Rheological properties of mineral-cement mix with foamed bitumen with the addition of redispersible polymer powder

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Abstract. The main objective of the research presented in this article was the search for response concerning the influence of redispersible polymer powder (RPP) on the rheological properties (evaluation of the complex modulus and phase shift angle) of a mineral-cement mix with foamed bitumen (MCAS). Redispersible polymer powders are used in the construction industry as ingredients of mortar, cement concrete, plaster, adhesives and tile cement. However, literature related to an analysis of the influence of redispersible polymer powders on the properties of a mineral-cement mix with foamed bitumen (MCAS). The objective of the research was executed through the use of known identification methods in terms of rheological properties. The analysis of the complex modulus was conducted spanning linear viscoelasticity for deformation spanning 25 to 50 µstrain per PN-EN 12697-24 annex D. The test set-up assumes the determination of the complex modulus $E^*$ and the phase shift angle $\phi$ at the full sine wave for direct expansion and compression using cylindrical samples (DTC-CY). Using the redispersible polymer powder for MCAS can improve hydrophobic properties and limit rigidity, together with an improvement of the elasticity of the mineral-cement mix with foamed bitumen (MCAS). The increase of elasticity of the MCAS can be caused by a modification of the mineral matrix and a disclosure of interactions between the aggregate-cement and the polymer. In case of a recycled substructure, this phenomenon is significant due to the placement of the substructure in an arrangement of construction layers (increased humidification). Test results have shown a reduction of the complex modulus and the emergence of the effect of the polymer at high temperatures and after a prolonged load period.

1. Introduction

An increase of the quality of construction materials is achieved in a process of modification, by introducing into the material an ingredient improving its properties. As a result of the modification, an improvement of the basic properties of the material is achieved. The modifier used most commonly in construction are polymers. Polymers used in the construction industry take one of several forms: solid polymers (redispersible powders, granules), water polymer dispersions, water polymer solutions, liquid synthetic resins [1-3]. Due to the character of changes depending on the influence of external factors, polymers can be subdivided into thermoplastic and duroplastic materials [3, 4]. Thermoplastic materials soften upon heating, and then harden upon cooling, whereas duroplastic materials harden irreversibly upon heating [4]. Thermoplastic polymers that are of most interest in the construction industry include: polyvinyl chloride, polyamides, ethylene-vinyl acetate copolymer (EVA), styrene-
butadiene-styrene block copolymer (SBS) [2-6]. The diversity of the available polymers necessitates the execution of comprehensive studies spanning their influence on the properties of the modified material. Polymers as modifiers are broadly used in cement concrete and bituminous mixture in road construction.

Polymers in cement concrete can be found in various forms and can be of various types. An analysis of literature had shown that polymers in the cement concrete industry are used for the purpose of modification of the contents of the cement concrete mix, as a result of which polymer-cement concretes (PCC), polymer impregnated concretes (PIC) and polymer concretes (PC) are obtained [6]. The use of polymer in the ingredients of cement concrete forces one to conduct detailed research. The process optimisation with the use of factorial experiment plans was used by many researchers for the purpose of identification of the influence of modifiers on the properties of cement concrete. A very detailed research plan was presented by Łukawski in his work [1]. The scope of study spanned an evaluation of the adhesion of polymer-cement concretes with respect to the modifier volume. An evaluation of the influence of redispersible polymer powder in the form of the ethylene-vinyl acetate copolymer (EVA) on the hydration process of Portland cement was described in their work by the authors [7]. The results of the research presented in the paper indicate the need of a newer approach to cement composites modified by EVA redispersible polymer powder additives. The period of usability of cement mixed with redispersible polymer powder (RPP) that was used in the mortar was studied following a period of conditioning at (40±1 °C) and relative humidity of 90 ±3% in [8]. Test results have shown that mortars containing RPP retain their mechanical properties and are better than mortars made of pure cement. An evaluation of durability of cement mortar that included various types of copolymers in their ingredients was the subject of [9-11]. Positive test results related to polymer modification of cement mortar and cement concrete types encourage researchers to take on this topic. Improvements in polymer quality and continuous processes of perfection of their make-up encourage ever newer research directions spanning the modification of cement concrete mixes.

The influence of modification on the properties of materials used in the execution of road surfaces is the subject of research. For modifications in the road construction industry, beside polymers, synthetic waxes are also used.

