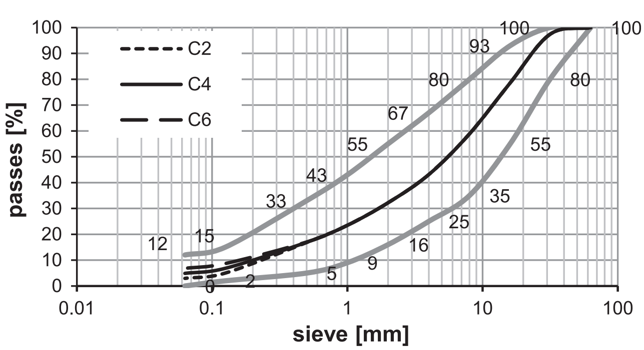


Fig. 3. Grading curves of tested MCE mixtures (C – the amount of cement, in%).

illustration of the change in mechanical properties of material over longer time of curing.

As can be seen in Figs. 5 and 6, dynamic moduli of tested MCE mixtures strongly depend on the temperature and frequency (time) of loading, as compared to other cement-bound materials, which showed almost constant values for different temperatures and frequencies of loading. For both curing periods, the difference between the highest and the lowest values measured in the laboratory test ranged from approx. 5500 to 9000 MPa. The difference was slightly higher for the longer curing period. Higher differences were visible for higher cement content. The amount of bituminous emulsion, for constant amount of cement, did not have strong influence on the range of the measured values of dynamic modu-

Table 2
Basic data of tested mineral-cement-emulsion mixtures.

MCE mixture designation	Mineral-cement-emulsion mixture (MCE)									Requirements according to Polish regulations [11]
	C2E2	C2E4	C2E6	C4E2	C4E4	C4E6	C6E2	C6E4	C6E6	
Cement content [%]	2	2	2	4	4	4	6	6	6	1–4
Emulsion content [%]	2	4	6	2	4	6	2	4	6	2–6
Air voids in Marshall samples (2 × 75 blows) [%]	14.3	17.1	16.7	13.9	15.8	15.8	not tested	not tested	not tested	8–15
ITSM at 5 °C, 7 days [MPa]	3491	4443	3454	7109	6289	3587	not tested	not tested	not tested	not required
ITSM at 5 °C, 28 days [MPa]	5867	4799	5985	8615	6056	6140	not tested	not tested	not tested	2000–7000
ITS at 5 °C, 7 days [MPa]	0.56	0.69	0.82	1.04	1.08	1.10	not tested	not tested	not tested	0.5–1.0
ITS at 5 °C, 28 days [MPa]	0.64	0.74	0.94	1.18	1.02	1.08	not tested	not tested	not tested	0.7–1.6
Air voids in Gyratory compacted samples (600 kPa, 1.25°, 30 gyr/min) [%]	15.2	18.0	17.5	14.7	16.6	16.7	14.2	15.3	14.9	not required

Table 3
Basic properties of cement concrete C8/10 and cement-treated aggregate C1.5/2.

Material	Cement concrete C8/10	Cement-treated aggregate C1.5/2
Gradation	0/16	0/5
Cement content [%]	6.5	5.2
Type of cement	CEM II/B-V 32.5R	CEM II/B-V 32.5R
Consistency	S1	not tested
W/C ratio	0.73	not tested
Air content [%]	2	not tested
Tensile strength, 7 days [MPa]	9.6	1.5
Tensile strength, 28 days [MPa]	16.9	2.0

lus. Similar behavior was visible in the case of phase angles. The influence of binding agents was more visible at higher temperatures. The difference between measured values for the frequencies of 10 Hz and 0.1 Hz was in the range from 30 to 70% in the case of 4 °C and from 100 to 300% in the case of 40 °C. Noticeable relationships between the amounts of binding agents and the dynamic moduli were visible for the constant content of bituminous emulsion and variable content of cement. In the case of 2% of bituminous emulsion, each 2% of additional cement content increased the average value of dynamic moduli by an increment of approx. 1500 MPa. The average value of the mentioned increment was lower for higher amounts of bituminous emulsion and equalled 1000 and 500 MPa for 4 and 6% of bituminous emulsion respectively. In the case of constant amount of cement and variable amount of bituminous emulsion no visible trends were observed.

