

QUANTITATIVE EVALUATION OF STRIPPING BY THE SURFACE REACTION TEST

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In this study, an apparatus and a technique were developed to measure the amount of exposed surface area on asphalt-coated mineral aggregate particles after they had been subjected to the stripping effects of water. The test procedure is based on the principle that calcareous or siliceous minerals will react with a suitable reagent and create a gas as part of the chemical reaction products. Within reasonable time limits in a sealed container, the generated gas creates a certain amount of pressure that can be considered proportional to the mineral surface area exposed to the reagent. With proper selection of reagents and reagent concentrations, asphalt, being a relatively inert substance, will not enter into the reaction and will not contribute to the created gas pressure. By using duplicate aggregate samples, one uncoated and the other asphalt-coated and partially stripped, the change in gas pressure of the respective samples can be compared to determine the amount of exposed surface area on the partially coated sample. This procedure was used to measure the amount of stripping evidenced by 11 aggregate-asphalt mixtures. The aggregates, obtained from various Oklahoma sources, included several different types of carbonate and siliceous materials. The quantitative results of the surface reaction test were compared with visual evaluations of similar mixtures that were subjected to static and dynamic immersion stripping procedures.

•MUCH of the limestone aggregate used in asphalt paving mixtures throughout the country has a tendency to polish or wear under traffic, and over a period of time the decrease in skid resistance of constructed pavements has become a serious highway performance problem. One approach to improving the skid resistance of the asphalt surfacing is to incorporate small quantities of siliceous aggregates in the mixture. The polishing tendency of these siliceous aggregates is generally much lower than that of carbonate aggregates. However, siliceous materials have been looked on with disfavor by the asphalt paving industry primarily because of their relatively poorer adherent properties with asphalt cement.

In addition to skid resistance, desirable properties of a bituminous paving mixture are stability and durability. Although several methods of mix design are available that ensure high stability, durability is more difficult to evaluate. Primarily, it is determined from field observations of in-place mixtures. One aspect of durability relates to pavement failures caused by stripping of asphalt cement from the aggregate in the mixture. Stripping occurs where there is loss of adhesion between the aggregate and the asphalt cement and is due primarily to water action. The resulting deterioration can be a serious problem causing a substantial reduction in total pavement performance.

Inherent factors that affect stripping in an asphalt paving mixture include absorption, surface texture and mineral composition of the aggregate, and chemical composition,

surface tension, and viscosity of the asphalt. External factors such as climate, traffic, and construction techniques also contribute to the stripping process. Thus, stripping is a complex phenomenon influenced by many variables, all of which have some effect on the adhesivity between the aggregate and binder used.

Because of the many factors that influence stripping and the various ways it can take place, a materials engineer should obtain quantitative information relative to the stripping propensities of the aggregates that will be used in the paving mixtures. Unfortunately, most stripping test procedures that are employed for this purpose yield qualitative results. Such tests force the engineer to make decisions based on less than adequate information that is primarily subjective in nature. A more objective approach to stripping measurement is needed to provide reliable quantitative information.

The purpose of this study was to develop such a test procedure whereby a relative measure of the exposed or uncoated surface area of an aggregate sample, partially stripped of its asphalt coating, could be determined. The test procedure was based on the principle that calcareous or siliceous minerals will react with a suitable reagent and create a gas as part of the chemical reaction products. Within reasonable time limits in a sealed container, the generated gas will create a certain amount of pressure that can be considered proportional to the mineral surface area exposed to the reagent.

ADHESION AND STRIPPING THEORIES

Adhesion is defined as that physical property or molecular force by which one body sticks to another of a different nature (1). Four major theories on the cause of adhesion have been formulated and were summarized by Rice (2). These are the chemical reaction, mechanical adhesion, surface energy, and molecular orientation theories.

The chemical reaction theory states that the acidic components of the bituminous material react with basic minerals of the aggregate to form water-insoluble compounds at the interface. Because good adhesion has been reported between acidic (siliceous) aggregate and some asphalts, this theory does not hold true in all cases. Aggregate properties affecting mechanical adhesion include surface texture, absorption and porosity, surface coating and area, and particle shape. It has been observed that rough, irregular-surfaced aggregate has better retention of asphalt than smooth, glossy-surfaced aggregate. Some components of asphalt, primarily the oily constituents, enter the pores or capillaries of an aggregate particle where they are preferentially absorbed. The interlocking of the asphalt coating with these pores makes the asphalt adhere more strongly so that it is less readily stripped by water action.

The surface energy theory is related to surface tension of the asphalt and the interfacial tension between the asphalt and the aggregate. When asphalt spreads over and wets the aggregate surface, a change in energy takes place called adhesion tension (2). Adhesion tension is a surface phenomenon and depends on closeness of contact, mutual affinity of the 2 materials, and time of contact. An aggregate tends to become coated by the liquid for which it has the greatest adhesion tension. Test results indicated that the adhesion tension for water to aggregate was higher than for asphalt to aggregate in most cases (2). When molecules of asphalt come in contact with the aggregate surface they orient themselves to satisfy all energy demands of the aggregate. Water molecules are strong dipoles whereas asphalt molecules appear to possess nonpolar or weakly polar characteristics. Thus, water possesses an advantage over asphalt to rapidly satisfy energy demands of polar aggregate surfaces. However, given enough time without water, the asphalt dipoles may orient themselves to obtain good adhesion between the asphalt film and the aggregate surface.