The influence of the modifier in the form of F-T synthetic wax on the properties of bitumen in their work was presented by the authors [11, 12]. The modification of bitumen by Fischer-Tropsch (F-T) synthetic wax was the subject of research presented by the authors in [12]. An evaluation of the influence of F-T synthetic wax on the rheological properties of 35/50 road asphalt was presented by Iwański and Mazurek [13]. However, the modifier most broadly used in road construction are polymers. Polymers are used in road construction for the purpose of modification of bitumen and the bitumen emulsion [14, 15]. Polymers belonging to the styrene-butadiene-styrene block copolymer group (SBS) are used for the purpose of modification of bitumen; these are composed of polyester blocks jointed by polybutadiene chains; another copolymer used for this purpose is the ethylene-vinyl acetate copolymer (EVA) [4, 16, 17]. Polymers used for the purpose of modification of bitumen most commonly take the form of granules, and the modification process requires the introduction of a high shear force [18]. To modify bitumen emulsions, synthetic latex SBR are used [19]. The possibility of replacement of the polymer by crumb rubber (CR) was the subject of research by authors in [20]. As shown by the research shown in this paper, modified bitumen with a rubber addition (CR) is characterised by a broad temperature range in terms of viscoelasticity as well as good properties in low and high temperatures.

An analysis of literature concerning the utilisation of the properties of polymer for the purpose of modification of a material permits the conclusion that the use of polymers as ingredients in mineral-cement mixes with foamed bitumen will influence the improvement of its parameters. The advantages obtained through the modification process will be similar to those that were identified following the addition of the polymer to cement concrete. Research shows that extension resistance and bending resistance are particularly improved, with a simultaneous slight drop of the elasticity modulus. Such parameters are desirable in mineral-cement mixtures with foamed bitumen. An increase in extension
resistance will protect the structure against excessive deformation caused by the moving vehicles. The deformation will be reversible and elastic. The introduction of a polymer powder to the ingredients of the mineral-cement mix with foamed bitumen will cause the emergence of a microstructure of the mineral polymer-cement composite. The phenomenon of the emergence of microstructures in polymer-cement concrete was described by [21-23].

Based on the conducted analysis of literature and the advantages stemming from the use of polymers in the ingredients of cement concrete, the determination of redispersible polymer powder on the properties of a mineral-cement mix with foamed bitumen becomes necessary. The article presents tests of a mineral-cement mix with foamed bitumen, for which various modifiers in the form of redispersible polymer powder were used. The main idea behind the conducted research was the determination of the influence that using modifies has on the complex modulus (E*) and the phase shift angle (φ). The evaluated modifiers are thermoplastic polymers (plastomers): VA-VeoVA - vinyl acetate-vinyl versatate copolymer, VA-VeoVa-Ac – vinyl acetate-vinyl versatate-acrylanes, EVA – ethylene-vinyl acetate copolymer and VA-Veo-E-Ac – vinyl acetate – vinyl cersatate-ethylene-acrylates. The volume of applied polymer in the contents of the mineral-cement mix with foamed bitumen was equal to 3% of the mix mass. The results of the tests showed a reduction of the complex modulus E* in low and high temperatures following the use of redispersible polymer powder with an unchanged cement volume in the ingredients of the mineral-cement mix with foamed bitumen.

2. The mineral-cement mix with foamed bitumen and its ingredients

2.1. Characteristics of redispersible polymer powders

Redispersible polymer powders used in the study are thermoplastic copolymers (plastomers), the protective colloid of which is poly-vinyl alcohol (PVA). Copolymers contain various polymer bases. They are used in the construction chemistry industry [24]. Table 1 shows basic information concerning redispersible polymer powders based on a literature analysis [24].

| Code | Description                  | Name                                         | Bulk density ISO 679 [g/l] | Polymer base                              |
|------|------------------------------|----------------------------------------------|----------------------------|-------------------------------------------|
| P1   | VA-VeoVA                    | vinyl acetate-vinyl versatate copolymer      | 550 - 570                 | vinyl acetate, vinyl versatate            |
| P2   | VA-VeoVa-Ac                 | vinyl acetate-vinyl versatate-acrylanes      | 530 - 670                 | butyl acrylate, vinyl acetate and vinyl versatate |
| P3   | EVA (VAE)                   | ethylene-vinyl acetate copolymer             | 450 - 500                 | ethylene and vinyl acetate                |
| P4   | VA/VV/E/Ac                  | vinyl acetate – vinyl cersatate-ethylene-acrylates | 350 - 550                 | ethylene, vinyl acetate, vinyl versatate and butyl acrylate. |

The polymers utilised for the research plan took the form of redispersible polymer powders, which are obtained by evaporating water from dispersion, and subsequent mixing with water which creates a dispersion again [25]. The size of polymer particles in the redispersible powder is usually 1-10 μm [3, 25].