All tested materials presented two major trends with the increase in curing time: a decrease in phase angle as well as an increase in modulus. Exemplary average values of both phase angles and moduli for MCE mixtures, cement concrete and cement-treated aggregate are presented in Table 6 (frequency 10 Hz, temperature of 20 °C).

Usually the increase in the values of dynamic moduli ranged from 2% to 35% (mean value from 9 to 14% dependent on the binding agents combination; up to 1000 MPa in the measured values). The change of values is greater for the highest tested temperature (40 °C), in which mostly cement influences the behavior of the MCE mixture. The range of increase is similar to other cement-bound materials and cement concrete (see Fig. 2). In the case of stiffness moduli single discrepancies were visible at the temperature of 4 °C. Three of the tested MCE mixtures (C2E2, C2E6, C4E6) presented a decrease in dynamic moduli for the highest tested frequencies (10–25 Hz). The decrease of the value was in the range from 1% to 6% (up to 500 MPa in the measured values). Unfortunately, despite the increase in dynamic moduli observed in most cases, no straight correlations or trends were visible between the composition of binding agents and the increase in the value of dynamic moduli, probably due to complex structure of tested materials and possible inhomogeneities of base materials used, especially in the case of reclaimed asphalt.

The change of phase angles was more complex than the change of dynamic moduli. For most cases at temperatures of 4 °C and 20 °C the value of phase angle decreased by 1% to 15% (mean value from 4% to 8%, dependent on the combination of binding agents; up to 2° in terms of value of phase angle). The only discrepancy was observed for the mixture C2E2 where at the temperature of 4 °C phase angle was 2% higher for highest frequencies. In the case of 40 °C the observed behavior was more complex. While for higher values of test frequencies all tested MCE mixtures presented a decrease in phase angle (in the range from 1% to 11%), at lower values of test frequency (from 0.01 Hz to 1 Hz), most of the tested mixtures presented an increase in phase angle (in the range from 1% to 9%). The cause of this phenomenon is unknown to the authors. Similarly to the values of stiffness moduli, no visible correlations between decrease of phase angle and combination of binding agents were found.

Preliminary trials were performed to evaluate whether the time of loading influences the change of dynamic moduli and phase angles. For this purpose the changes of their values are presented as the interquartile range (middle 50% of results) along with minimum and maximum values separately plotted for each tested frequency (in Figs. 8 and 9).

Similarly to single values, in the case of the interquartile values, the changes of dynamic moduli for all three contents of cement and almost all frequencies range from 3% to 22%, and mean values range from 9% to 14%. In the case of phase angles, the interquartile values of the change for cement content of 2 and 4%, across all frequencies, range from 1% to 12%, and mean values range from 4% to 8%. Some discrepancies are visible for cement content of 6%: at the temperature of 40 °C and frequencies lower than 2 Hz the values increased over the longer curing period. The results obtained for the temperatures of 4 and 20 °C are similar to the results for the cement content of 2% and 4% – the mean value of the change (decrease) ranges from 2% to 13%. The resulting values are very similar to those obtained for cement-treated materials, both tested in laboratory and described in the literature.

4. Statistical analysis

Values of mechanical properties obtained from the conducted laboratory tests showed scatter of results for different specimens. It was necessary to determine whether the differences in laboratory test measurements between two curing periods were the result of the curing process or rather a natural scatter of results, probable especially when base materials such as reclaimed asphalt are considered. To confirm whether the differences between test results obtained for both curing time periods are significant, statistical analysis using Student *t*-test was performed. To illustrate the process, the data only for 10 Hz and 20 °C was considered. The test was conducted for both measured values: dynamic modulus and phase angle. It was assumed that the measurements can be described using normal distribution. Results for each MCE mixture were described using mean value and standard deviation. Null

Table 4
Stiffness moduli and phase angles for selected mineral-cement-emulsion mixtures.