Stripping, in which water, through some mechanism, causes the bond between aggregate and asphalt to be diminished, is the reverse of adhesion. Several mechanisms of stripping have been advanced. These mechanisms, summarized by Majidzadeh and Sanders (3), include detachment, displacement, film rupture, and pore pressure. Detachment results when the asphalt cement, with no obvious break in the continuity of the coating, is separated from the aggregate surface by a thin film of water. Displacement occurs where there is a discontinuity or break in the asphalt coating and the aggregate, asphalt, and free water are all in contact.

Film rupture may occur when adhesion of the asphalt cement is not uniform over the entire aggregate surface and the action of traffic causes the coating to break or rupture at points of weakest bond. When ruptured, the asphalt film takes the form of lowest potential energy by retracting to spherical globules. Pore pressure may cause hydraulic scouring to take place in a saturated pavement where the impact of a tire presses water into the pavement surface in front of it and then as the tire leaves the spot the water is sucked out. This water movement facilitates stripping the coated aggregate, and any dust or particulate matter mixed with the surface water assists by abrading the asphalt films.

It is obvious from the foregoing discussion that no single, simple explanation of the stripping process will suffice for all cases. Each of the mechanisms, individually or in combination, may be operating in a given instance of stripping.

STRIPPING TESTS

Stripping tests may be divided into 2 groups, depending on the type of bituminous mixture. The first group, related to appraising materials for layered systems of construction, comprises tests on uncompacted single-size aggregate particles coated with asphalt. The second group includes tests on compacted samples or specimens of a bituminous mixture, which represent a hot-mix, hot-laid, or road-mix type of paving material.

Coated-Aggregate Tests

Tests devised for layered system materials have a common procedure. The aggregate to be evaluated is usually 1 size; commonly, it passes a $\frac{3}{8}$ -in. sieve and is retained on a $\frac{1}{4}$ -in. or No. 4 sieve. The aggregate is coated with the asphalt material, subjected to the effects of distilled water, and then evaluated to ascertain the percentage of asphalt coating still adhering to the aggregate.

Various methods of subjecting the coated aggregate to the effects of water such as dynamic immersion, static immersion, and boil or chemical immersion techniques are used in these tests (4). The amount of stripping is determined by visually estimating the percentage of the total area that remains coated with asphalt. ASTM D 1664 specifies evaluation by visual examination, and the estimate is reported as either above or below the 95-percent-coated level (5).

In the past, considerable research effort has been expended to develop a more quantitative method to measure the amount of stripping that occurs (4). These procedures have used radioactive isotope tracers, lithium tracer-salt, dye adsorption, and leaching. Generally, these tests measured a change in some property of the coated aggregate material after stripping occurred. None of these tests has been widely accepted.

Compacted Mixture Tests

Tests on compacted mixtures measure change in a physical property of the mix that is caused by the effects of water. This change in physical property is then related to stripping effects of water on the bituminous mixture. Several test methods that have been developed include immersion-compression tests, laboratory test tracks, vertical swell tests, abrasion weight-loss tests, and sonic vibration tests (4). Although all of these tests have their merits, the immersion-compression test has been the only one standardized by ASTM (6).

The following are some advantages attributed to tests of compacted bituminous mixtures over stripping tests of coated aggregate particles:

1. Test results are in quantitative terms (that is, some change in a physical property of the mixture is measured),
2. Compacted test specimens represent the actual bituminous mixture that will be used in highway construction, and
3. Laboratory specimens are subjected to water action in a coherent mass, which more nearly simulates actual field conditions (7).

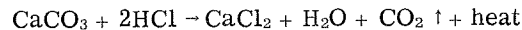
These advantages are offset somewhat by the necessity for more elaborate test equipment and by lengthy and laborious test procedures.

TEST DEVELOPMENT

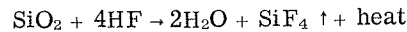
Despite the large number of tests already devised to study the effects of water on coated aggregate particles and compacted mixtures, an examination of the technical literature revealed continuing research in this area (4). Presently, there is no "standard" test to directly evaluate in a quantitative manner the amount of stripping that occurs when asphalt-coated aggregates are subjected to the detrimental action of water. There appears to be a definite need for such a test.

Existing tests were either too complex for routine testing or had questionable accuracy because results were based on the operator's visual judgment. The general procedure proposed for this study was to obtain a measure of the surface area of an uncoated aggregate sample and then, by using a similar asphalt-coated sample, to obtain a measure of the surface area stripped after exposure to water action. The ratio of these measured quantities, that is, the stripped value divided by the uncoated value, was considered a quantitative criterion of the amount of stripping that occurred.

If an aggregate sample is contacted with a suitable reagent, the resulting chemical reaction between the acid and the exposed surface minerals of the aggregate will liberate a gas as well as a certain amount of heat. For example, when hydrochloric acid (HCl) is added to limestone, which is predominantly calcium carbonate (CaCO₃), the following reaction occurs:



If hydrofluoric acid (HF) is added to a predominantly siliceous aggregate, one that is predominantly silicon dioxide (SiO₂), a similar type of reaction occurs:



It was reasoned that the amount of these reaction products should be proportional to the exposed aggregate surface area. If the reaction is confined, the generated gas creates a certain amount of pressure that is easily measured and can be used as a determinant for surface area. With proper selection of reagents and reagent concentrations, asphalt, because it is a relatively inert substance, will not enter into the reaction and will not contribute to the created gas pressure.

