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Investigation of thermal properties of raw materials of asphalt mixtures

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Abstract. Asphalt mixtures are composite materials, which are made of different grades of mineral aggregates and bitumen. During the mixing process mineral materials were blended with bitumen at relatively high temperature (~200 °C). As the binding process come off in these higher temperature range, thermal properties of asphaltic materials are important.

The aim of this project is to reveal the thermal properties of raw materials. During our research two types of mineral aggregates were tested (limestone and dolomite) by different methods. Differential thermal analysis, thermal expansion and thermal conductivity were investigated at technologically important temperatures.The results showed that the structure of mineral materials did not change at elevated temperatures, expansion of samples was neglible, while thermal conductivity changed by temperature.

1. Introduction

Asphalt mixtures are the most used pavement materials all over the world. The mixtures consist of mineral aggregates and bitumen as binding material [1-5]. One of the most important components of asphalt are fillers (*d<0.063mm*) which are the finest part in mineral aggregate. Mixed together with the binder a so-called asphalt mastic is formed.The contact between fillers and bitumen is not exactly known, until today.

During the lifetime of asphalt mixtures and the compacted pavements heat will play a significant role [6]. Raw mineral aggregates are stored in cold aggregate bins. At the beginning of the production aggregates are heated up in the drying drum in order to evaporate the adsorbed water from their surface. Furthermore, raw materials need to reach the mixing temperature. Then aggregates are transferredby the hot elevator to the hot aggregate bin, then to the mixer where hot bitumen is also added from the bitumen tank.After mixing, asphalt mixtures are loaded into heated trucks and transported to the field to be paved. Compacting process and traffic temperature both affect the properties of asphalt pavements.

The effect of temperature on the properties of mineral materials is not well known till today. Only a few research dealt with this area [7, 8]. Devecseri [8] investigated the changes of physical properties (*weight change, particle size change and colour change*) of different asphalt aggregates (*effusive and sedimentary rocks with the fraction of11/16 mm*) by elevated temperatures (*240°C and 480°C*). She found that high temperatures (480 °C) cause mineralogical, physical and colour changes in aggregates. She also stated that during heating aggregates behave contrary to each other, therefore there is no exact relation between their thermophysical and mechanical properties.

Considering the above mentioned, the aims of this research is to investigate the thermal properties of raw materials (*mineral fillers*) for a better understanding of the cohesion between fillers and bitumen.

2. Materials and methodology

2.1. Sample preparation
Limestone (*Mexikóvölgy*) and dolomite (*Pilisvörösvár*)fillers were tested during the research. Both minerals are widely used in Hungary as asphalt fillers.

Samples were prepared for further tests using standard sieves. Fractions of mineral materials that are smaller than a given particle size (*d<0.063 mm*) were created. After sieving,fillerswere dried to weight-constancy then they were hermetically sealed in containersafter cooling down. The reason of this was to avoid any moisture uptake.

2.2. Testing procedures
Determination of particle size distribution of fillers were doneby the use of Horiba LA-950V2 instrument.Wet method, sodium-pyrophosphate addition and ultrasonic treatment was used.

Morphological tests were carried out on Carl Zeiss EVO MA10 scanning electron microscope (SEM).Electron micrographs were taken in different magnifications. The aim of this test was to observe morphology and geometrical features of fillers.

Differential Thermal Analysis were done on MOM Derivatograph-C type instrument in temperature ranges of 50°C–1000°C. Heating rate was 5°C/ min. Weightchange of fillers and theirendothermic or exothermic reactions can be revealedwith this method.

With differential scanning calorimetry enthalpy changes and heat capacity of fillers were determined by Mettler Toledo DSC 823 E type instrument. Heating and cooling rates of 10°C/min were used in ranges of 30°C–200°C. Mineral fillers were placed in a 40 µl aluminium crucible.

In order to investigate the change in length of fillers a heating microscope (*manufactured by Camar Elettronica*) and Linseis L75 laser dilatometer were used. Heating microscope was used to measure optically the change of height of compacted specimens. Compacted samples (with a diameter of 2 mm and a height of 4 mm) were heated up to 200 °C with a heating rate of 5 °C/min. Both fillers could be compacted for this test.

Relative length change was determined during heating and cooling runs with the use of laser dilatometer. For this test a sample with a length of 20 mm and a diameter of 7 mm was needed. Only limestone filler was tested, because of compaction difficulties.