Mixture Designation (curing period)	Temp. [°C]	Stiffness modulus [MPa] Frequency [Hz]				Phase angle [°] Frequency [Hz]			
		10	1	0.1	0.01	10	1	0.1	0.01
C4E4 (28 days)	4	6 993	5 740	4 513	–	7.54	9.39	11.80	–
	20	4 224	2 946	1 940	–	13.40	16.26	18.20	–
	40	1 580	918	581	423	20.57	19.70	16.71	12.14
C4E4 (1.5 year)	4	7 200	5 994	4 815	–	6.76	8.37	10.49	–
	20	4 352	3 072	2 034	–	12.59	15.43	17.79	–
	40	1 895	1 127	699	506	19.15	19.38	17.25	14.67

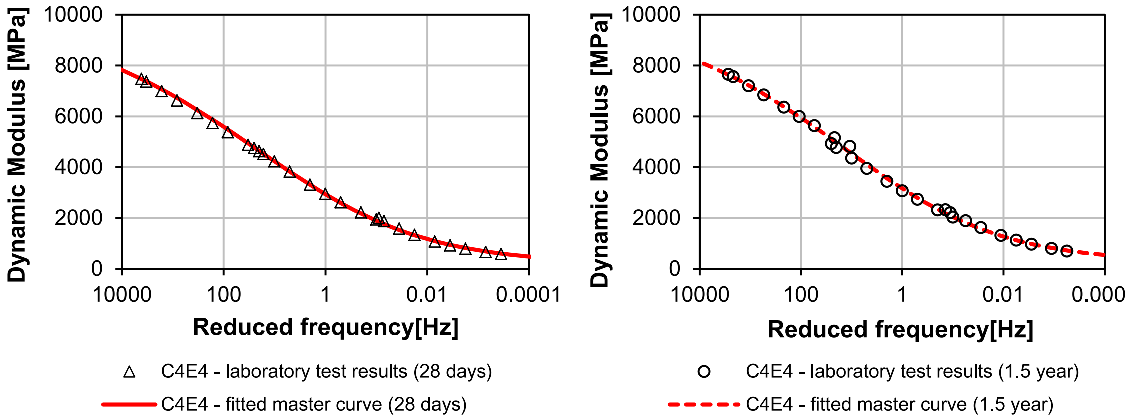
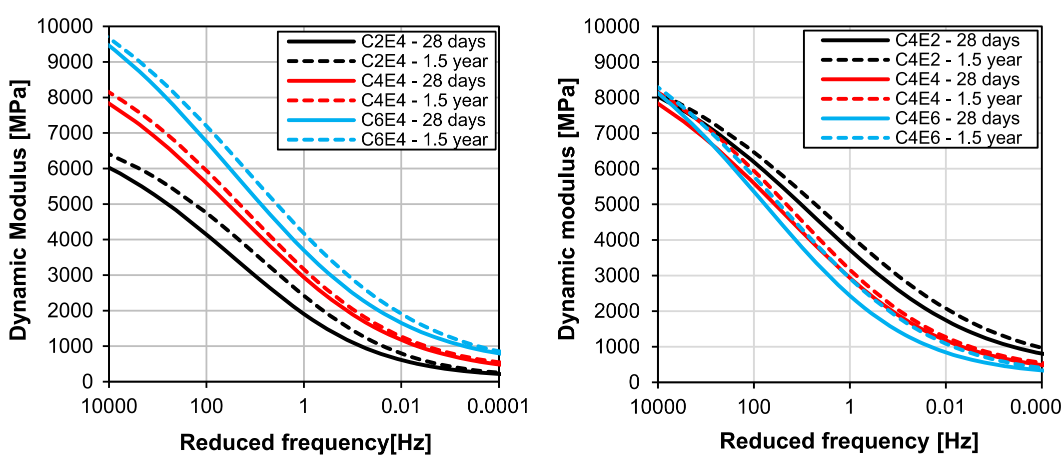


Fig. 4. Master curves for selected MCE mixtures developed on the basis of laboratory results (C4E4 for 28 days and 1.5 year of curing), T_{ref} = 20 °C.