Reagents

Hydrochloric and hydrofluoric acids were found to be suitable for this investigation. A suitable reagent was considered to be one that, when placed in contact with an aggregate surface, would react to create a measurable gas pressure and would not react with asphalt cement. Reagent strength or concentration such that the chemical reaction would not deeply etch the aggregate surface was desired.

There was concern that a strong reagent, with sufficient time to react, would not only dissolve the exposed surface molecules of the aggregate, but also would continue to react and undermine adjacent asphalt-coated areas of the aggregate surfaces. If this occurred, the resultant increase in gas pressure would cause considerable error in determining the exposed aggregate surface area. This problem was minimized by using the weakest reagent solution that would create a measurable change in gas pressure. This change in pressure in a minimum amount of time was measured.

Preliminary laboratory work indicated that CaCO₃ in limestone would react in the desired manner when about 1.0 normal HCl was used. A 100-g sample of aggregate, when reacted with 200 ml of 1.0 normal HCl acid solution, created between 4 and 10 psi of gas pressure. The acid solution was depleted in about 10 min of reaction time at normal laboratory temperature.

For aggregate composed mainly of SiO₂, about 23.0 normal HF was required to obtain the desired reaction and a measurable gas pressure. This reaction creates

silicon tetrafluoride gas (SiF_4). Both the acid fumes and SiF_4 gas are highly toxic, so all work with the hydrofluoric acid was carried out in a well-ventilated fume hood with appropriate safety equipment.

Mixed composition aggregates are those containing appreciable amounts of both CaCO_3 and SiO_2 as well as other constituents. Preliminary work indicated that a mixture of HF and HCl would create a measurable gas pressure when reacted with aggregate of mixed composition. A 200-ml acid solution used with this type of aggregate was composed of 27 ml concentrated HF, 54 ml concentrated HCl, and 119 ml of distilled water.

Equipment

The proposed method of testing required the measurement of the gas pressure generated during a chemical reaction. Because the temperature of the reaction affects the volume of the gas, it was necessary to measure and record simultaneously the pressure and temperature generated during the reaction. The apparatus developed to contain the reaction and measure the reaction products was essentially a modified 6-quart stainless steel pressure cooker equipped with suitable instrumentation to monitor and record the desired quantities evolved.

A dual-arm recorder was used to record both temperature and pressure in the pressure container. A pressure transducer with a range of 0 to 30 psig and a thermistor (linked to a scanning telethermometer) were mounted on the lid of the container and connected to the recorder (Fig. 1). With this equipment, pressure in the container could be determined to the nearest 0.025 psig and temperature to the nearest 0.5 C. This sophisticated instrumentation for measuring and recording the pressure and temperature was not absolutely necessary. A simple manometer or pressure gauge and a thermometer, suitably graduated, could be used and observers could record the values at stipulated time intervals.

The pressure transducer was centrally mounted on the removable top of the pressure vessel. The temperature probe cavity extended through the top about 1 in. and was just large enough in diameter to contain the thermistor. A stainless steel pressure release valve also was mounted on the top. This valve was used in calibration of the pressure transducer and to release the pressure in the vessel at the completion of the test. Details of the pressure container are shown in Figure 2.

Because the pressure vessel was to be operated as a closed system, it was necessary to provide a means of adding the acid to the aggregate without changing the ambient pressure. A 250-ml stainless steel beaker was attached by a threaded joint to a rod extending through the body of the pressure vessel. The rod opening was sealed by using neoprene O-rings both inside and outside the wall of the pressure vessel. A handle was attached to the exterior end of the shaft. To inundate the aggregate specimen the beaker was positioned upright and filled with 200 ml of the acid solution, and the handle was turned until the contents of the beaker were poured into the plastic container holding the aggregate sample.

Because of the corrosive nature of the reagents being used, stainless steel was used for all the component parts of the pressure apparatus exposed to the acid solutions and gases generated during the tests. To reduce contact between the acid solutions and the pressure vessel, a small polyethylene container was placed inside the vessel to hold the aggregate-acid mixture during the reaction period. The size of this container was such that a 100-g aggregate sample would be completely inundated by the 200-ml acid solution.

INVESTIGATIVE PROCEDURES

To compare the surface reaction procedure with other methods of evaluating stripping, we conducted a series of stripping tests by using carbonate and siliceous aggregates from various Oklahoma sources. Static immersion and dynamic immersion stripping tests were performed and the results collated with those obtained from the surface reactions test.

Materials

The asphalt cement used in this study (penetration grade 85-100) was chosen because it is a common binder used in asphalt pavement construction in Oklahoma. Selection of the mineral aggregates was based on a research study conducted by the Oklahoma Department of Highways (8). This study indicated that the incorporation of small quantities of polish-resistant siliceous aggregates would improve the skid resistance properties of their standard paving mixtures. Eleven different sources were sampled and the aggregates included 3 types of limestone, 3 types of sandstone, 1 chert, and 4 types of gravel. These aggregates were named for the town adjacent to the source location. They are identified for insoluble residue, specific gravity and absorption, geologic unit and age (period), and general aggregate classification in Table 1.

Sample Preparation

We sieved the material from each of the respective sources to obtain approximately 2000 g of aggregate passing the $\frac{3}{8}$ -in. sieve and retained on the $\frac{1}{4}$ -in. sieve. We then washed, oven-dried, and quartered the aggregate to obtain representative samples of approximately 100 g each. The dry aggregate was weighed (100.0 ± 0.2 g) and placed in large aluminum moisture boxes for storage until required in testing. Ten samples of each of the various aggregates were prepared in this manner. Six of these samples, i.e., duplicate samples, were used in performing the static immersion and dynamic immersion stripping tests and the surface reaction test. The remaining samples were used for specific gravity and absorption tests and for checks on results of the other stripping tests.

