Utilization of aluminium dross as asphalt filler

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Abstract
Asphalt industry finds itself battling ongoing economic difficulties and an urge to achieve a more sustainable development and growth. It means constant searching is needed for alternative materials and possibilities to use recycled and processed waste materials in asphalt mixes as long as an expected level of performance and durability is provided.

Aluminium dross is a recyclable by-product of the casting process of melted aluminium. In this study an attempt was made to reveal the potentials of using aluminium dross as filler for asphalt wearing course mixes. During the research, filler fractions (d<0.063 mm) were prepared by milling and microstructural tests were conducted both on the alternative and control filler for a better understanding of the materials and their composition. The effect of replacing limestone filler with aluminium dross filler on the performance of asphalt mixes was analysed by performance-based and performance related asphalt mechanical tests according to common standards. In the paper, the properties of fillers and various mechanical test results are presented and by interpreting the tests and results final conclusions are presented regarding the use of aluminium dross as filler in asphalt mixes.

Keywords: asphalt, aluminium dross, filler, rheology, stiffness

1. Introduction
Handling, storage and wrecking of several waste material (construction and demolition waste, oil-drill cuttings, industrial by-products, etc.) is produced year by year which causes significant problems all over the world. To avoid these materials to damage the environment, they have to be recycled. Besides storing them on landfills, another way is the industrial utilization which may forward the reduction of yielding the available raw materials. A possible field of application of waste is building industry.

Utilization of different waste materials in concrete has been the subject of several research work. Aliabdo et al. [1] have done a comparative research work on the utilization of crushed clay brick in concrete. Kim [2] dealt with the application of waste concrete powder in self-consolidating concrete and its attribute characteristics. An extensive literature [3, 4, 5, 6, 7] is dealing with the application of concrete waste as aggregate in asphalt mixtures.

Utilization of different types of slags (basic oxygen furnace slag, steel slag) [8, 9, 10, 11,12] produced during various metallurgical processes as asphalt aggregate also showed good results. Based on the above mentioned research, it can be stated that mixtures made with slags have nearly the same performance as mixtures made with regular aggregates. Besides, slag – among others – improves resistance to plastic deformation and fatigue of asphalt pavements and it also decreases failure taking place due to sensitivity against

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moisture. Friction characteristics between vehicles and pavements can be improved by application of slag aggregates in wearing course and failure phenomena (rutting, cracking) can also be decreased in a great extent. By using slags, an improvement can be reached in the values of indirect tensile test, creep modulus and stripping.

Automotive casting was strengthened over the past decade in Hungary. A large amount of aluminium alloy is used during the aluminium melting process, significant amount of dross is formed due to oxidation process, which is a recyclable by-product. This waste material is produced during melting of alumina scrap; its general composition is: 15-30% Al₂O₃, 30-55% NaCl, 15-30% KCl and 5-7% of metallic aluminium and other impurities (carbides, nitriles, sulphides, phosphides). Several researches were made in the scope of recycling such drosses, mainly in the field of concrete technology [13, 14, 15]. These results showed that strength of the concrete made by substituting cement by 15% aluminium dross approached strength values of conventional concrete. Besides, alumina slag can be used for producing paver blocks and refractory blocks and can also be used where conventional concrete is applied.

2. Experimental

2.1 Sample preparation

Two types of fillers were used in this research. One was the aluminium dross, the other was limestone which was used as a reference material. The required particle size of fillers (d<0.063 mm) was obtained by sieving. Samples were then dried to weight constancy.

During the research the following tests were done on the fillers to reveal their properties. Mineralogical composition of samples was determined by X-ray powder diffraction (XRD) on a Rigaku MiniFlex II diffractometer. Particle size distribution (PSD) of the sieved material was measured by a Malvern Mastersizer X laser diffraction particle size analyzer in dry mode using air as dispersing media. Specific gravity of fillers was determined by pycnometer method according to MSZ EN 1097-7 standard. Hydrophilic coefficient was also determined by sedimentation method. Rigden Void of fillers were determined according to MSZ EN 1097-4 standard. For morphological tests Carl Zeiss EVO MA10 scanning electron microscope (SEM) was used. Specific surface area (SSA) of the powders (by BET-method) was determined by Micromeritics TriStar 3000 instrument.

