Inactive Mineral Filler as a Stiffness Modulus Regulator in Foamed Bitumen-Modified Recycled Base Layers

Przemysław Buczyński ¹, Marek Iwański ¹

¹ Department of Transportation Engineering, Faculty of Civil Engineering and Architecture, Kielce University of Technology, al. Tysiąclecia Państwa Polskiego 7, 25-314 Kielce

p.buczynski@tu.kielce.pl

Abstract. The article presents the results of a cold recycled mix test with a foam bitumen including the addition of the inactive mineral filler as a dust of basalt. Basalt dust was derived from dedusting system by extraction of aggregates in the mine. Assessment of the impact of a basalt dust on the properties of a recycled base layer was carried out in terms of the amount of mineral filler (basalt) in the composition of the mineral mixture. This experiment involved a dosing of mineral filler in range from 5 to 20% with steps of 7.5% in the mineral mixture composition. The foamed bitumen was performed at optimum foaming process settings (ie. bitumen temperature, air pressure) and at 2.5% of the water content. The amount of a hydraulic binder as a Portland cement was 2.0%. The evaluation of rheological properties allowed to determine whether the addition of inactive mineral fillers can act as a stiffness modulus controller in the recycled base layer. The analysis of the rheological properties of a recycled base layer in terms of the amount of inactive fillers was performed in accordance with given standard EN 12697-26 Annex D. The study was carried out according to the direct tension-compression test methodology on cylindrical samples. The sample was subjected to the oscillatory sinusoidal strain ε₀ < 25με. Studies carried out at a specific temperature set-points: -7ºC, 5ºC, 13ºC, 25ºC and 40ºC and at the frequency 0.1 Hz, 0.3 Hz, 1 Hz, 3 Hz, 10 Hz and 20 Hz. The obtained results allow to conclude that the use of an inactive filler can reduce the stiffness of an appropriate designed mixes of the cold recycled foundation. In addition, the analysis of the relation E’-E” showed a similar behaviour of a recycled base, regardless of the amount of inactive fillers in the mix composition, at high temperatures/high frequency of induced load.

1. Introduction
The bitumen foaming process alters the viscosity of binders and aids in the coating aggregate grains with no need to heat them to high temperatures [1]. Half warm mix asphalt (HWMA) [2] technologies are an excellent example of foamed bitumen application in opposition to hot mix asphalt (HMA) technologies. Additional advantages from bitumen foaming are observed in recycled cold mixes (RCM) technology. The cold recycling technology with foamed bitumen requires that optimal amount of the finest (below 0.063 mm) mineral material be used. According to the requirements for the optimal grading, recycled cold mixes should contain a minimum of 4% and maximum of 20% fines of fraction below 0.063 mm. Insufficient content of fillers in a recycled cold mix results in the formation
of bitumen rich agglomerations which prevent the formation of a mastic that binds coarse grains, improves compaction through the slip of larger grains, and tightens the asphalt mix structure.

Insufficient content of fillers below 0.063 mm can be remedied by incorporating into the recycled mixture with foamed bitumen mineral dust derived from dedusting systems in bitumen mix production facilities or from the washing/dedusting of aggregates in quarries. Possible applications of mineral dusts from the gabbro aggregate dedusted in bitumen mix production plants were presented by [3]. Studies show that those ideas should be developed further. Mineral dusts (inactive filler) are used in asphalt mixes as the mineral filler fraction to form the mastic. Possible use of inactive fillers (from limestone and basalt) in conjunction with polymer modified bitumen was discussed in [4]. The results of studies allow stating that inactive mineral fillers can be successfully applied as elastic forming material to asphalt mixes without the risk of losing mechanical properties. Applications of fine grain mineral material such as ordinary Portland cement (OPC), fly ash (FA), ground granulated blast furnace slag (GGBS) and silica fume (SF) to cold recycling technology with bitumen emulsion were reported by authors of [5] and to the use of coal waste in recycled asphalt mixes with bitumen emulsion by authors of [6]. Further insight into possible management of waste material (mineral dust) in cold recycling technology with foamed bitumen appears to be necessary. A wide spectrum of dust fraction gradation offers such an opportunity without the violation of permissible technological regimes.

2. Methodology of recycled base complex modulus testing

2.1. Research methods

A comprehensive evaluation of the effect of inactive mineral filler (mineral dust) on the stiffness modulus of recycled mixtures with foamed bitumen was performed through identification of rheological properties. All mineral mixtures with bitumen binder exhibit viscoelastic behaviour [7, 8]. Complex modulus ($E^*$) and phase angle ($\phi$) characterize the rheological properties of a recycled mix with foamed bitumen very well. Analysis of these properties will clearly show the influence of inactive fillers present in the recycled mixture with foamed bitumen on the changes in complex modulus ($E^*$).