2.2. Design of contents and physical properties of the MCAS with the addition of RPP

The design of the laboratory recipe of the mineral-cement mix with foamed bitumen was made per requirements of [26, 27]. The content of the mineral mix included the following ingredients: reclaimed asphalt pavement (RAP), broken natural aggregate (VA) of constant granularity 0/31,5 and 0/4 of dolomite origin, Portland cement CEM I 32,5 R and foamed bitumen. Foamed bitumen was formed using bitumen with a penetration of 50/70. The quality evaluation of the foamed bitumen was done based on the maximum Expansion Ratio (ERm) and the half-life H-l (Half life) [26]. Usability of
50/70 bitumen was determined per the experiment plan, according to the methods used by authors of [28, 29]. The optimum volume of water necessary to obtain foamed bitumen was determined based on the requirements of stipulations of [26]. The evaluation results are presented in figure 1.

![Figure 1. Characteristics of bitumen foam for bitumen 50/70](image)

The design of the mineral-cement mix with foamed asphalt assumed the achievement of continuous granularity and its curve that would fit in the field of good granularity [27]. As a result of the design, a mix content was obtained described by the granularity curve shown in figure 2.

![Figure 2. Granularity curve of the mineral mix](image)

The detailed content of the mineral mix (MM), the mineral-cement mix with foamed bitumen (MCAS) and the mineral-cement-polymer mix with foamed bitumen (MCAS+P) is shown in table 2.

| Ingredient name                                                      | Ingredient content (%) |
|---------------------------------------------------------------------|------------------------|
| Reclaimed asphalt pavement (RAP) (#22.4mm)                         | MM: 50.0               |
|                                                                     | MCAS: 47.3             |
|                                                                     | MCAS+P: 45.8           |
| Dolomite aggregate of continuous granularity 0/32 (#22.4 mm)       | MM: 25.0               |
|                                                                     | MCAS: 23.6             |
|                                                                     | MCAS+P: 22.9           |
| Dolomite aggregate of continuous granularity 0/4                   | MM: 25.0               |
|                                                                     | MCAS: 23.6             |
|                                                                     | MCAS+P: 22.9           |
| Portland cement CEM I 32.5R                                        | MM: -                  |
|                                                                     | MCAS: 3.0              |
|                                                                     | MCAS+P: 3.0            |
| Redispersible polymer powder (P1-P4)                                | MM: -                  |
|                                                                     | MCAS: -                |
|                                                                     | MCAS+P: 3.0            |
| Foamed bitumen 50/70                                               | MM: -                  |
|                                                                     | MCAS: 2.5              |
|                                                                     | MCAS+P: 2.5            |

Table 2. Content of the mineral-cement-polymer mix
The mineral mix (MM) content for all analysed mineral-cement mixes with foamed bitumen (MCAS) and including the addition of redispersible polymer powder (MCAS+P) is the same. The volume of binders is equal: cement 3.0%, foamed bitumen 2.5%, and in line with the values set forth in the provisions of [27]. Redispersible polymer powder were added at 3.0%. The volume of cement, foamed bitumen and RPP used was calculated with respect to the dry mass of mineral materials.

As a result, five mixes were obtained. Four MCAS+P mixes included redispersible polymer powders from P1 to P4 as per table 1. The fifth mix was a reference mix (MCAS-R) not containing RPP. Such an approach will permit a comparison and determination of the influence of redispersible polymer powders on the rheological properties of the mineral-cement mix with foamed bitumen.

3. Preparation, curing of the MCAS and MCAS+P samples

The research samples were prepared under laboratory conditions on the WLM30 mixer, and the bitumen foam was created using WLB10S. In order to reduce the influence of changes in the ingredient volumes due to their humidity, they were first dried to a fixed mass. The evaluation of the optimum moisture content (OMC) in the mineral mix was performed per requirements of standard EN 13286-2 [30] using the Proctor method, and the OMC result for the analysed reference MCAS mix is 5.8%.

