JMEPEG (2009) 18:282–286
DOI: 10.1007/s11665-008-9293-6

Determination of the Test Methods Sensitive to Free Mica Content in Aggregate Fine Fractions

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(Submitted June 4, 2008; in revised form July 12, 2008)

The detrimental influence of mica rich aggregates on structural deterioration of road pavement has been discussed in the literature for over a half century. This negative effect is of great importance especially for regions with crystalline, mica-rich bedrock and temperate, subarctic climates. Recent investigations reveal that elevated fractions of free mica particles in unbound granular materials, used in road constructions, greatly reduce bearing capacity and influence the hydraulic behavior of the road structure. Despite the awareness of mica's potential harmful effect, the absence of properly adapted analytical methods is noticeable. The scope of the current study was to test two possible analytical methods which, are susceptible to mica content and could be used as an indicating technique for quantitative determination of free mica particles in unbound granular materials. Two standard methods: Sand equivalent test and methylene blue (MB) test were assumed as the most sensitive to mica presence. The rock samples used in the tests are representative for the common crushed rock aggregates for construction purposes with different contents of mica. Both methods showed susceptibility to mica content and gave strong correlation in terms of mica content. The result of the MB test can be explained by the schistose structure of mica particles and the ability of mica to absorb liquids. Another important explanation is the increased reaction surface of mica particles, which leads to an increase in the total reaction surface of the sample. The receptiveness of the sand equivalent test to mica content could be caused by the ability of mica to stay in suspension due to its flake-shaped grains.

Keywords fine aggregates, free mica, methylene blue test, moisture damage, quality of fines, sand equivalent test

1. Introduction

A high content of free mica particles in the fine aggregates, in both bound and unbound applications, will negatively influence the stability of road construction when exposed to water or moisture. The impact of such mica-water interaction can greatly reduce bearing capacity and influence the hydraulic behavior of the road structure (Ref 1, 2). This detrimental effect is of great importance especially for regions with crystalline, mica rich bedrock and temperate, subarctic climates (Ref 3, 4).

Mica minerals have very distinctive characteristics that are important when considering their effect on aggregates and bituminous materials:

1. The ability to form a flat, microscopic to submicroscopic-sized crystals explains a large specific surface area of the mica particles compared with quarts and feldspar ones. Even relatively small amounts of free mica particles in fine fraction can have a significant effect on aggregate or bituminous mix properties (Ref 5).
2. Water absorption or water loss can cause unexpected aggregate mass loss or gain. This may affect the calibration process for the ignition test used to determine the composition of the asphalt mix (Ref 5).
3. When mixed with limited amounts of water, the mica particles can become plastic. This phenomenon can have serious consequences on the performance of unbound layers within the highway structure (Ref 5).
4. The mica particles that absorbed water may expand as the water fills the spaces between the stacked silicate layers. The repetitive wetting/drying processes can affect the aggregate/bitumen adhesion. Expansion of the aggregate particles could cause the asphalt material to swell affecting its stiffness and fatigue characteristics (Ref 5).

The negative influence of mica rich aggregates on structural deterioration of road constructions has been discussed in the literature for over a half century. Despite the awareness of mica's potential harmful effect, the absence of properly adapted analytical methods is noticeable (Ref 6). Modern test methods for aggregate properties, related to moisture damage, generally fall into two categories: tests that evaluate the surface properties of the aggregate related to the adhesion of the binder to the aggregate and tests to identify clay-like fines (Ref 7). None of those takes into consideration the mineral composition of plastic fines in particular.

The scope of current study was to test two possible analytical methods which are susceptible to mica content and could be used as an indicating technique for the determination of the quantity of free mica particles in unbound granular materials. Another practical implementation is the consideration of the possible presence of mica materials as the

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influencing factor on the results of the tested methods. The overall goal of this research is to improve the quality control of the aggregates.

Micas have a phyllosilicate structure forming flat pseudo-hexagonal sheets. Therefore, the sand equivalent test and the methylene blue (MB) test were assumed to be the most sensitive to mica presence. Both methods were developed to determine the amount of clay and dust in the fine aggregate, which was believed to be detrimental to the performance of hot mix asphalt (HMA) mixtures (Ref 7, 8). These tests have advantages that they are quick to perform, require very simple and cheap equipment, and can be used with minimal training or experience (Ref 7).