The complex modulus was determined by the direct tension-compression test on cylindrical specimens (DTC-CY) following recommendation in EN 12697-26. The DTC-CY method the specimen is subjected to cyclic sinusoidal loading, which induces low strains, $\varepsilon_0 < 25 \mu e$. This approach allows collecting data pertaining to complex modulus within the linear viscoelastic response [9]. The stress is described by equation (1), the strain is defined by equation (2) and the complex modulus is represented by dependence (3) [10, 11, 12]:

$$\sigma = \sigma_0 \sin(\omega \cdot t)$$

$$\varepsilon = \varepsilon_0 \sin(\omega \cdot t - \phi)$$

$$E^*_1 = \frac{\sigma}{\varepsilon} = \frac{\sigma_0}{\varepsilon_0} \cdot e^{i\phi} = |E^*| \cdot \cos \phi + |E^*| \cdot i \cdot \sin \phi = E_1 + E_2 \cdot i$$

According to (3), the complex modulus value has two components: the elastic (4) and loss (viscous) (5) moduli:

$$E_1 = |E^*| \cdot \cos \phi$$

$$E_2 = |E^*| \cdot \sin \phi$$

where: $\sigma_0$ – initial stress, $\omega$ – angular frequency, $t$ – time, $\varepsilon_0$ – initial strain, $\phi$ – phase angle, $E_1$ – elastic modulus (real part of the complex modulus), $E_2$ – loss modulus (imaginary part of the complex modulus), $i$ – imaginary unit.
Test results for the complex modulus of recycled mixtures with foamed bitumen (FB-RCM) allow the modelling of complex modulus master curves and demonstrating the effects of the inactive mineral filler content in the FB-RCM. The influence of active fillers on the properties of FB-RCM was analysed through a non-symmetric Richards’s model [13], which is a modified version of the model set out in NCHRP 9-29: PP 02 [12]. The model is a non-symmetric sigmoidal mathematical model to which a $\lambda$ asymmetry parameter was introduced. The non-symmetric sigmoidal function has the form (6):

$$
\log|E'| = \delta + \frac{(E_{\text{Max}} - \delta)}{[1 + \lambda e^{\beta + \gamma \log \omega}]^{\lambda}}
$$

, where: $|E'|$ – complex modulus, $\omega$ – angular frequency, $\delta$ – value of the lower shelf asymptote, (master curve fitting parameter), $(E_{\text{Max}} - \delta)$ – the difference between the upper and lower asymptotes, (master curve fitting parameter), $\lambda, \beta, \gamma$ – master curve fitting parameters.

The stiffness modulus master curve design is based on the time-temperature superposition principle [14], thereby the temperature shifts factor ($\alpha_T$) needs to be defined. For the majority of asphalt mixtures, the reference temperature is determined from the Williams-Landel-Ferry model (WLF) [15]:

$$
\alpha_T = \frac{C_1 (T - T_{\text{ref}})}{C_2 + (T - T_{\text{ref}})}
$$

, where: $\alpha_T$ – shift factor, $T$ – temperature at which the shift factor is determined, $T_{\text{ref}}$ – reference temperature, $C_1, C_2$ – WLF model parameters (material constants).

2.2 Recycled mixture design

The FB-RCM was designed for the optimal grading curve following the guidelines in [16]. The percentage of dolomite inactive filler in the mixture was varied (5%, 12.5% and 20%). The percentage of mineral constituents was reduced by the percentage content of foamed bitumen. The design grading curve is shown in Table 1. The graphical representation of the grading curves is shown in Fig. 1.

| Constituent | Percent content (%) |
|-------------|---------------------|
|             | FB-RCM (F=5%) | FB-RCM (F=12.5%) | FB-RCM (F=20%) |
| RAP – reclaimed asphalt pavement 0/32 (#22.4) | 45.4 | 42.4 | 39.0 |
| VA – well graded aggregate 0/32 (#22.4) | 45.3 | 41.0 | 37.1 |
| Mineral dust– dolomite | 4.9 | 12.2 | 19.5 |
| Foamed bitumen 50/70 | 2.4 | 2.4 | 2.4 |
| Portland cement (CEM I 32.5 R) | 2.0 | 2.0 | 2.0 |

The bitumen binder was obtained from paving bitumen 50/70 and used in the recycled base course at 2.5%. The content of foamed bitumen was established according to the national guidelines [17]. The quality of the foamed bitumen was evaluated based on maximum expansion ratio, ERm, and half-life, H-1 [16]. The ERm was 25.3 at H-1 of 14.1 and at the optimal foaming water content of 2.5%.