2.2 Asphalt mix tests

The use of secondary and waste materials, slag and dross materials, to substitute certain parts of asphalt mixes is not a novelty in the past decades with a constant pursuit for a better sustainable environment, and asphalt industry [16, 17, 18]. Research has shown that the use, instead of disposal, of such by-products may be a promising way, amongst others, having at least no negative effects on performance while solving part of some issues related to the accessibility of virgin materials [19, 20].

In order to test performance of asphalt mixes having non-standard composition everyday methods as indirect tensile strength, wheel tracking, water sensitivity or fatigue may be inadequate, and require high volumes of the experimental material [21]. Furthermore as bituminous mixes have a rather complex, time and temperature behaviour, being a challenge to model and understand, multiple types of tests are required to assess performance at multiple testing conditions [22, 23, 24].

To obtain an overlook on the expected performance effect of replacing (part) of the pure limestone filler with dross simple performance test (SPT) as developed by NCHRP Project 9-19 was made on multiple temperatures, was made using a frequency sweep between 0.1 Hz – 25 Hz, due to the various possibilities to assess the data despite the simplicity of the test and the specimens [22].

To perform the preliminary tests three asphalt mix types have been made, one reference mix with 100% limestone as filler (Mix B), one mix made with 100% aluminium dross (Mix A), and one mix using 50% aluminium dross as filler (Mix C).

2.3. Mix composition

In order to assess the effect of the filler composition itself, asphalt wearing course mixes with nominal aggregate size of 11 mm (AC11) were made with the same aggregate composition, and the same binder type and content. Coarse aggregates have been washed and the dust loss has been compensated with fillers of the given three compositions. Thus, the total filler added was the filler by mix design and the mass of dust. Fig. 1 shows the gradation of the asphalt mixes.

Mixes were made using B50/70 standard bitumen with a content of 4.6%. Two gyratory specimens have been made for each mix, achieving a void of 4.7-4.8%, and the selected performance test has been conducted on all specimens.

2.4. Performance tests

Simple Performance Tests (SPT) were performed at temperatures between 0-40°C and frequencies between 0.1-25 Hz. Followed by proper conditioning intervals and resting times. Moduli and phase angle have been measured.

3. Results and discussion

3.1. Filler tests

Table 1 shows the results, which were obtained on fillers.
Table 1. Test results of fillers

| Parameter                          | Unit  | Limestone | Dross |
|-----------------------------------|-------|-----------|-------|
| Specific gravity, \( \rho \)      | g/cm³ | 2.795     | 2.904 |
| Average particle size, \( d_{50} \) | \( \mu \)m | 13.77     | 24.96 |
| Specific surface area, SSA        | m²/g  | 1.55      | 1.03  |
| Rigden Void, RV                   | vol%  | 47.2      | 45.7  |
| Hydrophilic coefficient, \( \eta \) |       | 0.75      | 0.67  |

Mineral composition:
- calcite (\( \text{CaCO}_3 \))
- quartz (\( \text{SiO}_2 \))
- dolomite (\( \text{CaMg(CO}_3)_2 \))
- muscovite 2M1 (\( \text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2 \))
- halite (\( \text{NaCl} \))
- sylvite (\( \text{KCl} \))
- potassium aluminium oxide (\( \text{K}_1.5\text{Al}_{11}\text{O}_{17.25} \))
- corundum (\( \text{Al}_2\text{O}_3 \))

According to X-ray diffraction it can be stated that limestone build up mainly of calcite. Besides, this filler also contains dolomite, quartz and muscovite in small quantities. The main phases of dross are corundum and potassium aluminium oxide in a high quantities. Some salts, which are important components during the melting process of aluminium, like halite and sylvite have also identified during XRD tests.

The results of particle size analysis show that average particle size of limestone is smaller than aluminium dross (\( d_{50 \text{ limestone}}: 13.77 \mu \text{m} \) vs. \( d_{50 \text{ dross}}: 24.96 \mu \text{m} \)), so limestone contains much more fines than the other filler. Because of the higher quantities of fines in limestone, its specific surface is also higher. Electron micrographs (Fig. 2), taken in different magnifications also demonstrate the particle size distribution and the surficial features of fillers.