Six rock samples (fine aggregates with a wide range of mineralogical compositions) used in the tests are representative of the common crushed rock aggregates for construction purposes with different contents of mica. The samples were dried and sieved. Grain size distribution for fractions 0-2.0 mm was achieved according to the Swedish standard for base-course aggregates (ATB ROAD—Swedish Road Administration technical specifications for roads, 2005) (Ref 9).

2. Materials and Methods

2.1 Materials

The experimental design has been based on narrow grain size fractions derived from crushed rock aggregates. Six aggregates were chosen to give a wide range of mineralogical compositions. The main material variable of interest has been the amount of free mica grains in the crushed rock aggregate. Thus, each of the grain size fractions 0.125-0.25, 0.25-0.5, and 0.5-1.0 mm were analyzed to ascertain mica content. Mica content has been determined as particle percent of the each grain size fraction. While performing the experiment only one fraction (0.125-0.25 mm) was used for comparison of mica content between the selected samples. This simplification can be clarified by the peak enrichment of mica, which is significant for this fraction size (Ref 10, 11).

Mica content of the collected grain size fractions was determined using a polarizing microscope and point counting technique according to the method RILEM AAR-1, Technique 2 (Ref 10, 11). Thin sections where prepared as grain mounts (Ref 12) from 2 to 4 g of the narrow aggregate for each collected grain fraction. The content of free mica for narrow grain size fractions is presented in the Table 1.

The materials used in the tests are all commercially available crushed rock aggregates from material producers throughout Sweden. Material 6, with the lowest mica content, was used as a reference material.

2.2 Sample Preparation

Representative materials of fine aggregates were dried and screened through 2.0 mm sieve (Swedish Standard SS-EN 933-1) (Ref 13) into following grain size fractions: <0.063, 0.063-0.125, 0.125-0.25, 0.25-0.5, 0.5-1.0, and 1.0-2.0. Derived fractions were proportionally mixed to achieve the required particle size distribution for standard base-course aggregates according to Swedish Road Administration (SRA) technical specifications for roads (ATB Road, 2005) (Ref 9), see Fig. 1.

2.3 Sand Equivalent Test

The sand equivalent (SE) test is used to determine the relative proportions of plastic fines or clay-like material in fine aggregates. This test has the advantages that it is quick to perform, requires very simple equipment and can be used with minimal training or experience. It has given reasonably good results (Ref 8).

The experiment was performed according to Swedish standard SS EN 933-8 (Ref 14). Fine aggregate passing the 2.0-mm sieve was placed in a graduated, transparent cylinder which was filled with a mixture of water and a flocculating agent (Fig. 2). After agitation and 20 min of settling, the sand particles separated from the clay-like fraction, and the height of the sand (flocculated particles), and the sand plus suspended clay fraction, were measured.

The sand equivalent value of a specific fine aggregate fraction is defined as dimensionless value and represents the ratio between the quantities of flocculated material (sand) and suspended material. It can be calculated with the following equation:

$$SE = \frac{h_2}{h_1} \times 100$$

where h_1 is the total height of flocculated material plus suspension, in millimeters and h_2 is the height of flocculated material, in millimeters.

Table 1 Content of free mica (particle %) for respective samples and fine fractions

	0.125-0.25 mm	0.25-0.5 mm	0.5-1.0 mm	Mica content in thin section	Description
Material 1	81	71	51	Biotite 10% Muscovite 20%	Medium grained, mica-rich gneiss
Material 2	57	54	34	Biotite 24%	Dark medium to coarse-grained mafic metatonalite
Material 3	56	46	40	Biotite 20.3% Chlorite 2.8% Muscovite 3.2%	Fine grained, schistose-biotite-rich semipelite
Material 4	40	29	20	Biotite 0-37% Chlorite 1.2% Muscovite 6.6%	Fine-grained, schistose-biotite-rich semipelite
Material 5	19	19	7	Biotite 5.6% Chlorite 0.6% Muscovite 2.5%	Fine- to medium-grained, red weakly foliated granite
Material 6	0.9	0.5	0.4	Biotite 0.6% Chlorite 0.9%	Fine-grained quartzite