The test specimens, compacted using a gyratory press to PN-EN 12697-31, had the following dimensions: D=100 mm and H=180 mm. To obey the maximum grain size requirement, the aggregate sieve size used was 22.4 mm. The curing period of the test specimens was 28 days at ambient temperature of 25°C±5°C and humidity of 40%±10%.
2.3. Inactive mineral filler

The inactive filler in the form of mineral dust was derived from the dedusting of dolomite aggregate in a quarry. Mineral dusts typically have gradation of less than 0.063 mm. Table 2 summarizes fundamental properties of the dolomite dust. Analysis of the grain distribution was performed with the use of laser diffraction.

Table 2. Fundamental properties of mineral dust

| Property                              | Symbol   | u. m | Dolomite dust (Dol) |
|---------------------------------------|----------|------|---------------------|
| Particle density                      | $\rho$   | g/cm$^3$ | 2.814               |
| Specific surface area                 | $P_w$    | cm$^2$/g | 26 729              |
| Void spaces, dry compacted dust       | AVR      | %    | 36.0                |
| Fines content                         | MBF      | -    | 1.5                 |
| pH                                    | pH       | -    | 8.1                 |
| Particle size distribution (µm)       |          | % passing by mass |
| 63.0                                  |          | 80.1 |
| 59.3                                  |          | 24.6 |
| 42.1                                  |          | 12.2 |
| 23.7                                  |          | 4.2  |
| 15.9                                  |          | 2.1  |

3. Test results

3.1. Complex modulus in the direct tension-compression test

The testing was performed at five temperatures (-7°C, 5°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 results were used to define complex modulus ($E^*$) and phase angle ($\phi$). The tests were carried out on cylindrical specimens under test conditions complying with EN 12697-26 Appendix D. Figure 2 summarizes the results for selected frequencies and temperatures.

It has to be noted that increased frequency in the initial phase of the test contributes to the lowest value of complex modulus of the FB-RCM with mineral dust. Similar relationship resulting from variable loading time was observed for RCM modified with bitumen emulsion and cement by [7, 11, 18]. Higher content of mineral dust in the recycled mix causes gradual decrease of complex modulus. This relation is associated with the fine structure of the recycled mix with foamed bitumen [21]. The complex moduli obtained were as follows: from 12 356 MPa to 3516 MPa for the FB-RCM+ 5% dolomite dust, from 10 836 to 2836 MPa for FB-RCM + 12.5% dolomite dust, and from 8004 MPa to 2264 MPa for FB-RCM + 20% dolomite dust. The highest $E^*$ was recorded at the temperature of -7°C and at the frequency of 0.1Hz, whereas the lowest values were recorded at 40°C and 20Hz.
Figure 2. Isotherms of complex modulus $E^*$ as a function of load frequency for the recycled cold mixes with foamed bitumen and inactive mineral filler a) 5% filler, b) 12.5% filler, c) 20% filler

To describe the effect of the inactive filler content on the distribution of elastic and viscous components, the viscous portion $E_2$ was represented as a function of the elastic portion $E_1$. The distribution of the complex modulus was possible owing to the determination of the phase angle ($\phi$). The complex modulus components were computed from equations (4) and (5). To confirm the time-temperature superposition principle, the complex modulus distribution is represented on the Cole-Cole plot (Fig. 3).
Figure 3. Cole-Cole diagram for the recycled cold mixes with foamed bitumen and inactive mineral filler

The relationship in Fig. 3 shows that the recycled base made with 20% mineral dust will undergo more elastic deformation than the bases made with 5% and 12.5% of mineral dust. Also, high complex moduli do not guarantee limiting the viscous component. The high percentage content of $E_2$ in the complex modulus translates into a lower durability of the recycled base layer. This phenomenon was observed when the filler was incrementally added. The highest complex modulus was obtained for the recycled base made with 5% of the mineral filler. However, this base layer has the highest portion of the viscous component, which will be reflected in the higher sensitivity of this layer to overloading and durability loss.