Samples for the static immersion and dynamic immersion tests were coated in the following manner. The aggregate and asphalt cement were heated to 120 C before coating. To each of the 100-g samples of aggregate, 6 g of asphalt was added. The mixture was stirred and manipulated with a spatula until each rock was coated with asphalt. A hot plate was used to heat the mixture, as required to achieve 100 percent coating. About 3 min of hand-mixing time ordinarily was required. The individual particles of asphalt-coated rock were placed in a pan of cold distilled water after mixing. Cold water prevented the coated rocks from sticking together. This sample preparation and coating procedure is in accordance with ASTM D 1664 (5).

Static Immersion Stripping Test

After cooling in the chilled water, the coated sample was placed in a glass jar and covered with 600 ml of distilled water. The jar was capped and placed, partially submerged, in a 25 C water bath and left undisturbed for 18 hours. The amount of stripping was then visually estimated by using the ASTM standard procedure. To facilitate this evaluation, a comparison chart was prepared. This chart was made by tracing the outline of typical aggregate particles inside a circle the same diameter as the glass jar in which the samples were immersed. A series of these tracings were made and the cross-sectional areas of the aggregate particles in each were darkened to represent different amounts of coated surface (ranging from 50 to 100 percent coated).

The chart was placed flat on the table adjacent to the jar sample to be evaluated. The top was removed from the jar, and any film of asphalt on the surface of the water was removed by skimming with a paper towel. The chart was moved until, by visual observation of the sample from above and through the water, the sample matched 1 of the covered or darkened areas of the comparison chart. It was difficult to estimate the area covered with asphalt any closer than to the nearest 5 percent. Therefore, duplicate test results were averaged and then rounded to the nearest 5 percent.

No stripping of any of the various aggregates was observed when they were coated with asphalt cement and subjected to the static immersion stripping (SIS) test at 25 C. With a longer period of immersion or higher immersion temperatures, we anticipated that some stripping of the aggregates would occur. Therefore, the SIS (25 C) samples were then placed in a 60 C water bath and left undisturbed for 18 hours. The amount of stripping (which was considerable) was then visually estimated by using the comparison chart as before.

Figure 1. Surface reaction test equipment.

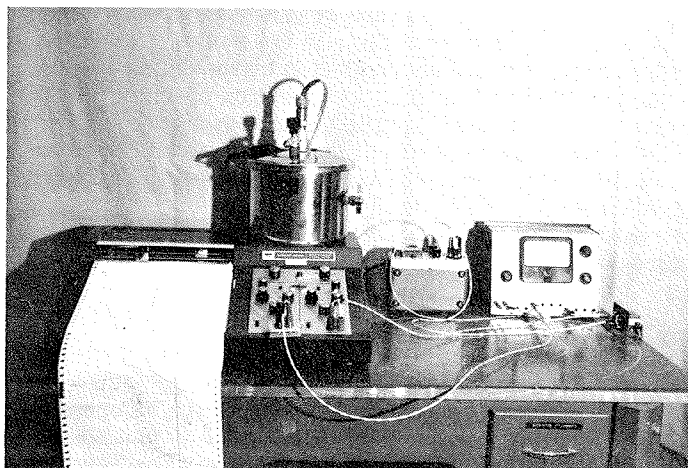


Figure 2. Details of pressure container device.

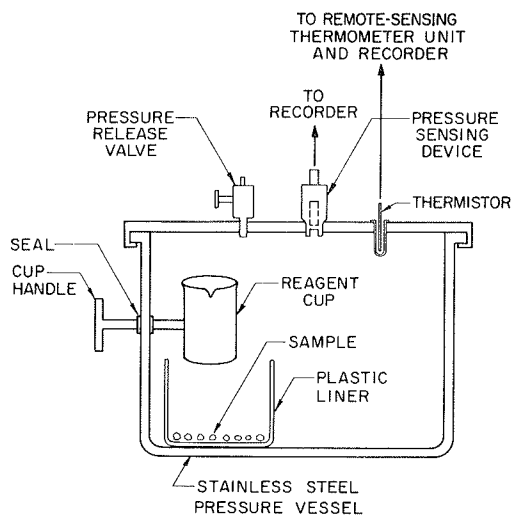


Table 1. Aggregate identification and properties.

Sample	Insoluble Residue (percent)	Bulk Specific Gravity	Absorption	Geologic Unit and Period	General Classification
Cooperton	1.2	2.67	0.8	Kindblade limestone, Ordovician	Limestone
Hartshorne	23.3	2.66	1.0	Wapanucka limestone, Pennsylvanian	Limestone (partly siliceous)
Stringtown	72.8	2.57	0.5	Wapanucka limestone, Pennsylvanian	Siliceous limestone
Cyril	59.2	2.64	0.9	Rush Springs, Permian	Calcareous sandstone
Keota	96.3	2.48	2.4	Bluejacket, Pennsylvanian	Siliceous sandstone
Onapa	92.1	2.47	4.1	Bluejacket, Pennsylvanian	Siliceous sandstone
Asher	99.8	2.46	3.2	Wellington-Admire, Permian	Chert gravel
Broken Bow	98.3	2.69	1.3	Alluvial Deposit, Quaternary	Siliceous gravel
Gore	97.9	2.68	0.6	Alluvial Deposit, Quaternary	Siliceous gravel
Hugo	99.0	2.52	1.8	Terrace Deposit, Quaternary	Chert gravel
Miami	95.4	2.56	1.2	Boone, Mississippian	Chert