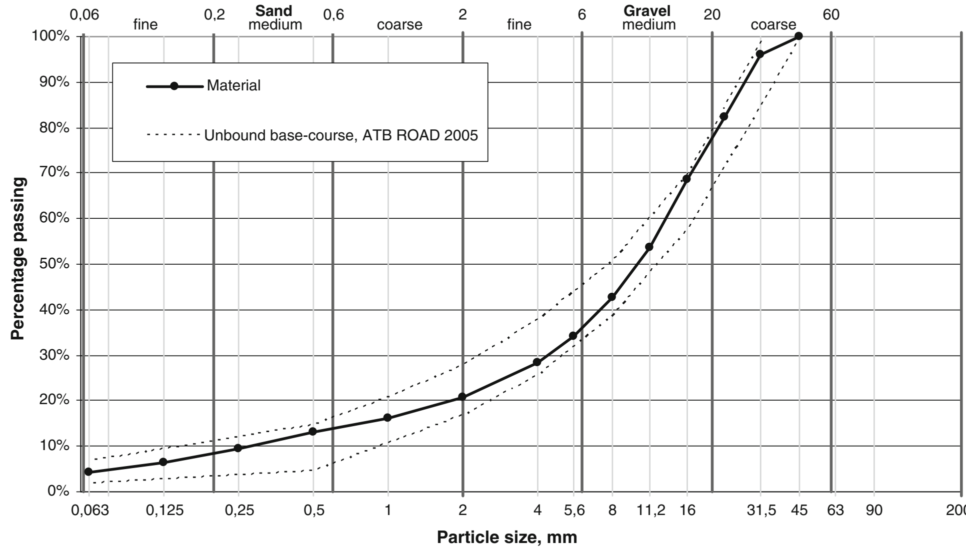

Fig. 1 Particle size distribution for material fractions 0-2 mm

Fig. 2 Sand equivalent test equipment

Higher sand equivalent will be obtained in case of a fine aggregate with lower content of clay-like materials.

The results of the test performed showed a good correlation between the content of mica and SE values and presented in Table 2.

2.4 Methylene Blue Test

This French test method is recommended by the International Slurry Seal Association (ISSA) to quantify the amount of harmful clay minerals; organic matter and iron hydroxides present in fine aggregate (Ref 15). The principle of the test is to add quantities of a standard aqueous solution of the dye (methylene blue) to a sample until adsorption of the dye ceases (Ref 8).

The experiment was performed according to Swedish standard SS EN 933-9 (Ref 16). Representative materials of dried fine aggregates were screened through 2.0 mm sieve in accordance with SS-EN 933-1 (Ref 13). The portions of the materials obtained after sieving were tested for methylene blue value (MBV). At least 200 g of the material are dispersed in 500 g of distilled water in a beaker (Fig. 3).

Ten grams of MB were dissolved in distilled water, to produce 1 L of solution, so that 1 mL of solution contains 10 mg of MB. This MB solution is titrated stepwise in 5 mL aliquots from the burette into the continually stirred fine aggregate suspension. After each addition of MB solution and stirring for 1 min, a small drop of the aggregate suspension was removed with a glass rod and placed on a filter paper. Successive additions of MB solution were repeated until the end point was reached. Initially, a well-defined circle of MB-stained dust was formed and surrounded with an outer ring or corona of clear water. The end point was reached when a permanent light blue coloration or "halo" is observed in this ring of clear water (Fig. 4).

The MBV of a specific fine aggregate fraction is defined as grams of MB per kilogram of fine fraction and calculated by the following equation:

$$\mathrm{MB} = \frac{V}{M} \times 10$$

where M is the mass of the test portion, in grams and V is the total volume of dye solution injected, in milliliters.

The MBV expresses the quantity of MB required to cover the total surface of the clay fraction of the sample with a monomolecular layer of the MB. Therefore, the MBV correlates to the content of phyllosilicate particles times their specific surface (Ref 8).

Table 2 Fine aggregate test results

Material	Mica content, %	MBV	SE1 value	SE2 value
Material 1	81	2	38.4	36.2
Material 2	57	1	40.8	40.8
Material 3	56	1	46.2	45.5
Material 4	40	1	50	48.5
Material 5	19	0.5	55.7	57
Material 6 (Reference)	0.9	0.35	57	59.7

Fig. 3 Methylene blue test equipment

The results obtained showed a high acceptance (see Table 2).