Table 5
Master curve parameters for all tested mixtures for curing times of 28 days and 1.5 year, T_{ref} = 20 °C.

MCE mixture designation	Curing period	Master curve parameters				
		Max	δ	β	γ	ΔEa
C2E2	28 days	6.067	4.119	−0.897	−0.434	210 133
	1.5 year	6.067	3.710	−1.229	−0.371	206 067
C2E4	28 days	6.045	4.042	−0.842	−0.518	200 542
	1.5 year	6.045	4.010	−1.124	−0.529	210 703
C2E6	28 days	6.169	3.854	−0.699	−0.498	209 148
	1.5 year	6.169	3.198	−1.195	−0.426	210 532
C4E2	28 days	6.150	4.628	−0.973	−0.469	214 246
	1.5 year	6.150	4.636	−1.122	−0.448	223 899
C4E4	28 days	6.165	4.374	−0.852	−0.469	215 422
	1.5 year	6.165	4.495	−0.840	−0.499	197 837
C4E6	28 days	6.201	4.256	−0.682	−0.490	196 470
	1.5 year	6.201	4.181	−0.925	−0.457	205 769
C6E2	28 days	6.227	4.305	−1.504	−0.378	253 258
	1.5 year	6.227	5.037	−0.968	−0.501	229 314
C6E4	28 days	6.255	4.672	−0.699	−0.456	205 958
	1.5 year	6.255	4.572	−0.939	−0.436	214 835
C6E6	28 days	6.197	4.438	−0.841	−0.471	215 328
	1.5 year	6.197	4.429	−0.997	−0.501	204 957



(a) (b)

Fig. 5. Master curves (T_{ref} = 20 °C) of tested MCE mixtures for the curing periods of 28 days and 1.5 year: a) influence of the content of cement, b) influence of the content of bituminous emulsion (C – the amount of cement, in%; E – the amount of emulsion, in%).

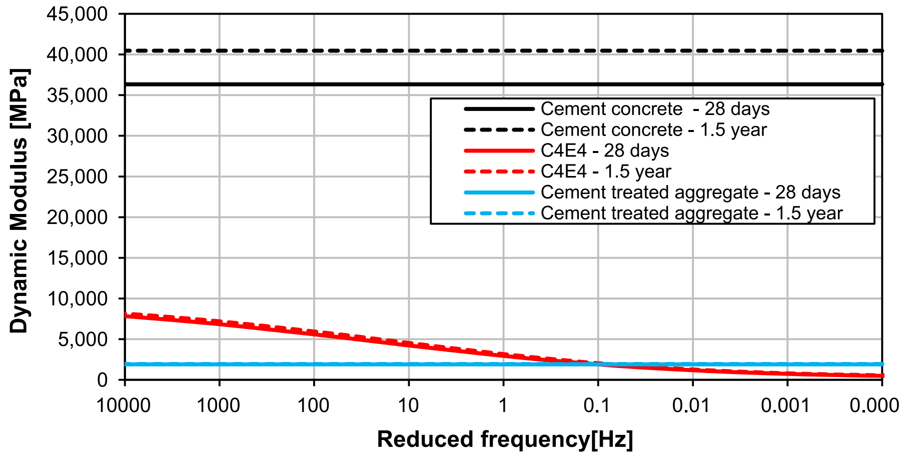


Fig. 6. Master curves of tested MCE mixtures for the curing periods of 28 days and 1.5 year (C – the amount of cement, in%; E – the amount of emulsion, in%) in comparison to other materials containing cement, T_{ref} = 20 °C.