Dynamic Immersion Stripping Test

To accelerate the stripping action of water on coated aggregate we constructed a dynamic stripping device. A literature review showed many previous investigators had used a dynamic immersion stripping (DIS) test to evaluate the effects of water on asphalt-coated aggregate. The method originally used by Nicholson (1) was followed in this study. Aggregate was coated with asphalt cement as in the SIS test and subjected to 4 hours of water agitation. A visual estimate of the amount of stripping was made at the end of 1, 2, and 4 hours. An apparatus was designed and built to hold 6 glass jars of approximately 1 quart capacity. The device was rotated about a horizontal axis at about 40 rpm. This caused the coated aggregate sample to fall from one end of the jar through the water to the other end during each revolution.

Preliminary tests using the DIS device revealed that the nonstripping aggregate (Cooperton limestone) would partially strip when the sample was tumbled continuously for 4 hours. The siliceous aggregate particles also retained more than 50 percent of their coating at the end of 4 hours of tumbling. Therefore, a 4-hour DIS test period was chosen, with the temperature maintained at about 20 C, which was the normal laboratory temperature.

The DIS test was intended to induce stripping by subjecting each aggregate sample to the same amount of agitation in a water medium, and then to compare the visually estimated retained coating with a measured amount as determined by the surface reaction test.

Surface Reaction Test

Half of the aggregate samples used in the surface reaction test (SRT) previously had been coated with asphalt cement and partially stripped in the DIS test. An uncoated duplicate sample of the aggregate was immersed in distilled water at the same time as the DIS specimen was immersed. At the end of the DIS test, the partially stripped and uncoated samples were dried by blotting with paper towels and spread out in pans to air dry for approximately 24 hours before the SRT.

We wanted to perform this test on oven-dried materials but when the partially stripped aggregate samples were oven dried at 100 C, the remaining asphalt cement diffused and completely recoated the stripped aggregate surfaces, so oven drying was eliminated and the samples were simply air dried before testing.

Each test required 200 ml of acid solution. Because duplicate samples (2 uncoated and 2 partially coated samples of each of the various aggregates) were tested, a litre of acid solution was prepared for each series of aggregate samples. The acid test solutions were prepared by using reagent grade acids and deaired distilled water. All proportions of these acid solutions were calculated on a weight basis.

The 200 ml of acid solution to be used in each test was measured with a graduated cylinder and placed in a 250-ml Nalgene jar. The weight of acid solution in each jar was determined and its density, in g/ml, was calculated. This density and weight determination was used as a check to obtain equal strengths of solution for each test. The balance of the litre of solution originally prepared was retained for titrations. The actual normality of the acid was determined by titration against a known weight of sodium carbonate by using methyl orange as an indicator (9). All work with the acid solutions containing HF was carried out with polyethylene or polystyrene containers.

Procedure—Before initiating a test, samples to be tested, acid solutions, and the pressure container were placed in a fume hood and brought to a constant temperature of 20 C. The recording equipment was placed adjacent to the fume hood. This test procedure was used.

1. The pressure release valve was opened and the lid of the pressure vessel removed.
2. The sample to be tested was placed in the plastic container and the container was positioned inside the pressure vessel.
3. The beaker was installed in the pressure vessel, leveled, and 200 ml of acid solution was poured into the beaker.

4. The lid was placed on the pressure vessel, the pressure recording pen was set to zero, and the chart drive of the recorder was started.

5. The pressure release valve was closed and the exterior handle turned to pour the acid from the beaker. A stopwatch was started at the instant the acid was poured onto the aggregate sample.

6. The reaction was monitored by observing the pressure and temperature traces on the recorder.

7. At the completion of the test, which ordinarily lasted 5 min, the pressure release valve was opened and the recorder chart drive stopped.

8. After the pressure was released, the top of the pressure vessel was removed and the acid beaker taken out. The sample was removed and the reaction of the acid solution and sample was terminated. For samples tested with HCl this was accomplished by flooding the mixture with tap water. For samples tested with HF the reaction was stopped by slowly adding a sufficient amount of calcium oxide slurry to deplete the HF in the mixture. Methyl orange indicator was used to determine when the solution was neutralized.

9. The acid beaker and plastic sample container were then washed and dried before starting another test.

Stripping Calculation—The pressure-temperature curves plotted on the recorder chart were analyzed. A horizontal line was drawn on the chart paper for each 15 sec of elapsed reaction time. The pressure and temperature readings were scaled from the chart paper and tabulated. Pressures were adjusted to 20 C for comparative stripping calculations. This adjustment of pressures was necessary because of the slightly different operating temperatures and the higher temperatures created by some of the reactions.

The surface area exposed was considered proportional to the change in pressure over a certain time interval. For limestone aggregates and aggregates of mixed composition, the change in pressure from 0.25 to 1.5 min of reaction time was used. The reaction between the siliceous aggregates and the HF solution was slower or less violent and required a longer reaction time (0.25 to 5.0 min) to obtain a significant pressure difference. The effect of inertia on the pressure transducer operation and the recorder chart pen response were the primary reasons for using the initial gas pressure value at 0.25 min of reaction time. The reaction time for the final pressure value was that required to obtain a measurable pressure without deeply etching the exposed aggregate surfaces.

