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Test Methods and Specification Criteria for Mineral Filler Used in Hot-Mix Asphalt

DETAILS

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Research Results Digest 357

TEST METHODS AND SPECIFICATION CRITERIA FOR MINERAL FILLER USED IN HOT MIX ASPHALT

This digest summarizes key findings of NCHRP Project 9-45, "Test Methods and Specification Criteria for Mineral Filler Used in HMA," conducted by the University of Wisconsin–Madison under the direction of the principal investigator, Hussain U. Bahia. The digest is based on the project final report authored by Hussain U. Bahia, Ahmed Faheem, and Cassie Hintz of the University of Wisconsin–Madison; Imad Al-Qadi of the University of Illinois at Urbana–Champaign; and Gerald Reinke and Erv Dukatz of Mathy Technical Engineering Services. The full text of the project final report is available for download at http://apps.trb.org/cmsfeed/ TRBNetProjectDisplay.asp?ProjectID=979.

INTRODUCTION

While there is no lack of evidence that mineral fillers can have important effects on the performance of hot mix asphalt (HMA) (and, by extension, that of warm mix asphalt, WMA), current HMA mixture design procedures and materials specifications include only general limits on fillerto-binder mass ratio. Thus, these procedures and specifications, notably AASHTO R 35, *Superpave Volumetric Design for Hot Mix Asphalt (HMA)*, are silent on the possible influence of fillers on key indicators of HMA performance.

The objectives of NCHRP Project 9-45, "Test Methods and Specification Criteria for Mineral Filler Used in Hot Mix Asphalt," were to (1) identify (or develop, as necessary) test methods for mineral filler that characterize its physical and chemical effects on the performance of mastics (blends of asphalt binder and mineral filler) and HMA and (2) propose specification criteria for mineral filler that help optimize HMA performance.

RESEARCH PLAN

A comprehensive literature review was conducted to evaluate current knowledge of the characterization of mineral fillers and the measurement of filler effects on mastics and HMA. The state departments of transportation were also surveyed for their views on the most critical aspects of the use and specification of mineral filler. Based on the findings of the literature review and survey, an experimental plan was developed to evaluate a select set of filler properties and associated test methods that were identified as having the potential to critically influence mastic performance, HMA mixture performance, or both. The tests included physical tests to characterize filler geometry and chemical tests to characterize filler composition.

The 32 fillers shown in Table 1 were selected for testing in the experimental phase of the project. These fillers represent the wide ranges commonly found in the United States of (1) *mineralogy* of natural fillers (those produced in quarries

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No.	Code	Туре	Rigden Voids (%)	F M ¹	CaO² (%)	MBV ³	SG⁴	Pl⁵
1	AN1	Andesite	41.9	5.18	6.80	24.56	2.60	Non-Plastic
2	BH1	Hard Basalt	33.2	5.31	8.20	5.92	2.72	Non-Plastic
3	BH2	Hard Basalt	33.8	4.65	7.00	11.19	2.77	Non-Plastic
4	BV1	Vesicular Basalt	37.8	4.59	9.10	11.65	2.79	3.00
5	CA1	Caliches	40.3	4.91	44.00	8.96	2.59	Non-Plastic
6	CA2	Caliches	45.0	5.13	40.00	10.25	2.49	Non-Plastic
7	CBC1	Carbon Black	NA	NA	NA	NA	NA	NA
8	CBF1	Carbon Black	NA	NA	NA	NA	NA	NA
9	CM1	Portland Cement	30.0	4.27	14.30	NA	2.87	Non-Plastic
10	DH1	Hard Dolomite	42.8	5.07	26.00	2.79	2.59	Non-Plastic
11	DH2	Hard Dolomite	27.0	4.54	32.01	0.99	2.61	Non-Plastic
12	DS1	Soft Dolomite	34.7	6.02	26.00	1.17	2.71	10.00
13	DS2	Soft Dolomite	29.4	4.73	27.00	1.82	2.70	Non-Plastic
14	FAC1	Fly Ash Type 'C'	26.2	3.30	25.03	0.00	2.47	Non-Plastic
15	FAC2	Fly Ash Type 'C'	26.2	3.88	23.13	0.00	2.37	Non-Plastic
16	FAF1	Fly Ash Type 'F'	30.1	4.29	5.10	0.39	2.14	Non-Plastic
17	FAN1	Fly Ash Non Spec	40.5	3.22	21.80	0.01	2.38	Non-Plastic
18	FAN2	Fly Ash Non Spec	28.9	2.98	24.45	0.02	2.54	Non-Plastic
19	FS1	Furnace Slag	40.0	4.87	50.30	0.00	2.80	Non-Plastic
20	FS2	Furnace Slag	49.1	4.32	45.07	0.02	2.89	Non-Plastic
21	GH1	Hard Granite	42.6	4.06	3.50	2.35	2.66	Non-Plastic
22	GH2	Hard Granite	38.8	3.76	2.80	31.56	2.53	8.00
23	GHB1	Hard Granite Baghouse	38.3	4.26	4.70	2.81	2.62	6.00
24	GRQ1	Gravel Quartzite	29.5	4.39	32.70	2.06	2.66	Non-Plastic
25	GRQ2	Gravel Quartzite	36.5	6.32	0.95	0.66	2.55	Non-Plastic
26	GS1	Soft Granite	40.2	3.85	4.60	14.47	2.58	Non-Plastic
27	GS2	Soft Granite	47.0	3.13	7.80	0.88	2.40	Non-Plastic
28	HL1	Hydrated Lime	38.8	4.74	27.60	0.80	2.70	Non-Plastic
29	HL2	Hydrated Lime	38.1	4.15	32.20	1.05	2.79	Non-Plastic
30	LH1	Hard Limestone	32.2	5.63	43.14	0.62	2.65	Non-Plastic
31	LS1	Soft Limestone	26.2	4.30	49.20	0.00	2.64	Non-Plastic
32	LS2	Soft Limestone	35.4	3.68	46.30	3.87	2.62	Non-Plastic
Notes: 1. 2. 3. 4. 5.	Size distribution reported as fineness modulus (FM). Total calcium content reported as percent calcium oxide (CaO). Active clay content reported as the methylene blue value (MBV). SG = specific gravity.							

