Outline of Research Proposed
for
Project B-15

ADHESION OF BITUMINOUS MATERIALS TO MINERAL AGGREGATES

Objective: The primary object of this proposed research is to investigate existing procedures or develop new means for evaluating the adhesive capacity of bituminous materials when applied to mineral aggregates. This includes bituminous materials in combination with additives or so-called anti-stripping agents; hence, the scope of the project includes:

1. A study of aggregates for hydrophyllic or hydrophobic properties as related to different types of bitumen, and the selection of a standard aggregate by which the adhesion of bituminous materials themselves may be judged.

2. Comparison of grades and sources of bituminous materials with respect to power of adhesion to (or tendencies to strip from) different aggregates, both when used separately and when combined with anti-stripping agents.

3. Evaluation of different products sold commercially as additives to prevent stripping.

The ultimate aim is to establish acceptance tests and specifications that can be applied to both the bituminous materials and the additives.

Materials: Three types of aggregates from as many as seven sources will be taken as follows:

1. Limestone - from four sources, selected for wide distribution geographically but mainly for variation in characteristics that could be expected to affect bituminous adhesion. In each instance, complete information regarding physical, chemical, and mineralogical properties will be compiled in order to correlate results with current studies of aggregates - Project C-22.

2. Slag - from one source.

3. Gravel - from three sources, Ohio River below Louisville (Owensboro), Ohio River above Louisville, and western Kentucky (Tertiary) pit.

Five types of bituminous materials will be considered, and these will be obtained from at least four separate sources, the three sources of asphalt being representative of the Appalachian, Southern Illinois, and Ozark fields. The grades of bituminous materials will be: MC-3, RC-2, RT-6, MC-5, and PAC-8.

To these a minimum of four different additives will be applied for a portion of the tests. All bituminous materials will be tested for characteristics in accordance with standard specifications, and when treated with an additive, a sample of the mixture will be analyzed to determine changes in characteristics that may have occurred. Additives will be combined with bituminous materials in accordance with methods recommended by the manufacturer in each instance.

Procedures: The research will be conducted in stages in order to minimize the number of tests required to reach the objective. The different stages, materials involved, and objectives will be:

1. Stage 1 - A single aggregate with asphalt from a single source (all grades) and tar from a single source, and additives from four sources all subjected to complete set of tests. Objective - elimination of testing techniques.

2. Stage 2 - A second aggregate with asphalt from the same single source (all grades) and tar from a single source, and additives from four sources subjected to the group of tests selected in Stage 1. Objective - elimination of grades of bitumen as being inconsequential (if such is so).

3. Stage 3 - All other aggregates tested with selected grades of asphalt from the second and third sources and tar from a single source, and additives from four sources all subjected to the selected tests. Objective - establishment of the test procedures as being universally applicable to any aggregate, all grades and sources of bitumen, and to bituminous materials in combination with additives.

Through correlation of results from the three steps it is probable that a test or combination of tests will be found valid in differentiating bituminous-aggregate and bituminous-additive-aggregate combinations on the basis of adhesion.

The test procedures which will be applied in Stage 1 and investigated for applicability may be classed as: (1) Immediate Immersion, (2) Delayed Immersion, (3) Three Minute Boiling, (4) Immersion-Agitation, (5) Immersion-Axial Compression, (6) Immersion-Radial Compression, and (7) Tension. In the first four types the rating will be visual and expressed as a percentage (by weight) of particles completely coated; in the remaining tests the rating will be expressed as percentage loss of stability or tensile strength as effected by immersion in water. Results from tests on three separate samples representing each combination of materials and test conditions shall be averaged to obtain a rating for that combination.

Specific and detailed accounts of proposed methods follow.

PREPARATION OF SPECIMENS AND PROCEDURES FOR TESTS (1), (2), (3), and (4)

The aggregate shall be crushed* and separated by size so that only that portion passing the 3/8" sieve and retained on the No. 4 sieve is used for test purposes. This material shall be washed to remove all dust, and stored in accordance with the uses to which it is to be applied.

Material for tests with the aggregate wet shall be immersed in distilled water (immediately after washing) in a container suitable for storage for a period of at least 48 hours. Aggregate for samples of this type upon removal from storage shall be allowed to drain on absorbent cloth or paper for a period of 5 minutes at room temperature before being mixed with the bituminous material.