The samples were compacted in a gyrating press [31]. The settings of the gyrating press were determined based on literature [32, 33]. The rotation count for compaction was chosen individually for the mixes so as to obtain densities, at which the free space content in the mineral-cement mix with foamed bitumen was equal to $V_m=10.0\%$.

Directly following creation, the samples were stored on the first day at a temperature of $+20\pm 5\, ^\circ C$ in forms. For the next day, the samples were taken out of forms and stored at relative humidity values between 40% and 70% for 14 days until the analysis of the complex modulus $E^*$.

3.1. Tests of the complex modulus $E^*$ and the phase shift modulus $\phi$

Tests of the complex modulus were done using the Direct Tension-Compression Test on Cylindrical Samples method (DTC-CY) [34], where the sample is subjected to a sine-cycle load at low deformation ranging from 25 to 50 $\mu m$ [34, 35]. The tests were performed for five temperature values (-15 °C, 5 °C, 13 °C, 25 °C, 40 °C) and six load periods (0.1 Hz, 0.3 Hz, 1 Hz, 3 Hz, 10 Hz, 20 Hz). Based on the obtained results of the complex modulus for the mineral-cement mixes with foamed bitumen, guide curve models were obtained. The guide curve model construction was performed using the Richards model [36], which is a modification of the model presented in a report of the NCHRP 9-29: PP 02 [37]. This model may be classified among asymmetric sigmoid mathematical models. The asymmetry is modelled using the curve course coefficient ($\lambda$). The asymmetric sigmoid function form is described by equation (1). The guide curve of the rigidity modulus used the temporal-temperature superposition rule. For this purpose, the introduction of the temperature shifts coefficient ($\alpha_T$) is necessary [35], as implemented in formula (1):

$$\log|E^*| = \delta + \frac{\alpha}{[1 + \lambda e^{\beta+\gamma \log(a+b T+c T^2)}]^2}$$  \hspace{1cm} (1)

where: $|E^*|$ – complex modulus, $\omega$ – angular frequency, $\delta$ – lower asymptote value, $\alpha$ – difference between upper and lower asymptote value, (guide curve adaptation parameter), $\lambda, \beta, \gamma$ – guide curve adaptation parameters, $T$ – test temperature, $T_{ref}$ – reference temperature, $a, b, c$ – model parameters.

4. Results of tests of rheological properties of MCAS spanning linear viscoelasticity - LVE

Using results of dynamic tests, e. g. the complex modulus ($E^*$) and phase shift angle ($\phi$), the influence of the RPP modifier on the rheological properties of the mineral-cement mix with foamed bitumen was determined. The dependence of the complex modulus on the phase shift angle is shown in figure 3. Such an approach permits a global evaluation of the character of viscoelasticity of mineral-cement...
mixes with foamed bitumen and the RPP polymer modifier. According to the formula (2), the complex modulus value is composed of the elasticity modulus (3) and the lost (viscosity) modulus (4):

\[
E^{*}_{(\omega)} = \frac{\sigma}{\varepsilon} = \frac{\sigma_0}{\varepsilon_0} \cdot e^{i\varphi} = |E^*| \cdot \cos\varphi + |E^*| \cdot i \cdot \sin\varphi = E_1 + E_2 \cdot i
\]

(2)

\[
E_1 = |E^*| \cdot \cos\varphi
\]

(3)

\[
E_2 = |E^*| \cdot \sin\varphi
\]

(4)

Figure 3. Comparison of Black curves for the analysed MCAS and MCAS+P mixes

It is clearly visible (figure 3) that the use of a modifier in the form of a redispersible polymer powder influences the change of rheological properties of MCAS. The smallest range of phase shift angles in terms of the analysed temperatures (-15 °C, 5 °C, 13 °C, 25 °C, 40 °C) and load periods (0.1 Hz, 0.3 Hz, 1 Hz, 3 Hz, 10 Hz, 20 Hz) was achieved by the reference mix MCAS-R, which confirms the domination of the elastic part in the complex modulus. Such dependence proves the possibility of emergence of dry cracks during cylindrical loads – the mix is characterised by low susceptibility. The use of redispersible polymer powder in the MCAS causes an increase of the phase shift angle value, and thus, an increase of mix susceptibility. Such behaviour of mineral-cement-polymer mixes with foamed bitumen (MCAS+P) spanning load times and temperatures is identical as for bituminous mixture [38] and different from those of recycled mixes composed barely of hydraulic binder material. A confirmation may be found by comparison of test results for mixes with the addition of polymer to the reference mix. The share of the viscous part in the reference mix is low. The low share of the viscous part as a component of the complex modulus is characteristic for mineral-cement mixes with bitumen binders [39, 40].