3. Results and discussion

3.1. Test results of mineral fillers
Table 1. contains all the test results performed on mineral fillers.

Table 1. Main properties of fillers

Material property	**unit**	**Limestone** *d<0.063 mm*	**Dolomite** *d<0.063 mm*
Mineral composition	wt%	100 % $CaCO_3$	100 % $MgCa(CO_3)_2$
Bulk density	$g \cdot cm^{-3}$	2.717	2.842
BET specific surface area	$m^2 \cdot g^{-1}$	1.02	0.38
Hydrophilic coefficient	-	0.76	0.89

Figure 1. shows the particles size distribution curves (*cumulative passing and hystogram*) of fillers. According to the results, it was found that these fillers can be characterized as polydisperse systems and they contain fine and coarse particles in different quantity by fraction. Mean particle diameter (d_{50}) of limestone is 16.82 μm, and the mean particle size of dolomite is 39.84μm. 20 percent of limestone particles are under 10 microns, while in case of dolomite the amount of these fine particles are under 2 percent.

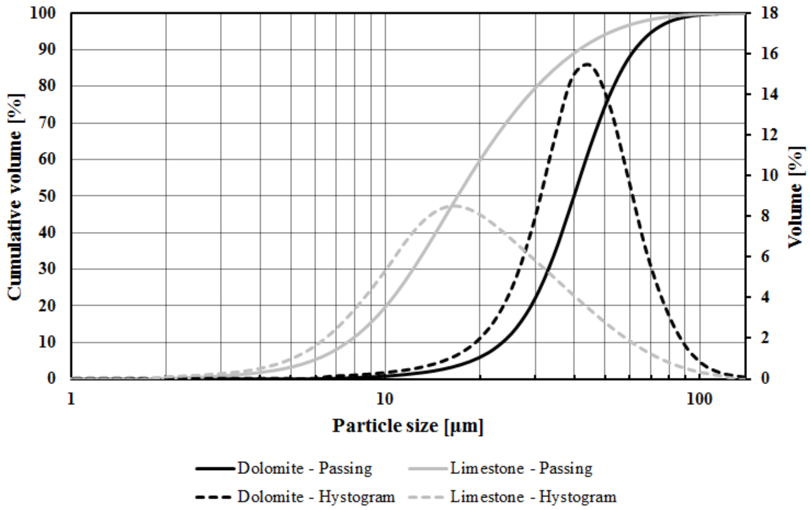
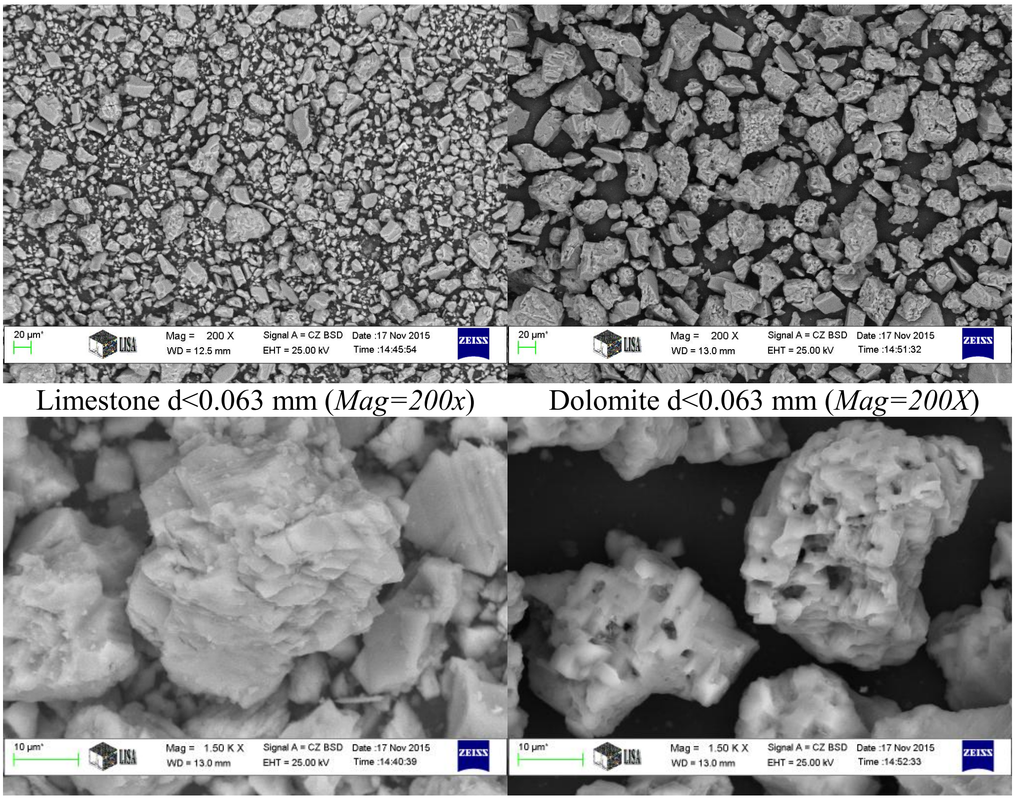