3. Results and Discussion

The investigations revealed the expected results. Both methods showed susceptibility to mica content and gave direct correlations. Table 2 and Fig. 5 and 6 summarize the results of the performed sand equivalent and MB tests.

The sand equivalent test gave a desirable negative correlation (Fig. 5). The increase of mica content led to decrease in sand equivalent values. Negative correlation between the content of free mica particles and SE values can be explained by the major feature of the sand equivalent test. The higher SE value gives the less quantity of phyllosilicate fines due to their flake-formed particles.

According to the formula for the calculation of sand equivalent value, the greater suspension heights give lower SE values. The obtained results showed that mica-rich materials have the lowest SE value, which indicates that mica particles appeared to be suspended rather than flocculated. Despite the fact that mica particles have relatively high density (3.0-3.2), in comparison with similar particles of quarts and feldspar (2.6), the flocculation rate of mica is much lower. This phenomenon can be justified by the geometrical parameters of mica particles. The flake-shaped mica minerals can remain suspended in liquids, while round-shaped grains of quarts and feldspar flocculate immediately.

The performed MB test showed positive nonlinear correlation. Figure 6 summarizes the results of the performed MB test. Analyzing the data showed that increased content of free mica in samples led to a significant increases in MBVs.

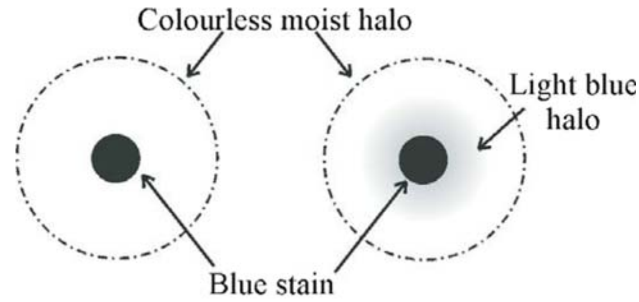

Fig. 4 Determination of MB value. Acquisition of light blue halo

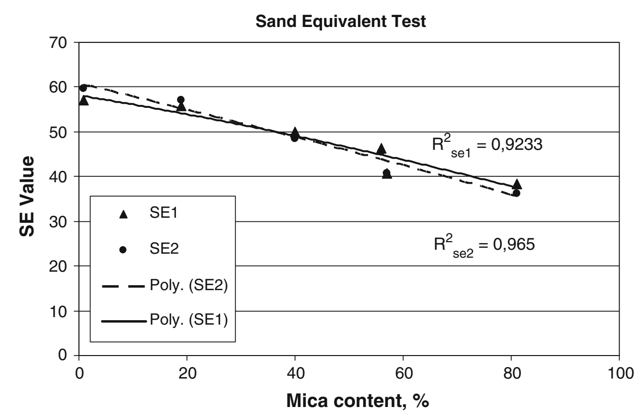

Fig. 5 Relation between sand equivalent values and an increasing content of free mica. Trendlines are polynomial, second order

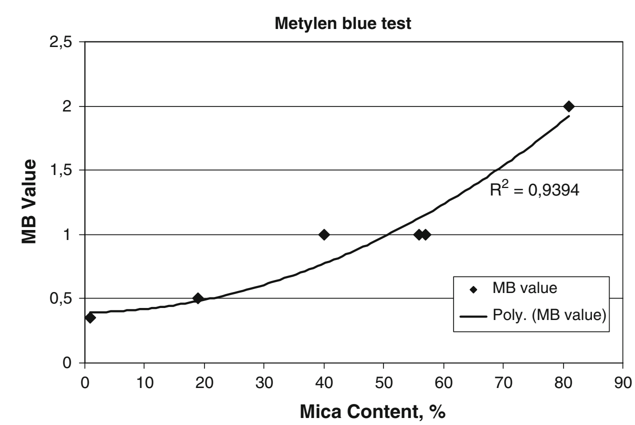

Fig. 6 Relation between Methylene Blue values and an increasing content of free mica. Trendlines are polynomial, second order

The results obtained from the test point out the following speculations.

Due to the high-reaction surface of mica particles the increase in specific surface area of the sample is noticeable; consequently, a greater amount of MB solution is required to cover the total surface of the sample with a monomolecular layer of the MB. Hence, the MBV is directly proportional to the content of mica particles.

Another factor that increases the absorption of MB is the mica's inter space structure.