A comprehensive evaluation of the influence of redispersible polymer powder (RPP) on the complex modulus \(E^*\) was conducted by comparing the course of guide curves for modifier-containing mixes (MCAS+P) to that of the reference mix (MCAS-R). The assumptions of the temporal-temperature superposition rule were used to construct the guide curves of the complex modulus of mineral-cement mixes with foamed bitumen [37, 41]. For this purpose, optimisation of the sigmoid function using the divergence square sum reduction method for the determined complex moduli. As a result of optimisation of function curves, it was necessary to determine parameters of guide curves (\(\alpha, \beta, \gamma, \delta, \lambda, a, b\)). An evaluation of the parameters was shown in table 4, and the course of guide curves of the complex modulus was shown in figure 4.
Figure 4. Guide curves for the complex modulus of the analysed MCAS and MCAS+P mixes

Table 3. Adaptation parameters for guide curves of the analysed mixes

| Mixtures   | Fitting parameters | Modulus [MPa] | Fit quality [%] |
|------------|--------------------|---------------|-----------------|
|            | a      | b      | α       | β       | γ       | δ       | λ       | E_∞    | E_0    | RMSE   |
| MCAS-R     | 0.905  | -0.058 | 2.498   | -0.132  | -0.114  | 2.498   | 0.1     | 315    | 99212  | 3.42   |
| MCAS+P1    | 1.468  | -0.04  | 2.204   | 0.083   | -0.236  | 2.204   | 0.1     | 159    | 25546  | 4.93   |
| MCAS+P2    | 1.645  | -0.059 | 2.141   | -0.009  | -0.288  | 2.141   | 0.1     | 138    | 19153  | 5.15   |
| MCAS+P3    | 1.913  | -0.026 | 2.319   | 0.532   | -0.196  | 2.319   | 0.1     | 208    | 43496  | 4.31   |
| MCAS+P4    | 1.750  | -0.036 | 2.243   | 0.436   | -0.228  | 2.243   | 0.1     | 175    | 30677  | 4.96   |

An analysis of the course of guide curves lets one conclude (figure 4) that the use of polymer in the content of the mineral-cement mix with foamed bitumen causes a reduction of the complex modulus (E*) irrespective of the polymer base type. A drop of the complex modulus is in line with the general rule of application of polymers [1, 3]. A change of the complex modulus for an infinitely short load time (table 3) in case of a copolymer mix used in the content causes a drop in the value of modulus E* of a factor between two to five with respect to the reference mix. For a long load time, only a fall by a factor of two was observed. Such behaviour of the mineral-cement mix with foamed bitumen, in which modifiers were used, may prove higher resistance to low-temperature cracking and higher resistance to long-term loads, e.g. creeping in high temperatures, in comparison to the reference mix.

5. Conclusions
The conducted research and their analyses permit the formulation of the following conclusions:

- The introduction of copolymers in the form of RPP to the mineral-cement-bitumen composite causes an expansion of the viscoelastic range, in which the mineral-cement mix with foamed bitumen will work. This dependence stems from the increase of the phase shift angle as compared to the reference mix.
- The use of a modifier in the form of a redispersible polymer powder in the content of the mineral-cement mix with foamed bitumen greatly influences the reduction of the complex modulus value at low temperatures (short load times). The achievement of such a dependence may indicate higher resistance to low-temperature cracking than is the case for traditional mixes.
- The use of a modifier in the form of ethylene vinyl acetate copolymer (EVA) the least affects on the value of the complex module for the mineral-cement-polymer mix with foamed
bitumen (MCAS+P). The evaluation of the Blacka curve for the MCAS+P4 mix shows the highest influence of the polymer at high temperatures/long load times.

The achieved research results for the mineral-cement-polymer mix with foamed bitumen may indicate the formation of the mineral-polymer-cement composite microstructure that may influence the improvement of the properties of MCAS. However, the determination of the entire spectrum of influence of redispersible polymer powders on properties of MCAS requires the execution of comprehensive studies spanning low- and high-temperature resistance, static loads and fatigue tests.

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