Figure1. Particle size distribution of fillers

Fig. 2. shows the microstructures of fillers (*magnification=200X and 1500X*).



Limestone d<0.063 mm (*Mag=200x*) Dolomite d<0.063 mm (*Mag=200X*)

Limestone d<0.063 mm (*Mag=1500x*) Dolomite d<0.063 mm (*Mag=1500x*)

Figure 2. Scanning electron micrographs of fillers

The SEM micrographs confirm the results of particle size distribution tests. Comparing the micrographs, which were taken at a magnification of 200X, it can be observed that limestone filler is rich in fine particles while dolomite does not contain fines. This is the reason of the low BET specific surface of dolomite (*Table 1.*). Higher magnifications show well the morphology and the surfaces of particles. Limestone filler looks solid, on which submicronal particles are sticked. The shape of grains are various, mainly polygonal, because of the comminution processes. Dolomite particles are also polygonal and more porous than limestone. Fig. 3. shows results of differential thermal analysis.

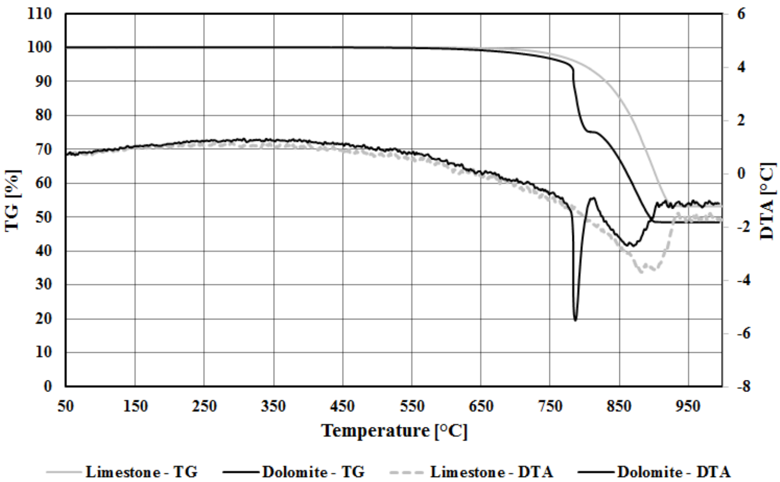


Figure 3. TG and DTA curves of fillers

TG (*thermogravimetric*) curves represent the weight change of fillers, while DTA (*differential thermal analytical*) curves show the temperature changes occurring within the materials.Limestone filler has a slow weight lossin 670–900 °C temperature range. This is due to the decarbonisation of $CaCO_3$. During this reaction CaO is formed while CO_2 is released. This phenomenon is an endothermic reaction, as the DTA curve shows a negative peak. TG curve of dolomite shows also a thermal decomposition, but in this case this process is a two-stage reaction. The first endothermic reaction belongs to the decomposition of dolomite, while the second endothermic reaction belongs to the decomposition of $CaCO_3$. Up to 200°C, fillers behave as inert materials (*in the viewpoint of asphalt technology*) because no reactions occur in this range. In Fig. 4. results of DSC analysis are summarized.