A drawing typical of the strip-chart recorder tracing of the pressures obtained for an uncoated limestone aggregate sample and a partially coated limestone aggregate sample is shown in Figure 3. The initial pressure reading, taken at 15 sec of reaction time, is shown as P_1 ; the final pressure reading, taken at 90 sec of reaction time, is shown as P_2 . The retained coating of asphalt was calculated as follows:

$$RC = 100 - \left(\frac{\Delta P_s}{\Delta P_u} \right) 100$$

where

RC = percent retained coating of stripped sample,
 $\Delta P_s = P_{2s} - P_{1s}$ = change in pressure for stripped sample, and
 $\Delta P_u = P_{2u} - P_{1u}$ = change in pressure for uncoated sample.

Relation Between Surface Area and Change in Gas Pressure—The hypothesis that generated gas pressure is proportional to exposed surface area was examined by using small disks cut from samples of the Cooperton limestone material. Large pieces of rock, weighing from 2 to 5 kg, were obtained at the quarry where this aggregate is produced. With a concrete saw, these rocks were sawed into slabs ranging from 5 to 10 mm in thickness. A diamond core drill with an inside diameter of 19.0 mm was used to cut uniform diameter disks from these slabs of limestone rock. Each disk was numbered and its average thickness determined by using a micrometer dial gauge. The

surface area of each disk was calculated and the disks were then divided into five different surface area groups. These groups had approximate surface areas of 6 000, 12 000, 18 000, 24 000, and 30 000 mm². Duplicate groups of approximately the same surface area also were prepared. Each group of disks was reacted with a HCl solution by using the test procedure previously outlined, and the resulting gas pressures were determined. A graph of the surface area of each disk sample with the corresponding increase in gas pressure for a reaction time of from 15 to 90 sec is shown in Figure 4.

Each plotted point on the figure is the average value for duplicate disk samples. A good linear relationship between disk surface area and change in gas pressure is noted. The increase in gas pressure was approximately 0.059 psi (0.041 g/mm²) for each 1 000 mm² of disk surface area. Verification tests on other aggregates used in this study have not been completed, but a similar relationship of surface area to gas pressure increase is anticipated.

DISCUSSION OF RESULTS

The results of the various stripping tests on each of the respective aggregates are given in Table 2. Each of the tabulated values of the percent of retained coating is the average of the values obtained from duplicate test samples.

None of the aggregates exhibited any stripping when tested according to standard ASTM procedure at 25 C. When the SIS test was made more rigorous by increasing the immersion temperature to 60 C, considerable stripping was evidenced. The visually estimated retained coatings ranged from 95 percent for the Hugo chert gravel to 40 percent for the Gore siliceous gravel.

According to ASTM D 1664 (5), the static immersion stripping results at 25 C are reported as above or below the 95 percent retained coating level. On this basis, each of the 11 aggregates would be rated as having a retained coating above 95 percent. The limitation of this test method is that it does not provide for evaluation below this level. The limitation results from the poor reproducibility obtained when rating the same sample by visual observation. It would appear that little or no useful information regarding the relative stripping tendencies of various aggregates could be obtained from visual evaluation in accordance with the ASTM method.

The DIS test results are reported for the various periods of tumbling used in the investigation. At the end of 1 hour, all of the aggregate samples retained 90 percent or more of their original asphalt coating and, at the end of 2 hours, most of the samples had greater than 85 percent retained coating. After 4 hours of tumbling the estimated retained coatings ranged from 85 percent for the Cooperton limestone, Keota sandstone, and Onapa sandstone to 65 percent for the Gore siliceous gravel.

A comparison of the retained coating percentages of each aggregate for the SIS test at 60 C and the 4-hour DIS test shows little correlation. Mathews, Colwill, and Yuce (10) also reported little correlation of test results from SIS and DIS tests performed on 16 different types of aggregates.

It should be repeated that, in the SRT, both uncoated and partially coated or stripped samples of each aggregate were tested. The partially coated samples were those resulting from the 4-hour DIS test. The change in gas pressure (over the selected time interval) exhibited by duplicate coated and uncoated test samples agreed closely. In most cases, these corresponding differential pressure (ΔP) test values differed by less than 0.014 g/mm². The average ΔP values for the coated and uncoated samples of a given aggregate were used to calculate the percentage of asphalt cement coating retained, as has been previously discussed. The SRT retained coating percentage for each aggregate is given in Table 2.

The measured retained coatings varied from 93 to 54 percent. The limestone aggregates (Cooperton, Hartshorne, and Stringtown) with 89 percent retained coating had the highest group average. The sandstone aggregates (Cyril, Keota, and Onapa) averaged 63 percent; the gravels (Asher, Broken Bow, Gore, and Hugo) averaged 68 percent.

These results show the same trend of resistance to stripping as was obtained from a series of immersion-compression tests on compacted mixtures containing the same aggregates. These tests were performed on compacted specimens of a standard

Figure 3. Typical SRT pressure-time curves for limestone aggregate.

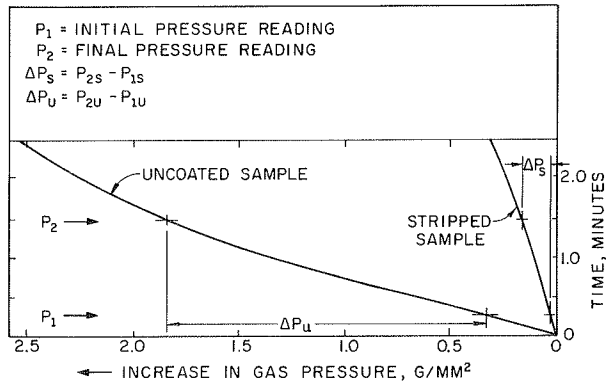


Figure 4. Original disk surface area versus gas pressure increase, Cooperton limestone.