For tests, with the aggregate dry, the washed material shall be spread uniformly (not more than 1 inch in depth) in a place free from dust and exposed at room temperature for a period of 48 hours. Subsequent storage shall be in a sealed container free from dust.

* Some tests will be made on uncrushed gravel of the size specified.

Test specimens with both wet and dry aggregate shall be made only with MC-3, RC-2, and RT-6; in tests on mixes containing MC-5 and PAC-8, the aggregate will be pre-heated as in plant mix operations; hence, only the dry aggregate shall be tested. The procedures used in preparing specimens containing the first group of bituminous materials* shall be as follows:

Immediate Immersion: A 50 gram sample of the wet or dry aggregate (as the case may be) shall be placed in an 8 ounce seamless tin box and thoroughly mixed until completely coated with 2 grams of the bituminous MC-3 (heated to 175°F.), RC-2 (heated to 150°F.), or RT-6 (heated to 120°F.) as the case may be. Immediately after mixing, the sample shall be immersed in 400 cc. of distilled water at room temperature and allowed to remain for 24 hours.

The sample shall then be removed carefully from the beaker and placed on absorbent paper for air-drying. When dry, particles completely coated shall be separated from those only partially coated. Both shall be weighed, and the percentage of completely coated particles computed on the basis of total weight.

Delayed Immersion: The procedures in this test shall be the same as that for the Immediate Immersion test except that after mixing, the material shall be cured for 24 hours in air at room temperature before being immersed in water for 24 hours. Rating of the materials shall be the same as before.

Three Minute Boiling: The procedure for this test shall be the same as that for the Delayed Immersion except that after the material has cured for 24 hours in air, it shall be immersed in 400 cc. of distilled water and brought to a vigorous boil for a period of 3 minutes. Air drying and rating shall then be done as before.

Immersion-Agitation: A 200 gram sample of aggregate shall be mixed with 8 grams of the bituminous material heated to temperatures specified under the heading Immediate Immersion test above. After the mixing, curing for 24 hours in air shall be allowed, after which the sample shall be placed in a pint jar and covered with distilled water having a temperature of 77°F. until the jar is about 3/4 full. The

*The only variations in procedures applied for mixes with MC-5 and PAC-8 are as follows: Aggregates for tests with MC-5 shall be heated in an oven at 140°F. for 30 minutes before being mixed with the asphalt heated to 225°F. For mixes with PAC-8 both the aggregate and the asphalt shall be heated to 250°F. immediately before mixing.

jar shall be capped and placed in the rotating frame of an agitation machine which will cause about 55 rotations per minute. After 30 minutes of agitation, the machine shall be stopped, the aggregate placed on porous paper and allowed to dry before being separated and rated for percentage of particles completely coated.

If the weight of particles completely coated equals more than 80 percent of the total weight, the sample shall be placed in the jar 3/4 full of distilled water at 100°F. and agitation resumed for 15 minutes, after which the sample shall be dried and rated as before.

PREPARATION OF SPECIMENS AND PROCEDURES FOR TESTS (5), (6), and (7)

The aggregate shall be crushed*, separated into different sizes and recombined in proportions that will provide either gradation No. 1 or gradation No. 2 (as defined below), whichever the test shall require. No attempt shall be made to eliminate dust either initially or in storage. The gradations mentioned above shall be:

Gradation No. 1			Gradation No. 2		
Sieve Size	Pct. Ret.	Pct. Passing	Sieve Size	Pct. Ret.	Pct. Passing
1"	0	100	1/2"	0	100
3/4"	10	90	3/8"	0	100
1/2"	20	70	No.4	35	65
3/8"	10	60	No.8	15	50
No.4	25	35	No.16	10	40
No.8	10	25	No.50	20	20
No.50	15	10	No.100	10	10
No.200	10	0	No.200	5	5

In tests with RC-2, MC-3, and RT-6, only gradation No. 1 shall be used, but both gradation No. 1 and gradation No. 2 shall be used in tests with MC-5 and PAC-8.