3.2. FB-RCM complex modulus master curves in terms of the mineral filler content

Complex modulus master curves for the recycled mixtures with foamed bitumen were constructed according to the content of inactive filler, using 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 ($\alpha$, $\beta$, $\gamma$, $\delta$ and $\lambda$). The goodness-of-fit of the sigmoidal function to the experimental values was evaluated using the coefficient of determination, $R^2$, and the mean normalized error, MNE, [20, 21]. The graphical representation of the master curves for the reduced frequency domain and for the reference temperature 25°C is shown in Fig. 4. The goodness-of-fit of the data to the sigmoidal function model is shown in Table 3.
The very high, close to 1.0, value of $R^2$ indicates very good fitting of the non-symmetrical sigmoidal function to the complex modulus data shifted by the $\alpha_T$ factor. Also, the MSE values are very low (MSE < 1.0%) thus, the difference between the measured and the model complex modulus values is very small, reaching a maximum of 1.00%. The maximum complex modulus ($E_{\text{max}}$) was determined at low temperature [22], which represents a perfectly elastic behaviour of the recycled base layer with foamed bitumen and mineral dust.

The master curves developed for the recycled base layers with foamed bitumen and mineral dust show similar behaviour under a long-term exposure to stress, regardless of the amount of dust used in the recycled base (minimum time rate – creep). A different behaviour is observed when the base layer is exposed to a short-term stress (frequency above 10Hz). The increased content of mineral dust affects the complex modulus by reducing its value.

4. Conclusions
Analysis of the test results leads to the following conclusions:

- The use of dolomite mineral dust as an inactive filler reduces the complex modulus of recycled bases. An increase in the mineral filler content changes the structure of the base layer in that the voids between the coarse fraction grains are filled with fines, and limits the mineral mix inner friction angle.
- The use of dolomite mineral dust in the recycled base layer with foamed bitumen at a 20% content reduces the complex modulus by about 40% relative to the recycled base layer made with a 5% content, regardless of test temperature or loading time.
- The highest complex modulus ($E^* = 8533 \text{MPa}$) at the reference temperature of 25°C at the loading time of 10Hz was recorded for the recycled mixture with foamed bitumen and 5%
mineral dust (FB-RCM – 5%). The lowest modulus was recorded for the FB-RCM – 20%, 
$E^* = 4977$ MPa.

The use of mineral dust in the FB-RCM has a strong effect on elastic response of the base 
layer. Increased content of mineral dust in the recycled mixture causes a decrease in complex 
modulus ($E^*$), but also limits the viscous component ($E_2$) of the complex modulus.

The results discussed in this paper indicate that mineral dust content of 20% can be used as an 
inactive filler in the recycled cold base layer with foamed bitumen without any negative influence on 
the mechanical parameters of the recycled layer. This application of the waste material (mineral dust 
from recycling) will considerably reduce the degradation of natural resources by limiting the 
extration of ravelling aggregate.

Acknowledgment(s)
The study results were developed within the framework of the project entitled “The use of recycled 
materils” under “RID” co-funded by the National Centre for Research and Development and the 
General Directorate for National Roads and Motorways in Poland.

References

[1] M. Iwański, A. Chomicz-Kowalska, “Evaluation of the pavement performance”, Bulletin of the 
Polish Academy of Science: Technical Science, vol. 63, no. 1, pp. 97–105, 2015. 
doi.org/10.1515/bpasts-2015-0011

[2] M. Iwański, A. Chomicz-Kowalska, “Evaluation of the effect of using foamed bitumen and 
bitumen emulsion in cold recycling technology”, 3rd International Conference on 
Transportation Infrastructure (ICTI), April 22-25, 2014, Pisa, Italy pp. 69-76, Proceedings 
Paper WOS:000342770000008

[3] M. Iwański, P. Buczyński, G. Mazurek, „The use of gabbro dust in the cold recycling of asphalt 
paving mixes with foamed bitumen”, Bulletin of the Polish Academy of Sciences Technical 
Sciences, vol. 64(4), pp. 763–773, 2016. https://doi.org/10.1515/bpasts-2016-0085

[4] F. Cardone, F. Frigio, G. Ferrotti, F. Canestrari, „ Influence of mineral fillers on the rheological 
response of polymer-modified bitumens and mastics”, Journal of Traffic and Transportation 
Engineering (English Edition), vol. 2(6), pp. 373-381, 2015. 
http://dx.doi.org/10.1016/j.jtte.2015.06.003

[5] A. I. Nassar, M. K. Mohammed, N. Thom, T. Parry, „Mechanical, durability and microstructure 
properties of Cold Asphalt Emulsion Mixtures with different types of filler” Construction and Building Materials, vol. 114, pp. 352-363, 2016. 
http://dx.doi.org/10.1016/j.conbuildmat.2016.03.112