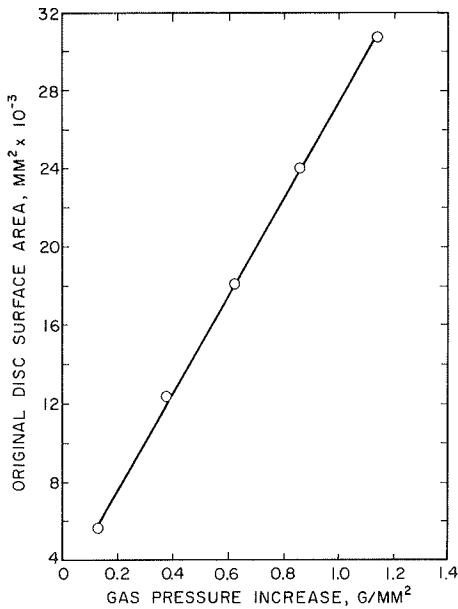


Table 2. Results of SIS and DIS tests and SRT.

Aggregate	Static Immersion Retained Coating (percent)		Dynamic Immersion Retained Coating (percent)			Surface Reaction Retained Coating	
	77 F	140 F	1 Hour	2 Hours	4 Hours	Percent	Variation (percent) ^a
Cooperton	100	85	95	90	85	90	±0.5
Hartshorne	100	75	95	90	75	85	±0.5
Stringtown	100	65	95	90	85	93	±0.7
Cyril	100	60	90	80	75	64	±0.7
Keota	100	50	95	90	80	56	±0.0
Onapa	100	50	95	90	85	68	±0.8
Asher	100	90	95	90	80	74	±3.4
Broken Bow	100	90	95	90	70	54	±3.6
Gore	100	40	90	85	65	65	±2.2
Hugo	100	95	95	90	80	78	±0.5
Miami	100	70	95	85	75	60	±3.5

^aVariation based on maximum and minimum values of duplicate tests.

Oklahoma Department of Highways surface course mixture in which these aggregates comprised the coarse aggregate fraction. The retained strength values from these immersion-compression tests also indicated that the limestones were better than the gravels, which were in turn better than the sandstones.

A measure of the reproducibility achieved with the SRT was obtained by using the results of the 4 tests performed on each type of aggregate. The smallest and largest of the individual ΔP values from each SRT series were used to calculate a maximum and minimum percent of retained coating. The difference between these maximum and minimum values is given in Table 2 as the variation in retained coating for each aggregate. A variation of less than 1 percent is noted for 7 of the aggregates, with the largest variation being only 3.6 percent (Broken Bow gravel).

There was low correlation between visually estimated retained coatings of the DIS tested samples and the SRT measured values for the same samples. This emphasizes some of the problems associated with the visual estimation procedure. Several factors affect the visual estimation of the amount of retained asphalt coating on the aggregate including color of the aggregate, visual perception limited to the plan or 2-dimensional view, undetectable microscopic breaks in the asphalt in a state of "detachment," and operator error or bias.

The color of the aggregate influences the operator's estimation of coating, with lighter colored aggregates being rated lower than darker colored aggregates when both may have the same amount of asphalt-coated surface. This is due to the greater contrast between the black asphalt cement coating and the light-colored aggregate, which causes the operator to assign a lower value of retained coating to the lighter colored material.

This color factor may be the reason that the light-colored Cooperton and Hartshorne limestones were visually rated about 5 to 10 percent lower than the SRT results indicated. Conversely, the dark-colored Keota and Onapa sandstones were visually rated about 20 percent higher in retained coating than the SRT results indicated.

The standard method of visual estimation considers only the exposed aggregate surfaces that appear in plan view and assumes that the stripping evident in a 2-dimensional scene is representative of that throughout the sample. This assumption may or may not be valid and depends, to a large extent, on the shape and orientation of the aggregate particles. In the SRT, however, the acid solution reacts with all exposed surfaces, and the results are indicative of the total aggregate surface area that has been stripped of its asphalt coating.

By unaided visual examination of stripped aggregate particles there appeared to be no disturbance in the asphalt surface where the coating remained in place. However, under a 30-power microscope, numerous pinholes or small breaks in the asphalt coating could be seen. After the SRT, there was evidence of etching or surface reaction at these small discontinuities.

Perhaps the most important factor in the visual estimation method is the operator. Repeatability of visual estimates of the aggregate surface area coating was examined by Brown, Sparks, and Marsh (11). In this work, 4 experienced operators were used to evaluate 36 different test samples of partially coated aggregates. Their average range in estimated retained coatings was 16 percent; their differences varied from 2 to 32 percent. Roediger (12) reported the results of a cooperative stripping test visual estimation project in which 10 laboratories estimated the amount of stripping evidenced by 22 different samples. Their visual estimates of percent of retained coating for the same test specimen ranged from 4 to 44 percent, with an average difference between laboratories of 24 percent. Different operators were noted to agree more closely when the amount of stripping was small.

All of these visual factors were eliminated when stripping was evaluated by SRT. The SRT results in a more precise and quantitative determination of exposed surface area. A high degree of reproducibility can be expected because the results of replicate surface reaction tests indicate less than 4 percent variation. Because the results are measurable rather than estimated quantities, they are more indicative of the relative stripping tendencies of various asphalt-aggregate mixtures and better correlation of these laboratory values with field performance of the materials should be possible.