4. Conclusions

The current study revealed the lack of laboratory methods for estimating free mica in aggregates. With the aid of the experimental study, the following things can be concluded:

- Both methods showed susceptibility to content of free mica in fine fraction and gave strong correlation in terms of mica content.
- The receptiveness of the sand equivalent test to mica content can be caused by the ability of mica to be suspended in liquids due to its flake-shaped grains. Higher sand equivalent values will be obtained with decreasing contents of mica minerals.
- The results of the MB test can be explained by the increased reaction surface of mica particles which leads to an increase in the total reaction surface of the sample. Other possible important factors are the schistose structure of mica particles and the pronounced ability of mica to absorb liquids.
- The test methods suggest that they can be used as an indicating technique for quantitative determination of free mica particles in fine fraction of unbound granular materials. For the final estimation, more detailed tests are required. Because the samples selected represented fine to coarse-grained rocks with supposed maximal content of free mica in different fractions, it would be prudent to perform an additional study of each rock type separately.

Acknowledgments

The authors gratefully acknowledge the financial support from the Swedish National Road and Transport Research Institute (VTI) and Luleå University of Technology (LTU). Furthermore, we acknowledge the contributions of Karl-Johan Loorents (VTI) and Håkan Arvidsson (VTI) to present research. The authors would especially like to thank Erling Nordlund (LTU) for providing an opportunity to perform the current research. Additionally we would like to thank all those persons who assisted in this work but are not mentioned above.

References

1. L. Uthus, et al., *A Study on the Influence of Water and Fines on the Deformation Properties and Frost Heave of Unbound Aggregates*, Cold Regions Engineering, Norway, 2006
2. J. Ekblad, *Influence of Water on Coarse Granular Road Material Properties*, KTH, Stockholm, Sweden, 2007
3. P. Höbeda, *Glimmer i vägmaterial. Inverkan på egenskaper och analysmetoder för glimmerhalt*, VTI Meddelande 527, Väg- och trafikforskningsinstitutet, Linköping, 1987
4. K. Miskovsky, Enrichment of Fine Mica Originating from Rock Aggregate Production and Its Influence on the Mechanical Properties of Bituminous Mixtures, *J. Mater. Eng. Perform.*, 2004, **13**(5), p 607–611
5. D. Woodward, et al., *Clay in Rocks*, University of Ulster, UK, 2002
6. K.-J. Loorents et al., *Free Mica Grains in Crushed Rock Aggregates*, VTI, Swedish National Road and Transport Research Institute, VTI's Publication, 2007
7. D. Brian, et al., *Aggregate Properties and the Performance of Superpave-Designed Hot Mix Asphalt*, Transportation Research Board, Washington, D.C., 2005
8. P.S. Kandhal et al., Tests for Plastic Fines in Aggregates Related to Stripping in Asphalt Paving Mixtures, NCAT Report 98-03, 1998
9. "ATB VÄG 2005", VV Publ nr 2005:113, Swedish Road Administration, Borlänge, 2005, in Swedish, http://www.vv.se/filer/30745/pdf-alla_kapitel_atb_vag_2005.zip
10. K.-J. Loorents et al., *Trends of Enrichment of Free Mica Grains in Crushed Rock Aggregates*, VTI, 2008
11. I. Sims and P. Nixon, RILEM Recommended Test Method AAR-1: Detection of Potential Alkali-Reactivity of Aggregates—Petrographic Method, *Mater. Struct./Matériaux Construct.*, 2003, **36**, p 480–486
12. W.D. Nesse, *Introduction to Optical Mineralogy*, Oxford University Press, 2004 (Swedish Standard)
13. "Tests for Geometrical Properties of Aggregates—Part 1: Determination of Particle Size Distribution—Sieving Method", EN 933-1:1997, European Committee for Standardization, 1997
14. "Tests for Geometrical Properties of Aggregates—Part 8: Assessment of Fines—Sand Equivalent Test", EN 933-8:1999, European Committee for Standardization, 1999
15. International Slurry Seal Association, "Test Method for Determination of Methylene Blue Absorption Value (MBV) of Mineral Aggregate Fillers and Fines," ISSA Bulleting 145, 1989
16. "Tests for Geometrical Properties of Aggregates—Part 9: Assessment of Fines—Methylene Blue Test", EN 933-9:1998, European Committee for Standardization, 1998