[6] A. Modarres, P. Ayar, „Coal waste application in recycled asphalt mixtures with bitumen 
emulsion” Journal of Cleaner Production, vol. 83, pp. 263-272, 2014. 
http://dx.doi.org/10.1016/j.jclepro.2014.07.082

[7] C. Godenzoni, A. Graziani, D. Perraton, „Complex modulus characterisation of cold recycled 
mixtures with foamed bitumen and different contents of reclaimed asphalt”, Road Materials and Pavement Design, vol. 18, no. 1, 130–150, 2017. 
http://dx.doi.org/10.1080/14680629.2016.1142467

[8] P. Radziszewski, K. Kowalski, J. Król, M. Sarnowski, J. Piłat, „Quality assessment of bituminous binders based on the viscoelastic properties: polish experience”, Journal of Civil Engineering and Management, vol. 1(20), pp. 111–120, 2014. 
doi.org/10.3846/13923730.2013.843586

[9] Y. R. Kim, “Modeling of asphalt concrete” ASCE Press, pp. 460, 2008.

[10] R. Timothy, Clyne, Xinjun Li, O. Mihai, Marasteanu, L. E. Skok, “Dynamic and Resilient 
Modulus of Mn/DOT Asphalt Mixtures”, Minnesota Department of transportation 
Minnesota, Raport no. MN/RC-2003-09, pp. 67, 2003.

[11] D. Sybilski, J. Kukielka, „The rheological properties of mineral-cement-emulsion (MCE) the
master curve leading”. I Kongres Drogowy Warszawa (2006). (in Polish)

[12] M. Jarczewski, J. Judycki, P. Jaskuła, “Viscoelastic modelling of asphalt mixtures under long time loading using master curves and its limitations”, Drogownictwo, vol. 10, pp. 336 – 340, 2015. (in Polish)

[13] G.M. Rowe, M. J. Sharrock, “Alternate Shift Factor Relationship for Describing the Temperature Dependency of the Visco-Elastic Behaviour of Asphalt Materials”, Transportation Research Record, vol. 2207, s. 125-135, 2011

[14] H. M. Naguyer, S. Pouget, H. Di Benedetto, C. Sauzeat, “Time-temperature superposition principle for bituminous mixtures” European Journal of Environmental and Civil Engineering, vol. 13 no. 9, pp. 1095-1107, 2009. doi: 10.1080/19648189.2009.9693176

[15] M. L. Williams, R.F. Landel, and J.D. Ferry, “The Temperature Dependence of Relaxation Mechanisms in Amorphous Polymers and Other Glass-forming Liquids”, Journal of the American Chemical Society, vol. 77, pp. 3701-3707, 1955.

[16] Wirtgen Cold Recycling Technology (3re ed.), Windhagen: Wirtgen GmbH, 2010

[17] OST D-04.10.01a, “The detailed technical specifications: The road base of the mineral-cement with foamed bitumen mixture made in cold recycling technology”. Warsaw, GDDKiA, 2013. (in Polish)

[18] B. Dołżycki, M. Jaczewski, C. Szydłowski, “The influence of binding agent on stiffness of mineral-cement-emulsion mixtures” Procedia Engineering, vol. 172, pp. 239-246, 2017. doi.org/10.1016/j.proeng.2017.02.103

[19] B. Staďańczyk, P. Mieczkowski, Mineral asphalt mixtures. Performing and research. WKŁ, pp. 322, 2009. (in Polish)

[20] Yusoff, N. I Md.; Mounier D.; Marc-Stéphane G.; Hainin M. R.; Airey G. D.; Di Benedetto, H. “Modelling the rheological properties of bituminous binders using the 2S2P1D Model”, Construction and Building Materials vol. 38, pp. 395–406. 2013. doi.org/10.1016/j.conbuildmat.2012.08.038

[21] G. Mazurek, M. Iwański, “Relaxation Modulus of SMA with Polymer Modified and Highly Polymer Modified Bitumen” Procedia Engineering, vol. 172, pp. 731-738, 2017. doi.org/10.1016/j.proeng.2017.02.093

[22] M. Jarczewski, J. Judycki, “Effects of deviations from thermo-rheologically simple behavior of asphalt mixes in creep on developing of master curves of their stiffness modulus”, The 9th International Conference “Environmental Engineering”, 22–23 May 2014, Vilnius, Lithuania, doi.org/10.3846/enviro.2014.157