 Table 1
 Summary of filler testing results.

during rock processing or collected in baghouse collectors during HMA production) and (2) *composition* of manufactured fillers (those such as fly ash and slag dust that are byproducts of industrial processes). The project team later dropped the two carbon black fillers (CBC1 and CBF1) from the experiment because they are extremely fine with low density, which made them easily airborne and difficult to handle. Also, the Portland cement (CM1) was not tested with the methylene blue (MB) method for active clay content because of this filler's reactivity with water. Based on the initial testing of these 32 fillers, a subset of 17 fillers was selected for blending with four asphalt binders to create a total of 68 mastics for testing using the methods in Table 2. The four binders were (1) a PG 64-22 with low asphaltenes content from a light crude source, (2) a PG 64-22 with high asphaltenes content from a heavy crude source, (3) the light crude PG 64-22 modified with PPA to a PG 70-22, and (4) the light crude PG 64-22 modified with SBS to a PG 70-22.

Upon completion of mastic performance testing, 16 mastics were selected for blending to pre-

Mastic Characteristic	Response Variable	Test Method	Aging
1. Workability	Viscosity	Rotational Viscosity (RV)	Unaged
	Accumulated Strain	Dynamic Shear Rheometer	
2. Rutting Resistance	Non Recoverable Compliance	(DSR)/Multiple Stress Creep and Recovery (MSCR) 25 mm PP	Unaged
3. Fatigue	Fatigue Life	Time Sweep	
Resistance	G*sin δ	DSR	Unaged and PAV only
4. Thermal Cracking Resistance	S and m	Bending Beam Rheometer (BBR)	PAV only
5. Moisture Damage Resistance	Water Sensitivity	EN 1744-4 Unaged	
nesistance	Bond Strength	PATTI/or DSR	

 Table 2 Mastic testing program.

pare coarse- and fine-graded mixtures for HMA performance testing. Mixtures were tested to characterize their workability and their resistance to rutting, fatigue damage, low-temperature cracking, and moisture damage using the performance-related methods in Table 3. The mastic and mixture test results were then statistically correlated to the filler test results to identify possible relationships among filler properties, mastic properties, and mixture performance, and models to estimate mastic performance in terms of filler and binder properties were

Mixture Characteristic	Measured Response	Test Method
Workability	Gyrations to 92%Gmm	Superpave Gyratory Compactor
Permanent Deformation	Dynamic Modulus (E*)	Asphalt Mixture Performance Test (AMPT)
	Flow Number (FN)	AMPT
Fatigue Resistance	Cycles to 45% Drop in E*	Indirect Tension Test (IDT)
Thermal Cracking	Fracture	IDT
Moisture Damage	Rut Depth	Hamburg Wheel Test

developed. Finally, the practicality and repeatability of the filler test methods to capture the critical filler properties were assessed. The assessment included testing of six fillers by multiple laboratories and operators using the selected filler test methods.