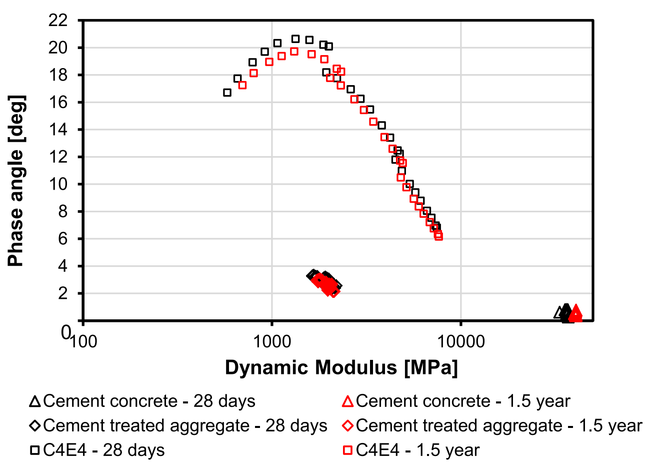


Fig. 7. Black diagram of mineral-cement-emulsion mixture (C4E4), cement concrete and cement-treated aggregate for the curing periods of 28 days and 1.5 year.

hypothesis is stated using equation: $H_0 : E_1(x) = E_2(x)$, while alternative hypothesis is stated using equation: $H_1 : E_1(x) \neq E_2(x)$. Where $E_1(x)$ and $E_2(x)$ are hypothetical mean values for two different sets of data that are being compared (results for 28 days of curing and 1.5 year of curing). The statistical analysis was conducted for the statistical significance p-value equal to $P = 1 - \alpha = 0.8$. P-value level was assumed taking into consideration natural inhomogeneity of properties of base materials used in the MCE mixtures, especially properties of the reclaimed asphalt.

Calculations of Student *t*-test values for mechanical properties of the tested materials were performed using the following equation:

$$t_{obl} = \frac{\underline{x}_1 - \underline{x}_2}{\sqrt{\frac{n_1 * s_1^2 + n_2 * s_2^2}{n_1 + n_2 - 2} * \left(\frac{1}{n_1} + \frac{1}{n_2}\right)}} \qquad (3)$$

where: t_{obl} – *calculated value of "t" parameter*, $\underline{x}_1, \underline{x}_2$ – *mean values*, n_1, n_2 – *the number of test specimens in test data*, s_1, s_2 – *standard deviation*, s_1^2, s_2^2 – *variance*.

Table 7 presents results obtained from the statistical analysis of dynamic moduli and phase angles determined for frequency of 10 Hz and temperature of +20 °C.

Table 6
Moduli and phase angles of all tested mixtures (frequency 10 Hz, temperature 20 °C) for curing times of 28 days and 1.5 year.

Material		Curing time	Modulus [MPa]	Change (increase) of modulus [MPa]/[%]	Phase angle [°]	Change (decrease) of phase angle [°]/[%]
Mineral-cement-emulsion mixture (2% emulsion)	2% cement	28 days	2 969	208/7.0%	13.10	1.49/11.4%
		1.5 year	3 177		11.61	
	4% cement	28 days	4 880	599/12.3%	10.38	0.79/7.6%
		1.5 year	5 479		9.59	
	6% cement	28 days	6 872	−228/−3.3%	9.42	1.19/12.7%
		1.5 year	6 644		8.23	
Mineral-cement-emulsion mixture (4% emulsion)	2% cement	28 days	2 662	679/25.5%	15.81	2.14/13.6%
		1.5 year	3 341		13.67	
	4% cement	28 days	4 224	128/3.1%	13.40	0.81/6.1%
		1.5 year	4 352		12.59	
	6% cement	28 days	4 997	541/10.8%	11.95	1.03/8.6%
		1.5 year	5 538		10.92	
Mineral-cement-emulsion mixture (6% emulsion)	2% cement	28 days	2 711	432/15.9%	18.72	2.51/13.4%
		1.5 year	3 143		16.21	
	4% cement	28 days	3 404	924/27.1%	15.43	1.94/12.6%
		1.5 year	4 328		13.49	
	6% cement	28 days	4 613	430/9.3%	13.24	0.79/6.0%
		1.5 year	5 043		12.45	
Cement concrete		28 days	36 499	4165/11.4%	0.67	0.15/21.7%
		1.5 year	40 664		0.52	
Cement-treated aggregate		28 days	2 029	2/0.1%	2.91	0.31/10.5%
		1.5 year	2 031		2.60	

