The Influence of Hydrated Lime, Portland Cement and Cement Dust on Rheological Properties of Recycled Cold Mixes with Foamed Bitumen

Przemysław Buczyński¹, Marek Iwański²

^{1,2} Department of Transportation Engineering, Faculty of Civil Engineering and Architecture Kielce University of Technology, Kielce, Poland E-mails: ¹p.buczynski@tu.kielce.pl (corresponding author); ²iwanski@tu.kielce.pl

Abstract. This article presents a laboratory evaluation of the viscoelastic properties of recycled base courses produced with different fillers. The aim of this study was to investigate the influence of loading time and temperature on the complex modulus (E^*) and the phase angle (φ) of recycled base courses with respect to selected additives used. The mixtures contained reclaimed asphalt pavement RAP, crushed stone from existing base courses and virgin aggregate. Foamed bitumen 50/70 at 2.5% was used as a binder. The hydraulic binder constituted 3.0% of the recycled base course mixture. Portland cement, hydrated lime and cement kiln dust CKD were added as fillers.

Evaluation of rheological properties of recycled base courses according to selected additives was carried out to the procedure set out in EN 12697-26 annex D. The evaluation of stiffness modulus was conducted in the direct tension-compression test on cylindrical samples (DTC-CY). The samples were subjected to the cycles of sinusoidal strain with an amplitude $\varepsilon_0 < 25\mu\epsilon$. All tests were performed over a range of temperatures (5 °C, 13 °C, 25 °C, 40 °C) and loading times (0.1 Hz, 0.3 Hz, 1 Hz, 3 Hz, 10 Hz, 20 Hz). The results were used to model stiffness modulus master curves of the recycled base courses containing selected additives in the hydraulic binder.

Keywords: recycled cold mix, foamed bitumen, complex modulus, rheological properties, active filler (binder)

Conference topic: Roads and railways.

Introduction

Full-depth cold recycling with foamed bitumen is an alternative to conventional technologies used in the production of HMA (Hot MIx Asphalt) base courses. Recycling of existing pavement materials for the construction of base courses contributes to the reduction of road project costs (Chomicz-Kowalska, Stepień 2016). A proper composition of mineral constituents provides base courses with the ability to carry wheel load induced stress to the subgrade. The presence of active fillers such as Portland cement, cement dust, fly ash and hydrated lime in recycled mixtures with foamed bitumen affects their basic physical and mechanical properties, as confirmed by numerous publications (Halles, Thenoux 2009; Niazi, Jalili 2009; Iwański et al. 2016). Active fillers are being widely used in bituminous mixtures (Iwański, Mazurek 2013). The study of their influence on the resistance to moisture and frost damage (Buczyński 2016) confirmed that the active filler mix used in the base layer could provide optimal test results. However, the identification of basic physical and mechanical properties and the resistance to weathering may not suffice to fully evaluate variable traffic induced loading. The identification of rheological characteristics of mixtures intended for cold recycled base courses is of crucial importance. Viscoelastic properties of cold mixtures with foamed bitumen have been widely investigated with respect to the reclaimed asphalt content (Godenzoni et al. 2017), foaming additives and fibre reinforcement (Martinez-Arguelles et al. 2015), and technologies of base course construction (Leandri et al. 2015). The use of active fillers for recycled mixtures with foamed bitumen strongly depends on the evaluation of their effect on these properties. High stiffness moduli of recycled mixtures with foamed bitumen and active fillers do not guarantee that the base course will demonstrate high bearing capacity or durability. The stiffness modulus of a recycled mixture will change at low temperatures (short loading time) and high temperatures (long loading time), as it does in HMA mixtures (Alam, Hammoum 2015). An active filler added to a recycled mixture with foamed bitumen will change its structure and reduce the viscous part. Nevertheless, due to a variety of available types of active fillers and hydraulic binders (mixes of active fillers) produced of them, the effects they may have on foamed bitumen stabilized recycled base rheology need to be thoroughly investigated.