It is well observable that the surface of coarser particles of limestone is quite smooth and small calcite particles are stucked on it. Traces of open pores are not identified on the angular particles. In contrast, the surface of dross is much varied. Due to the mineral composition, smooth angular particles, plately parts and whisker-like surfaces can also be observed.

According to hydrophilic coefficient tests it can be stated that both fillers are hydrophobic which is favourable in asphalt technology.

3.2. Performance test results

3.2.1. Master curves

Master curves of viscoelastic materials are used to give a comprehensive assessment of the material stiffness for various temperatures and loading frequencies [25, 26]. Master curves can be constructed using the temperature-frequency superposition principle. Sigmoid model was used to construct the master curves of the specimens, based on Eq. (1).

$$\log |E'| = \delta + \frac{\alpha}{1+e^{(y-\gamma)/\delta_{\text{red}}}}$$  \( (1) \)

Where is the stiffness [MPa], \( \alpha, \beta, y, \delta \) constants, \( f_{\text{red}} \) reduced frequency. Required constant parameters have been iterated.
using the least squares method. Sigmoid functions obtained and the measured values are shown on Fig. 3.

Fig. 3. Master curves of the tested specimens

As the master curves depict the mixes are stiffened by replacing limestone filler with aluminium dross. It is interesting to note that Mix A (100% dross) is significantly stiffer throughout the frequency range but Mix C (50% dross) is more similar to mix B (100% limestone) on high temperatures, and more like between the lesser two at high temperatures.

3.2.2. Black diagrams

Black diagrams give a good overlook on the viscoelastic properties of viscous materials such as bitumen and asphalt mixes [27, 28]. Plotting the measured complex moduli $E^*$ against the phase angle $\phi$ for each measurement in the case of all three mixes results in the Black diagram shown on Fig. 4. The tool developed to assess the rheological properties of binders may be, with limitations used for asphalt mixes as well, as viscoelastic materials.

Fig. 4. Black diagrams of the tested specimens

The diagram enables the preliminary assessment of stiffness and non-linear behaviour. As the diagram shows, Mix A has higher stiffness values at given phase angles than the other two mixes, being similar at high phase angles, indicating that Mix A would perform more stiff at high temperatures and/or high frequencies. Notice that the ‘return’ of the curve is missing in all cases, which is normal taken into account the fact that asphalt mixes have been measured.

3.2.3. Cole-Cole diagrams

By using the known function of the complex modulus $E^*$ as shown on Eq. (2), storage modulus and loss modulus $E_1$ and $E_2$, respectively, can be calculated according to Eq. (3) and Eq. (4).

$$E^* = \frac{\sigma_0}{\epsilon_0} = \sqrt{E_1^2 + E_2^2}$$

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

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

Where $E^*$ is the complex modulus, $\sigma_0$ is the maximum stress, $\epsilon_0$ is the maximum strain, $\phi$ is the phase angle [°], $E_1$ is the storage modulus [MPa], and $E_2$ the loss modulus [MPa].

Storage modulus represents the part of the deforming stress that is stored in the deformed material and at the end of the deformation is used to cease the deformation. Loss modulus is the part of the stress that is usually lost during a deformation in heat form. Plotting the loss modulus against the storage modulus results the Cole-Cole diagram, as shown on Fig. 5.

Fig. 5. Cole-Cole diagrams of the tested specimens

The left part of the diagram indicates the mix behaviour at high temperatures, whereas the right hand side indicates the behaviour at low temperatures. Angle of given vectors pointing from zero to an arbitrary point on the graphs is called the loss angle.

The farther the intersections on the right side are, stiffer the materials are at low temperatures. As seen, the more the ratio of dross in the filler is, the stiffer the mix becomes in all temperature ranges compared to the limestone only filler. Furthermore, the stiffening effect is relatively higher on high temperatures than on low temperatures.

4. Conclusions

Simple Performance Test results have been conducted to preliminarily assess the effect of replacing limestone filler with
dross on the potential performance of the resulting asphalt mix. An interesting effect which requires further research is that the stiffening effect of the limestone is higher on low temperatures (low frequencies), and lower on low temperatures.

As seen, replacing the limestone filler with dross not only stiffens the asphalt mix, but increases the phase angle as well. However, replacing only 50%, i.e. only a part of the filler with dross may be a good direction for further research.

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