FINDINGS

Table 4 presents (1) the primary filler characteristics experimentally identified as critical for defining the influence of fillers on mixture performance and (2) test methods for measuring these properties. The test methods are well established—most as AASHTO, ASTM, or European standards—and so would be suitable in the future as a basis for filler specifications. However, some proposed testing equipment are not yet available in the North American market (e.g., that specified in EN 1097-4 to measure Rigden Voids, RV) or are relatively costly (e.g., the X-ray fluorescence spectrometer needed to measure calcium content).

The test results in Table 1 for the initial set of 32 fillers indicate that mineral fillers currently used in HMA production in the United States vary significantly in the physical and chemical properties measured by the selected test methods. In general, manufactured fillers show a more extreme range of properties compared to natural fillers and their effects on mastic and mixture performance indicators

Filler Property	Test Method
Fractional Voids, reported as Rigden Voids (%)	EN 1097-4, Tests for mechanical and physical properties of aggregates. Determination of the voids of dry compacted filler
Size Distribution, reported as fineness modulus (FM)	ASTM D4464, Particle Size Distribution of Catalytic Material by Laser Light Scattering
Calcium Content, reported as % CaO	X-ray Fluorescence, e.g., ASTM D5381, Standard Guide for X-ray Fluorescence (XRF) Spectroscopy of Pigments and Extenders
Active Clay Content, reported as methylene blue value (MBV)	AASHTO T 330, The Qualitative Detection of Harmful Clays of the Smectite Group in Aggregates Using Methylene Blue
Specific Gravity, SG (required for determination of Rigden Voids)	Helium Pycnometer method, e.g., ASTM D5550, Specific Gravity of Soil Solids by Gas Pycnometer

Table 4 Suggested test methods to measure performance-related filler properties.

cannot be easily estimated from the filler and binder properties.

Mineral fillers were found to significantly affect mastic and mixture performance indicators and the effects were, in most cases, highly specific to the fillerbinder combination. Additionally, the filler volume concentration was found to have a significant effect even when the mass ratio of filler to binder was kept constant at 1:1. These results suggest that the current practice of limiting dust (filler)-to-binder ratio by mass is insufficient as the practice cannot account for the effect of filler volume concentration on mixture or mastic performance. The experimental results clearly showed that fractional voids (packing) characteristics measured by RV vary significantly among natural fillers and have an important influence on mastic and mixture behavior. Varying mass ratio was not studied; therefore, further study would be required to evaluate the role of the filler concentration on mastic performance.

The effects of natural fillers were found to be significantly different from those of manufactured fillers. Natural fillers appear to influence mastic and mixture performance through a uniform physicochemical mechanism. Therefore, it was feasible to use regression analysis to obtain a generalized prediction model for some properties of mastics containing natural fillers. Manufactured fillers, on the other hand, showed unique influences on the performance of mastic and mixtures studied in this project, and generalized trends could not be obtained; manufactured fillers should be tested thoroughly after mixing with intended binders before they are introduced into the HMA mixture.

CONCLUSIONS

The following conclusions are based on the statistical correlations and models developed for the five specific HMA performance characteristics studied in the project.

HMA Workability

Mastic viscosity was successfully related to mixture workability, as measured by the number of gyrations to 92% G_{mm} (N92). Although N92 values were found to be highly dependent on aggregate gradation, the RV value was identified through multi-linear regression as the filler property that has an important influence on mastic viscosity and mixture workability. Tentative maximum limits for mastic relative viscosity were defined to ensure acceptable mixture compactability for coarse-graded mixtures. Specification limits for mixtures were estimated from the project data set since no specific guidance could be found in the literature. In cases where mastic testing is not possible, a statistical model to estimate mastic relative viscosity based on the RV value of the filler and the binder viscosity was proposed. The model can be used to check that the relative viscosity of mastic is below the maximum proposed limit.

HMA Rutting

Mastic rutting resistance was successfully related to mixture rutting resistance as measured by the flow number (FN) with AASHTO TP 79, *Determining the Dynamic Modulus and Flow Number for Hot Mix As-* phalt (HMA) Using the Asphalt Mixture Performance Tester (AMPT). A statistically significant relationship was found between the mastic non-recoverable compliance, J_{nr} , measured with ASTM D7405, Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using the Dynamic Shear Rheometer, and the mixture FN; however, this relationship is not as important to mixture rutting resistance as the aggregate gradation. Tentative maximum limits for mastic J_{nr} were defined to ensure acceptable mixture FN values for coarse- and fine-graded mixtures. A statistical model to estimate mastic J_{nr} from filler RV and binder J_{nr} is proposed to ensure acceptable FN values.