Evaluation of rheological properties of recycled base courses

Research methods

Prior to choosing the type of active filler for use in the recycled mixture, physical and mechanical properties of the mixture have to be identified. A comprehensive evaluation of the influence of active fillers on the properties of mixtures requires determining rheology of the material. All mineral mixtures with bituminous binders exhibit viscoelas-

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tic behaviour (Pokorski *et al.* 2015; Olard, Di Benedetto 2003). Parameters that adequately describe rheological parameters of recycled cold mixtures with foamed bitumen (FB-RCM) are the complex modulus (E^*) and phase angle (φ). Analysis of these parameters will facilitate defining the influence of active fillers in recycled cold mixtures with foamed bitumen on their rheological properties.

This paper discusses the results of rheological properties testing of recycled mixtures with foamed bitumen in terms of active filler types used. The parameters were determined in the direct tension-compression test on cylindrical samples (DTC-CY) in compliance with the requirements of the EN 12697-26 standard. The samples were subjected to sinusoidal loading that induced low strain $\varepsilon_0 < 25 \ \mu$ E. The test was carried out at four temperatures (5 °C, 2 °C, 13 °C, 25 °C, and 40 °C) and six loading times (0.1 Hz, 0.3 Hz, 1 Hz, 3 Hz, 10 Hz, and 20 Hz). The values obtained were used to calculate the complex modulus (E*) and phase angle (φ). Due to viscoelastic response of bituminous mixtures, the strain (ε_0) will lag the stress (δ_0) by a phase angle (φ), as described by (Godenzoni *et al.* 2017; Gaweł *et al.* 2014). The stress is described by equation (1), the strain by equation (2) and the complex modulus by equation (3). The same dependencies were used by (Clyne *et al.* 2003; Sybilski, Kukiełka 2006):

$$; \sigma = \sigma_0^* \sin(\omega \cdot t) \tag{1}$$

$$; \varepsilon = \varepsilon_0^* \sin(\omega \cdot t - \varphi)$$
⁽²⁾

$$\mathbf{E}_{(i\omega)}^{*} = \frac{\sigma}{\varepsilon} = \frac{\sigma_{0}}{\varepsilon_{0}} \cdot \mathbf{e}^{i\varphi} = \left| \mathbf{E}^{*} \right| \cdot \cos\varphi + \left| \mathbf{E}^{*} \right| \cdot i \cdot \sin\varphi = E_{1} + E_{2} \cdot i \,. \tag{3}$$

As shown in equation (3), the complex modulus comprises the elastic modulus (4) and the loss modulus (viscous) (5):

$$\mathbf{E}_{1} = \left| \mathbf{E}^{*} \right|^{*} \cos \varphi ; \tag{4}$$

$$\mathbf{E}_2 = \left| \mathbf{E}^* \right|^* \sin \varphi \,, \tag{5}$$

where: σ - stress, ε - strain, $E^*_{(i\omega)}$, E^* - complex modulus, σ_0 - initial stress, ω - angular frequency, t - time, ε_0 - initial strain, φ - phase angle, E_1 - elastic modulus (realy part of the complex modulus), E_2 - loss modulus (imaginary part of the complex modulus), i - imaginary unit.

The results from the complex modulus tests of the recycled mixture with foamed bitumen (FB-RCM) allowed the modelling of master curves for the complex modulus and demonstrating the effects of selected active fillers.

Analysis of the influence of active fillers on the properties of FB-RCM used a non-symmetric Richards model applied by (Witczak, Root 1997; Rowe, Sharrock 2011) which is a modified version of the model set out in NCHRP 9-29: PP 02 used by Jaczewski, Judycki (2014) to construct master curves. The model can be classified as a non-symmetric sigmoidal mathematical model with a λ fitting parameter added. The non-symmetric sigmoidal function is described by equation (6):

$$\log \left| \mathbf{E}^* \right| = \delta + \frac{\alpha}{\left[1 + \lambda e^{\beta + \gamma \log \omega} \right]^{1/\lambda}} , \qquad (6)$$

where: $|E^*|$ – complex modulus, ω – angular frequency, δ – value of the lower shelf asymptote, (master curve fitting parameter), α – difference between the values of upper and lower asymptote, (master curve fitting parameter), λ , β , γ – master curve fitting parameters.