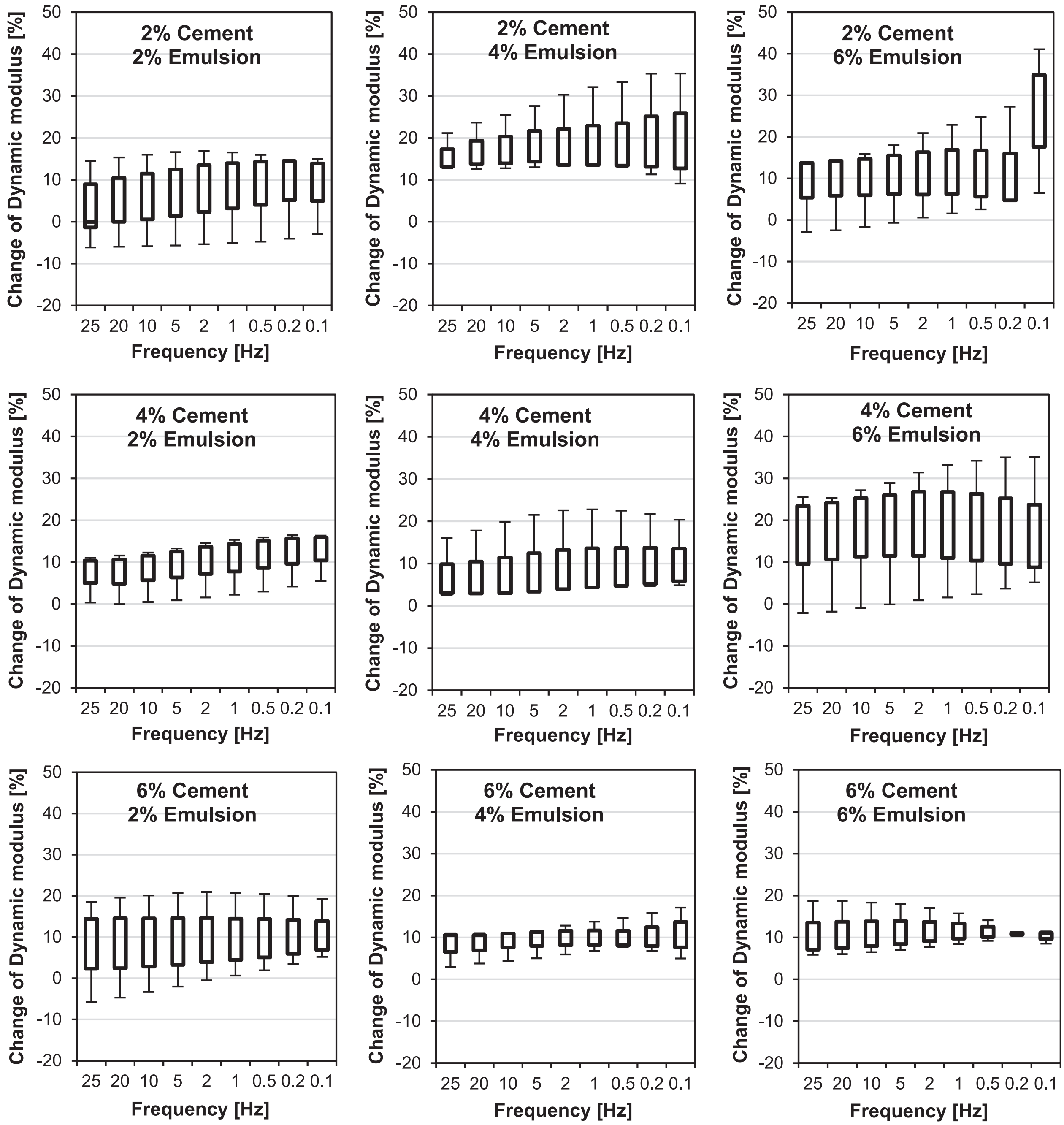


Fig. 8. The change of dynamic modulus for mineral-cement-emulsion mixtures as a function of frequency.

Conducted analysis showed that for the assumed confidence level of p-value equal to 0.8, the changes of dynamic moduli between curing periods of 28 days and 1.5 year are significantly different for five of the nine tested mixtures. Results obtained for cement concrete showed significant difference between the two curing periods. In the case of cement-treated aggregate, obtained results were the same from the statistical point of view for both curing periods. In the case of phase angles, statistical analysis presented significant differences for nearly all mineral-cement-emulsion mixtures and for cement concrete as well. In the case of cement treated-aggregate, similarly as in the case of dynamic modulus, statistical test did not show significant difference.

5. Conclusions and further studies

Mineral-cement-emulsion mixtures showed very complex behavior in the conducted laboratory tests. While general trends were observed, no direct correlations between the tested properties of MCE mixtures and combination of binding agents were found. It is probably caused by high inhomogeneity of base materials. For both curing periods all tested mixtures presented evident viscoelastic behavior, even when the amount of cement was equal to 6%. Nevertheless, the addition of cement as a binding agent strongly influences the curing process:

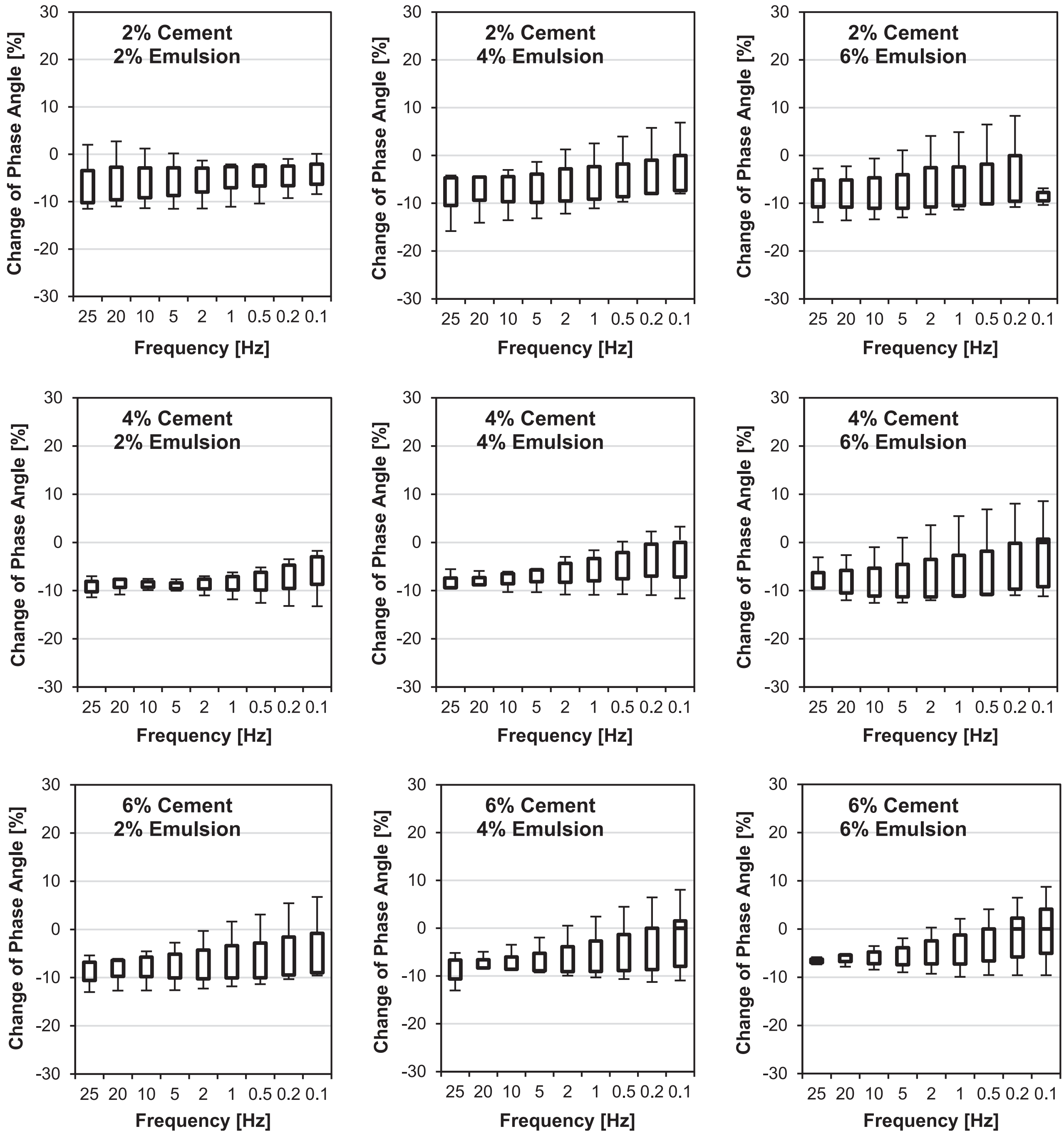


Fig. 9. The change of phase angles for mineral-cement-emulsion mixtures as a function of frequency.

1. The values of dynamic moduli increased for majority of tested mixtures. The increase of values ranged from 3% to 22%, with mean value from 9% to 14%. The range of increase is similar to other cement-treated materials and cement concrete (Fig. 2). While differences in absolute values are evident, the statistical analysis confirmed significant difference for only five of the nine tested mixtures.
2. The values of phase angles decreased over time of curing for majority of tested mixtures. The decrease of values ranged from 1% to 13%, with mean value from 4% to 8%. Despite relatively small changes in absolute values, the statistical analysis showed that almost all changes were significant.
3. The change of tested properties was more evident in the MCE mixtures treated with higher amount of cement, where the interquartile ranges of change are narrower than in other tested mixtures.

Further studies are planned in order to: (1) assess the impact of type of binding agents and their chemical composition on the change in rheological, mechanical and shrinkage properties of MCE mixtures; (2) measure further changes of the parameters for longer curing times (which would allow for better assessment of the mechanisms of mixture deterioration and improved design of pavement structures); (3) verify whether the change of properties

Table 7
The results of the statistical significance Student *t*-test, for the change in phase angles and dynamic moduli, t(P, k) = t(0.8; 4) = 1.533.

Material	Are the obtained results significantly different? Significance level: P=80%			
	t_{obl}	S	t_{obl}	φ
C2E2	1.255	NO	10.955	YES
C2E4	3.879	YES	3.014	YES
C2E6	2.840	YES	2.713	YES
C4E2	1.646	YES	2.140	YES
C4E4	0.297	NO	0.976	NO
C4E6	2.296	YES	2.681	YES
C6E2	0.755	NO	3.051	YES
C6E4	1.545	YES	1.691	YES
C6E6	1.469	NO	2.495	YES
cement concrete	1.903	YES	1.897	YES
cement treated aggregate	0.208	NO	0.409	NO

is caused mainly by hydration of cement or by other processes which have been described for bitumen bound materials, such as steric hardening.

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