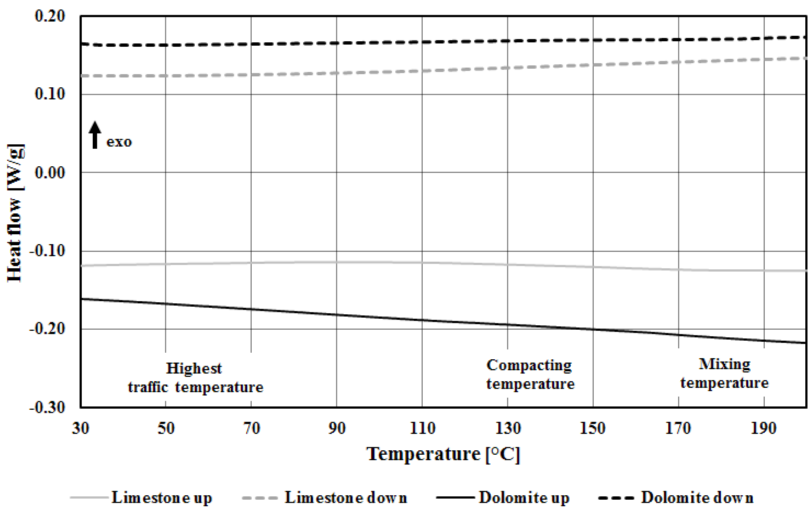


Figure 4. DSC curves of fillers

Heat flow curves recorded in the heating and cooling runs are shown. It can be concluded that there is no reaction, no significant enthalpy change occurs during the runs, no peaks are observed. As the diagram shows, no reaction occurred at the important temperatures.

Specific heat capacity can also be determined from DSC results, as Fig. 5. shows. As it can be seen dolomite has a higher specific heat capacity in the tested temperature ranges (*30°C–200°C*) than limestone. Specific heat capacity of fillers increased almost linearly in the tested ranges.

Heating microscope was used to measure the height change of compacted specimens during heating and it showed no change (*Fig. 6.*). The instrument is able to detect the silhouette of a lighted specimen by a digital camera. By image analysis the height change of the sample can be determined. In our case the height of the fillers was not changed during the test.

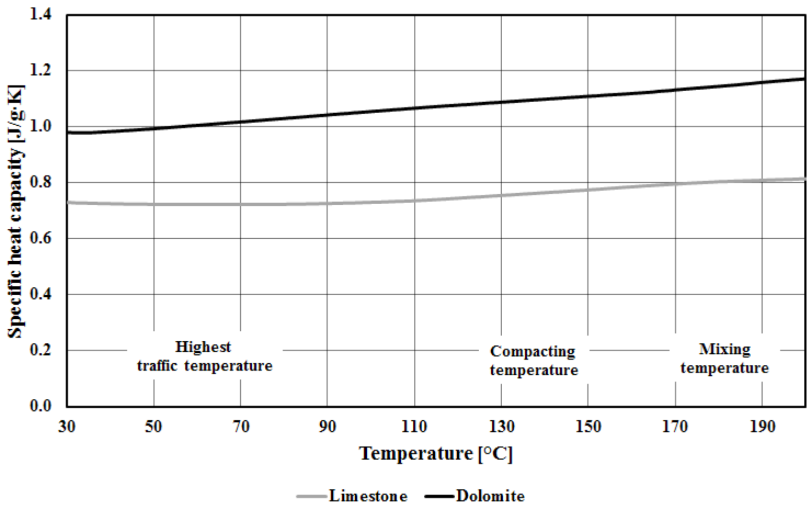


Figure 5. Specific heat capacity curves of fillers

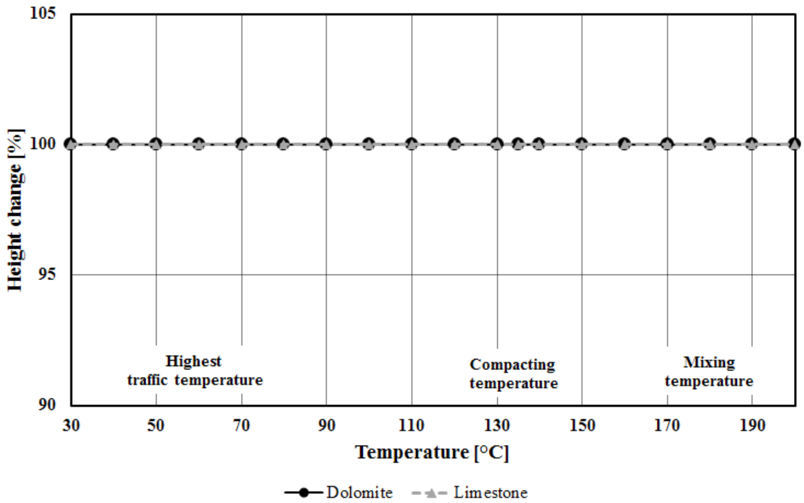


Figure 6. Height change of compacted fillers during heating

Dilatometric test by a precise laser dilatometer was also obtained. This method was used only on limestone filler, because dolomite was not able to compact. The reason of this is the particle size distribution of the mineral material (*Fig.1. and Fig. 2.*).