Since the development of the stiffness modulus master curve is based on the time-temperature superposition principle, a temperature shift factor (α_T) has to be added. With the shift factor (equation (7)), it is possible to describe the changes in the FB-RCM properties, temperature and loading time (superposition principle). To describe the changes of the stiffness modulus, the Arrhenius model used by (Rowe, Sharrock 2011; NCHRP 614 2008; Medani, Huurman 2003) was applied:

$$\log \alpha_{\rm T} = \frac{\Delta E_{\rm a}}{19.14714} \left(\frac{1}{\rm T} - \frac{1}{\rm T_{\rm R}} \right) = \frac{\Delta E_{\rm a}}{2.303\rm R} \left(\frac{1}{\rm T} - \frac{1}{\rm T_{\rm R}} \right),\tag{7}$$

where: α_T – shift factor, T – temperature at which the shift factor was determined, T_R – reference temperature, ΔE_a – activation energy treated as a fitting parameter, R – the universal gas constant R = 8.314 J/(mol·K).

Material under test - recycled mixture design

Analyses in this paper focused on rheological properties of recycled base courses with foamed bitumen in view of different active fillers used. The recycled mineral mixture for cold recycling (RCM – Recycled Cold Mix) with foamed bitumen (FB-RCM) was designed based on the optimal grading curve, in compliance with the guidelines set out in (Wirtgen 2010). The combination of the constituents in the FB-RCM design guaranteed the correspondence to and simulation of the cold recycling process.

The following constituents were used: RAP (Reclaimed Asphalt Pavement), crushed stone from existing base courses and well graded aggregate 0/4 mm. To provide the required aggregate size to fit the Marshall mould, aggregates larger than 31.5 mm were sieved out. Figure 1 shows the design grading curve. Table 1 summarizes the percentage content of mineral constituents.



sieve size[mm]

Fig. 1. Design grading curve of the mineral recycled base mixture

Percentage content (%)		
Cold Recycled Mix		
48.8		
19.5		
26.3		
2.5		
3.0		

Table 1. Percentage content of FB-RCM constituents

The bitumen binder was derived from road bitumen 50/70 and used in the recycled base course at 2.5%. The foamed bitumen content was consistent with the national requirements. The quality of the foamed bitumen was characterized based on the maximum expansion ratio, ERm = 11.0 and the half-life, H-l = 9.0 [20] at the optimal foaming water content of 2.9%.

The rheological properties of the FB-RCM were tested with selected fillers, such as Portland cement (CEM I 32,5R), hydrated lime (Ca(OH)₂) and cement kiln dust (CKD) in the proportion 1/3 ($\frac{1}{3}$ CEM+ $\frac{1}{3}$ Ca(OH)2+ $\frac{1}{3}$ CKD). Portland cement is the basic binding materials used in the cold recycled mineral mixture design. It allows comparing alternative active fillers relative to the conventional filler.

Laboratory specimens of the FB-RCM were prepared according to the specifications set out in EN 12697-26, with the required diameters (D) from 50 mm and 160 mm and heights (H) between 1.8 and $3 \times$ diameter of the specimen. The dimensions of the FB-RCM specimens prepared for testing were D = 100 mm and H = 180 mm. The specimens were sawed from the compacted slab prepared in compliance with EN 12697-33. The curing period was 90 days at a temperature of 25±5 °C and humidity of 40±10% established according to the methods described by the authors (Telejko, Zender-Świercz 2016). The extended curing duration, from conventional 28 to 90 days, eliminated a delay in the active fillers setting time. The authors (Wawrzeńczyk *et al.* 2016) demonstrated a minor effect of active fillers on strength levels after 90 days of curing.