HMA Fatigue Damage

A significant variation in mastic fatigue damage resistance was found across the range of fillers used in the project. The role of fillers in mastic fatigue damage resistance was highly dependent on binder modification type and binder chemistry. Mixture fatigue damage resistance was found to be affected mainly by gradation and binder modification, and only marginally by properties of the filler or base binder. Thus, the results of this research were not sufficient to define the role of fillers in mixture fatigue damage resistance or to propose filler specification criteria for this aspect of HMA mixture performance.

HMA Low-Temperature Cracking

The only experimental variables with a statistically significant relationship to mixture low temperature *stiffness* were gradation and base binder source. Mastic relative stiffness (i.e., the stiffness of the mastic compared to that of its base binder) has a significant effect on mixture *strength* at low temperatures that is only slightly less important than the effect of gradation.

Mastic low temperature stiffness (S) and creep rate (m) measured by AASHTO T 313, *Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)*, were found to be sensitive to filler type and binder modification. It was found, as expected, that all fillers increase binder stiffness, but fillers can either increase or decrease (m) value, depending on binder modification type. The statistical analysis found that the mastic low temperature stiffness is dependent on the RV value and calcium content (reported as % CaO) of the filler, and a model was developed to estimate mastic stiffness at low temperatures as a function of filler and binder properties.

Although relationships were found between mixture strength and relative mastic stiffness and between relative mastic stiffness and filler RV and % CaO, it was not possible to propose limits on mastic or filler properties to ensure acceptable low-temperature cracking resistance. Mixture cracking resistance depends on stiffness and the capability for stress relaxation (as indicated by the creep rate, m) in addition to strength, and since no trends were found for mixture stiffness, no specification limits could be proposed here.

HMA Moisture Damage

The resistance of mastic to moisture damage was found to be highly binder specific; filler properties had limited influence. Therefore, mixture moisture damage testing was conducted on a limited scale as compared to that for other HMA mixture performance characteristics. These limited results indicated that mixture moisture resistance is highly dependent on mastic performance, but that this dependency is mainly related to binder type, rather than to the filler used in the mastic.

PROPOSED TESTS AND SPECIFICATIONS

Filler Characterization Tests

A multi-laboratory experiment was conducted to assess the repeatability and practicality for routine use of the proposed suite of filler characterization tests in Table 4. The results, although limited in scope, indicate that the tests are highly repeatable. In particular, test methods for RV and specific gravity, which have been identified as the most important filler properties for mixture performance, were found to be highly repeatable and able to distinguish between different fillers with high accuracy. The tests were rated by operators as user-friendly and can produce repeatable results after modest operator training. The tests for measuring calcium content by X-ray florescence and fineness modulus by laser diffraction showed good repeatability but require costly equipment and so may not be practical for routine testing of fillers.

Filler Specification Criteria

Proposed specification criteria to ensure adequate mixture performance with respect to workability

Table 5 Proposedworkability limits.

Maximum N92	43
Maximum Relative	5.0
Viscosity	

and rutting resistance are presented in Tables 5 and 6. Due to the high filler-binder interactions measured in this study, these specification criteria are based on mastic properties rather than filler properties. The experimental results did not support any proposed specification criteria for resistance to fatigue damage, low temperature cracking, or moisture damage.

In instances where mastic properties cannot be measured directly, these specification criteria are supplemented by best-fit models (Equations 1 and 2) developed to estimate mastic properties in terms of filler and binder properties.

$$Mastic \ Viscosity = -8244 + 4.68 * Binder \ Viscosity + 205 * RV \tag{1}$$

Table 6 Proposed maximumvalues for mastic J_{nr} at 3.2kPaby gradation type.

Mixture	Maximum Mastic J _{nr} at	
Gradation	3.2kPa (1/kPa)	
Fine	0.40	
Coarse	0.55	

Mastic
$$J_{nr} = 1.01 + 0.160 * Binder J_{nr} - 0.230 * RV$$
(2)

Where J_{nr} = non-recoverable compliance.

The R^2 values for Equations 1 and 2 are 0.684 and 0.749, respectively.

In addition, the best-fit model ($R^2 = 0.606$) shown in Equation 3 was obtained to predict relative low-temperature mastic stiffness from lowtemperature binder stiffness and filler RV and CaO values:

$$Mastic \ Stiffness_{relative} = 2.32 + \left[\frac{145 + 4.84RV - 1.71CaO}{Stiffness_{binder}}\right]$$
(3)

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