CONCLUSIONS

The SRT provides a quantitative measure of total exposed surface area on a stripped mineral aggregate sample and eliminates the problems associated with visual estimation techniques.

The results of the SRT are reproducible with a minimum amount of variation between duplicate test sample values.

The SRT is simple and straightforward and usually can be performed in less than 10 min. The required equipment is neither expensive nor complicated.

The use of highly corrosive and toxic reagents is a disadvantage of this procedure. However, with proper laboratory equipment and safety precautions this drawback can be minimized.

With proper verification methods, the test procedure could be applied to surface area measurements of a variety of materials that have been crushed or broken down into irregularly shaped fragments.

The standard SIS test showed no stripping in the asphalt-aggregate combinations used and would be of little or no value in predicting the relative stripping tendencies of such mixtures.

Generally, the asphalt-aggregate mixtures containing the limestones were more resistant to stripping than those containing the gravel and sandstone type aggregates. Within the group of gravels, the aggregates with appreciable amounts of quartz (Broken Bow and Gore) were more susceptible to stripping than those composed predominantly of chert.

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DISCUSSION

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I wish to congratulate the authors of this paper on their brilliant attempt to put

forward a new quantitative test to evaluate the stripping action of asphalt. It is well known that the standard stripping test, ASTM D 1664 (5), is highly qualitative in nature and subject to personal errors. Therefore, a better procedure is necessary to make this test quantitative and to eliminate personal judgment.

The test procedure outlined in this paper is based on the principle that a suitable reagent (such as an acid) reacts with the mineral aggregate and liberates a gas as part of the reaction. The gaseous pressure developed is considered to be proportional to the exposed surface area of the aggregate. Therefore, the chemical composition of the aggregate surface is an important factor. It appears to me that the authors do not take into consideration the presence of non-gas-producing reactions when acid comes in contact with aggregate surface. I think that the direct evaluation of amount of acid reacted with the aggregate surface, under a suitable set of experimental conditions, may be more fundamental, simpler, and a better approach to the problem. Therefore, I wish to share some observations made on evaluating stripping by an elementary acid-base titration.

THEORY

If

S_a = total surface area of uncoated aggregate,

V_a = volume of standard acid reacted for a certain time with S_a ,

S_b = total surface area of the aggregate coated with asphalt, and

V_b = volume of the same standard acid reacted for the same interval of time with S_b ,

and, if after stripping

S'_a = surface area of the uncoated aggregate,

S'_b = surface area of the aggregate coated with asphalt, and

V'_a = volume of the same standard acid reacted for the same interval of time,

then we know that

$$S'_a + S'_b = S_a \quad (1)$$

Dividing Eq. 1 by S_a gives

$$\frac{S'_a}{S_a} + \frac{S'_b}{S_a} = 1 \quad (2)$$

and

$$V'_b = \frac{S'_a}{S_a} \cdot V_a + \frac{S'_b}{S_b} \cdot V_b \quad (3)$$

Since $S_a \approx S_b$

$$V'_a = \frac{S'_a}{S_a} \cdot V_a + \frac{S'_b}{S_a} \cdot V_b \quad (4)$$

That is to say

$$V'_b = \frac{S'_a}{S_a} \cdot V_a + V_b \left[1 - \frac{S'_a}{S_a} \right] \quad (5)$$

Therefore,

$$\frac{S'_a}{S_a} = \frac{V'_b - V_b}{V_a - V_b}$$

or $\frac{S'_a}{S_a}$ = fraction of surface area stripped.

PROCEDURE

The limestone aggregate passing a $\frac{3}{4}$ -in. sieve and retained on a $\frac{1}{2}$ -in. sieve was washed with distilled water and oven dried at 110 C for 2 hours. Then the aggregate was allowed to cool to room temperature. Two hundred grams of the oven-dried aggregate were heated to 150 C and coated with asphalt at 150 C. The asphalt content in all mixes was maintained at 4.5 percent by aggregate weight. The coated aggregate was allowed to cool to room temperature.

Test 1

Two hundred grams of uncoated aggregate was reacted with 200 ml of N/10 HCl for 5 min. By titrating the excess acid with N/10 sodium carbonate using methyl orange as indicator, the volume of the acid reacted was obtained (V_a ml).

Test 2

The same procedure used in test 1 was repeated with 200 g of aggregate coated with asphalt (V_b ml).

Test 3

The same aggregate used in test 2 was washed with distilled water 3 times to free it from acid and boiled for 15 min in 400 ml of distilled water. The water was drained off quickly and the material was allowed to cool for about half an hour and test 1 was repeated (V'_b ml). Because the reaction of HCl with limestone is fast, a weak acid such as oxalic acid is found to be more suitable. The reaction of 1N-oxalic acid with limestone reaches completion in about 15 min because of the formation of acidic nonsoluble calcium oxalate on the exposed surface. The oxalic acid used up may be determined with N/10 sodium hydroxide with phenolphthalein as an indicator.

This procedure does not require elaborate and expensive apparatus, although a mixture of HCl and HF acids should be used for siliceous or mixed aggregate.

AUTHORS' CLOSURE

We appreciate Fernando's discussion. He illustrates another approach to the quantitative evaluation of stripping. Lortscher, Snyder, and Filbert (13) reported a similar approach to evaluation of stripping of limestone aggregate by reacting the aggregate with dilute HCl and measuring the depletion rate of the acid by titration. We hope that Fernando also will publish test results to confirm the validity of the SRT.

REFERENCE

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