Results

Dynamic testing – complex modulus E^* and phase angle φ

The complex modulus and the phase angle provide comprehensive information about the FB-RCM. Diversified mineral compositions, various content of foamed bitumen and type of active filler all affect the performance of the recycled base course within the linear viscoelastic range (Godenzoni *et al.* 2017). Variable values of the stiffness modulus translate into the bearing capacity of the base course produced from the recycled mixture (Martinez-Arguelles 2015). The stiffness modulus values were determined according to EN 12697-26 in the direct tension-compression test (DTC-CY). The test results are presented in Figure 2.



Fig. 2. Isotherms of complex modulus E* as a function of load frequency and different temperatures. a) temperature 5 °C, b) temperature 13 °C, c) temperature 25 °C, d) temperature 40 °C

An increase in loading time (lower frequency) causes the complex modulus to decrease in all recycled mixtures with foamed bitumen, regardless of active filler type used. The lowest value of the complex modulus over the temperature range under analysis (5 °C, 13 °C, 25 °C, 40 °C) is seen in the FB-RCM with hydrated lime (Ca(OH)₂). Unlike other active fillers used, hydrated lime does not improved binding properties. The lowest complex moduli for all recycled mixes being analysed was recorded at the test temperature of 40 °C. It has to added that the lowest sensitivity to both loading time and temperature was recorded for the FB-RCM with Portland cement (FB-RCM - CEM). The characteristics obtained for this mixture confirm its stability in the response to temperature and loading time

changes. The recycled base course with Portland cement guarantees attaining a high complex modulus and resistance to high traffic-induced normal stresses. The base course demonstrating high values of stiffness modulus ($E^* > 15\,000$ MPa) will be vulnerable to cracking due to excessive stiffness. In summary, the results show that the behaviour of FB-RCMs with regard to the time of loading and test temperature is convergent with that of bitumen and bituminous mixtures, as reported earlier by Iwański and Mazurek (2013), Daniel *et al.* (1998).

To conduct a comprehensive assessment of the influence of active fillers on rheological properties of the mixture with foamed bitumen, it is necessary to represent the complex modulus as a function of frequency (Fig. 3). The elastic portion of the complex modulus increases with decreasing phase angle.



Fig. 3. Black Curve for the complex modulus of the recycled cold mixes with foamed bitumen

The recycled mixture with foamed bitumen and portlad cement (FB-RCM - CEM) shows the lowest phase angle values, with the minimum of 2.2° and maximum of 4.5°. This means that the mixture exhibits minor sensitivity to changes in loading time and temperature. The low maximum phase angle also means that the elastic modulus dominates in the FB-RCM – CEM, which gives the recycled base the properties of an elastic material ($\varphi = 0^\circ$). Different characteristics were observed in the recycled bases produced with other fillers. Considering viscoelastic materials in general, the degree range of the phase angle was significant. The widest range of phase angle values was recorded for the mixture with foamed bitumen and CKD active filler. This filler guarantees attaining complex moduli similar to those for Portland cement (CEM I 32,5R) but it does not guarantee attaining the recycled base course that is stable and resistant to loading time.

Relationships similar to those in the Black curve can be observed in Figure 4, which shows the viscous modulus E_2 as a function of the elastic modulus E_1 .



Fig. 4. Cole-Cole diagram for the recycled cold mixes with foamed bitumen

It must be remembered that the type of active filler affects the performance of the base layer within the viscoelastic range. The recycled mixture with foamed bitumen and Portland cement shows the highest portion of elastic component. Analysis of the RCM with hydrated lime (Ca(OH)₂ indicates that this mixture has the lowest portions of the complex modulus components, E_1 (viscous) and E_2 (elastic).