Material for tests with the aggregate wet (which applies only to gradation No. 1 in combination with RC-2, MC-3, or RT-6) shall be immersed in distilled water in a container suitable for storage for a period at least 48 hours. Aggregate for samples of this type, upon removal from storage shall be placed in a 200 mesh sieve in a uniformly thin layer

*Some tests will be made on uncrushed gravels from the different sources.

and allowed to drain for a period of 10 minutes at room temperature, before being mixed with the bituminous material. Preparation for tests with dry aggregate in combination with RC-2, MC-3, or RT-6 shall require no advance treatment of the aggregate after crushing and grading and before mixing with the bituminous material. Temperatures to which the bitumen shall be heated before mixing with either wet or dry aggregate shall be:

RC-2 --------- 150°F.
MC-3 --------- 175°F.
RT-6 --------- 120°F.

Aggregate of both gradations No. 1 and No. 2 for combination with MC-5 and PAC-8 shall be heated prior to mixing with the bitumen, the temperatures and time of heating to be as follows:

Bitumen		Aggregate		
Type	Temperature	Gradation	Temperature	Time
MC-5	225°F.	No.1 or No.2	200°F.	30 min.
PAC-8	250°F.	No.1 or No.2	250°F.	Constant Temp.

Procedures for the different tests shall be as follows:

Immersion-Axial Compression. Mixes containing MC-5 and PAC-8 shall be molded immediately after mixing; all others shall be cured loose in air for 24 hours before molding.

The objective in molding materials shall be to obtain specimens 4" in diameter by 4" in height, all specimens of a given aggregate gradation having a given weight of the mix in order to keep a constant density throughout all tests on samples of that category. Hence, the pressure required may vary slightly, but a basic density for each gradation determined under a load of 3000 lbs. per square inch shall govern and thereafter the load shall be of secondary importance. The remaining steps shall be:

1. Specimens 4" in diameter by 4" high shall be molded to the prescribed density under a load approximately 3000 lbs. per square inch applied by plunger at both ends of the specimens. The load shall be held constant for 2 minutes, and then released.

2. All molded specimens shall be cured for 24 hours in an oven at 140°F.

3. After oven curing, the specimens shall be allowed to cool in air, then weighed in air, measured with calipers

for height and diameter, and weighed in water for volume and density determinations.

4. One set of specimens shall be tested in compression at room temperature, and a companion set shall be submerged in water before being tested in compression. The temperature of the water and the length of exposure in water for different mixes shall be:

Bitumen	Temperature	Time
RC-2, MC-3, RT-6	77°F.	4 days
MC-5, PAC-8	140°F.	1 hour

5. In the compression test, the rate of vertical deformation shall be 0.2 inch per minute as measured by an Ames Dial.

Rating of materials shall be on the basis of percentage loss of stability (pounds per square inch in compression) of the immersed samples as compared with the stability of those not immersed prior to the compression test.

Immersion-Radial Compression. This procedure shall be similar to the stability analyses which have been made in the laboratory for some time under the designation of Marshall Stability. As in the Immersion-Axial Compression test, mixes containing MC-5 and PAC-8 shall be molded immediately after mixing, and all others shall be cured loose in air for 24 hours before molding.

Samples 4" in diameter and 2-1/2" in height shall be prepared and tested as follows:

1. Initial compaction with 15 blows of an 18 pound weight dropped for a distance of 18 inches.

2. Leveling of sample and 10 additional blows of the tamper.

3. Application of a static load of approximately 550 lbs. per square inch or that load necessary to bring the sample to exactly a 2-1/2" height.

4. Specimens shall be cooled in the molds for at least two hours before being extruded and placed in an oven at 140°F. to cure for 24 hours.

5. Upon removal from the oven, the specimens shall cool to room temperature, and then be weighed in air, measured for diameter and height, and weighed in water for volumetric and density determinations.

6. One set of specimens shall be tested in radial compression at room temperature, and a companion set shall be submerged in water before being tested. The temperature of the water and the length of immersion in water shall be as specified in part 4 under Immersion-Axial Compression above.

7. In the radial compression test, loads shall be applied at a rate such that the rate of deformation shall be 0.1 inch per minute as measured by a Strain dial.

Rating of materials shall be on the basis of percentage loss of stability expressed in pounds of load for the immersed samples as compared with the stability of those not immersed prior to the compression test.

Tension. The equipment and procedure will, of necessity, be developed as the investigation progresses.

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