In Fig. 7. the relative length change curves (*heating and cooling runs*) are shown. At the maximum testing temperature (T_{max}=*200 °C*) the relative length change of the sample reached 0.15%. After cooling this change remained at 0.04 %, which is not a significant value. According to the dilatometric test and heating microscopic tests it can be concluded that neither filler change its dimensions during the heating process.

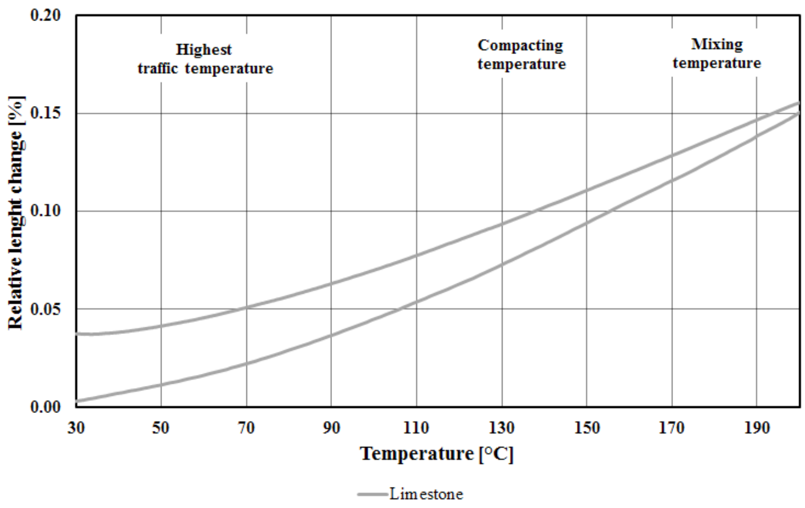


Figure 7. Relative length change oflimestone filler

Fig. 8. shows the results of thermal conductivity measurements. 4 testing temperatures were used (*20°C=room temperature, 60°C=highest traffic temperature, 135°C=compacting temperature and 180°C =asphalt mixing temperature*). Thermal conductivity values are increased by the increase of temperature. Dolomite has slightly higher conductivity values than limestone, but this difference is not significant (~0.03 W/m·K), except at room temperature. At this temperature the difference is almost two times higher.

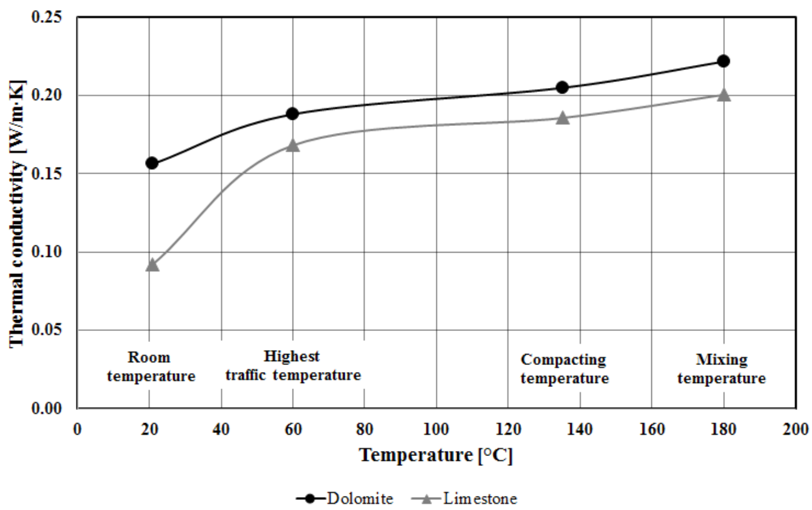


Figure 8. Thermal conductivity of fillers

4. Conclusions

During this research work two types of fine particle mineral materials were tested as asphalt fillers. Examinations of thermal properties of fillers were made by different techniques.

According to the particle size distribution, these mineral fillers can be characterized as polydisperse systems and they contain fine and coarse particles in different quantity by fraction.

The Authors have also observed the morphological features of the mineral materials. The morphology of fillers is various;dolomite has higher porosity in the particles, but does not contain much fine particles.

The samples were tested to reveal the thermal properties.According to the results, heat during the technological processes do not influence the properties of fillers (*no reactions, no change in length*). Difference was found only in specific heat capacity and in thermal conductivity. Dolomite has a higher specific heat capacity and thermal conductivity than limestone.

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