Complex modulus master curves for FB-RCM in terms of additives/fillers

The complex modulus master curves for the recycled mixtures with foamed bitumen were constructed according to active filler types, based on the time-temperature superposition principle. For this purpose, optimization of the sigmoidal function was performed by minimizing the sum of squares in the frequency domain for the complex moduli under consideration. The optimisation allowed determining master curve parameters (α , β , γ , δ and λ). To assess the goodness-of-fit of sigmoidal function to the experimental values, the coefficient of determination R² and the mean squared normalized error MSE were employed (Yusoff *et al.* 2013). The graphical representation of the master curves for the reduced frequency domain and reference temperature 20 °C is shown in Figure 2. Table 2 shows the assessment of fitting the FB-RCM data to the mathematical model of the sigmoidal function.



Fig. 4. Master curve for FB-RCM and different types of active fillers

Table 2. Evaluation of parameters of fitting data to the mathematical model of the sigmoidal function for the FB-RCM

Mixture type	Master curve fitting parameters					Goodness of fit	
	α	β	γ	δ	λ	R2	MSE [%]
FB-RCM – CEM	0.703	-1.803	-0.541	3.628	1.000	0.99	0.20
FB-RCM - Ca(OH) ₂	1.217	-2.646	-0.520	2.828	1.000	0.99	0.10
FB-RCM – CKD	2.822	-3.301	-0.450	1.423	1.000	0.99	0.12
FB-RCM - $\frac{1}{3}$ CEM+ $\frac{1}{3}$ Ca(OH) ₂ + $\frac{1}{3}$ CKD	1.008	-2.659	-0.521	3.135	1.000	0.99	0.11

The high, close to 1.0, value of R^2 confirms very good fitting of the function to the complex modulus data shifted by the α_T factor. The MSE values are very low (MSE < 1.0%) thus, the differences between the measured and the model values of complex modulus are very small, reaching a maximum of 0.20%. Analysis of the " λ " parameter indicates a symmetric distribution of the complex modulus at low and high frequencies for each mixture-filler combination tested.

The master curves developed show how the active fillers affect the viscoelastic properties of recycled mixtures with foamed bitumen. The effect of the CKD on the mixture was found to be different from those observed with other analysed fillers. The long time of loading (high temperature) causes a rapid change of complex modulus of the recycled base layer, which may have a negative impact on its bearing capacity under real conditions. The base layer may deteriorate as a result of overloading (excessive stress). Active fillers such as hydrated lime and Portland cement

used in the mixture do not change considerably the behaviour of the recycled base under variable load-temperature conditions.

Conclusions

Analysis of the rheological properties of recycled mixtures with foamed bitumen showed different influence of active fillers (CEM, Ca(OH)₂, CKD and $\frac{1}{3}$ CEM+ $\frac{1}{3}$ Ca(OH)₂+ $\frac{1}{3}$ CKD) on the performance of base layers at varied loading time and temperature. The testing results allow formulating the following conclusions:

- The highest portion of elastic component was found in the recycled mixture with foamed bitumen and Portland cement (FB-RCM CEM). However, high complex modulus values may result in excessive stiffness of the recycled base layer and shrinkage cracking;
- The widest degree range of phase angle (ϕ) was observed in the recycled mixture with foamed bitumen and cement kiln dust (FB-RCM CKD) over the entire range of temperature and loading time or loading frequency. This behaviour will contribute to a high rate of durability changes in structures with this recycled base layer at variable loading time;
- The lowest portion of viscous component but, at the same time, also elastic component with respect to all fillers tested was observed in the recycled mixture with foamed bitumen and hydrated lime (FB-RCM Ca(OH)₂);
- High complex moduli with an alternative filler (CKD) used in the recycled base layer with foamed bitumen at low loading times (above 8Hz) do not guarantee obtaining similar bearing capacity levels over long loading times (at high temperatures);
- The base layer containing cement dust attained the complex modulus of 15 147 MPa at a frequency of 10Hz and a temperature of 20 °C, but with short loading times and temperature of 20 °C, the complex modulus value was 15 times lower;
- It seems necessary to determine the fatigue life of recycled mixtures with foamed bitumen according to the type of active filler to be able to define the behaviour of the base layer under dynamic loading.

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