Effect of Aggregate Properties on Stripping

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A laboratory investigation was conducted to relate some measurable aggregate properties to the stripping propensity of a mix of aggregate and asphalt cement. Several different types of aggregates were used in this study; each was characterized in terms of its physical properties, such as pore volume and surface area, and its chemical and electrochemical surface properties. Stripping propensity was determined by using a boiling water test. Under certain conditions, aggregates that have a relatively high surface electrical potential and those that impart a high pH value to water in contact with their surface were found to exhibit a high susceptibility to stripping. These results were interpreted in terms of electrochemical properties of the aggregate surface. It was also observed that the effectiveness of some types of antistripping additives was sensitive to the pH of water in contact with the aggregate and that curing the asphalt-aggregate mixture and precoating the aggregate improved stripping resistance considerably.

Stripping of asphalt films from aggregate surfaces occurs when there is a loss of adhesion between the aggregate surface and the asphalt cement. This is primarily due to the action of water or moisture. The resulting deterioration can be a serious problem, which causes loss of integrity of the asphalt concrete and subsequent failures that require early and costly maintenance. The primary factors that affect stripping in an asphalt paving mixture include the physical and chemical properties of both the aggregate and the asphalt cement (such as surface characteristics and mineral composition of the aggregate and chemical composition, surface tension, and viscosity of the asphalt). Construction methods and external environmental factors also contribute to stripping. Stripping is, therefore, a complex phenomenon influenced by various factors.

Over the years, a great deal of basic and applied research has been conducted to determine the nature of adhesion and stripping in asphalt paving mixtures (1-9). Several major theories of stripping have been detailed and are summarized by Majidzadch and Sanders (10) and Taylor and Khosla (9). The most widely accepted mechanisms of stripping are detachment, displacement, spontaneous emulsification, and pore pressure.

The rationale for these stripping mechanisms is provided by the mechanical, thermodynamic, or interfacial energy or chemical concepts of adhesion and loss of adhesion, or both. The mechanical concept that explains the stripping mechanisms suggests that the bond strength between the asphalt and the aggregate surface is dependent on a mechanical interlock developed by the penetration of the asphalt into pores and cracks on the surface of the aggregate particle (1). The thermodynamic or surface energy concept involves the wetting behavior of asphalt at the asphalt-aggregate-water-air interface (1, 7, 11–14). The degree to which stripping occurs depends on the interfacial free energy relationships at the aggregate-asphalt-water-air interface. The chemical concept involves adsorption of asphalt on aggregate surfaces and chemical reactions between the adsorbed asphalt compounds and the constituents of the aggregate surface (7, 8, 15). Herein, water solubility of the asphalt-aggregate bond is the main factor affecting stripping.

It is generally thought that the primary mechanism responsible for stripping involves the displacement by water of an asphalt cement film from the aggregate surface. The actual role of water in stripping is, however, still not entirely understood. Also, it has been difficult to relate quantitatively stripping potential to materials selection and mixture design parameters.

The purpose of this research work was to study the nature of stripping in asphalt paving mixtures and to determine the importance of the physical and chemical properties of the aggregate in the mix.

Table 1 gives a summary of the physicochemical properties of asphalt, aggregate, and the asphalt- aggregate mixture that might influence stripping, according to different mechanistic stripping theories. Although all of these properties are involved, this research was primarily concerned with aggregate properties such as surface area, porosity, and chemical and electrochemical properties.

Several different types of aggregates were selected for this study, and physical properties, such as surface area and pore volume, were measured. Changes in the pH of water after the addition of aggregate and in the surface charge of aggregates in water were also measured. These measurements gave some information on the chemical properties of the aggregate surface. For each aggregate characterized (in terms of its physical and chemical properties) and evaluated, the boiling water test was performed to determine the stripping propensity of the aggregate when coated with a given asphalt cement (16). If the asphalt separated from the aggregate surface during this test, it was said to have stripped, and it was concluded that the asphaltaggregate bond was weak and had a high stripping propensity.

On the basis of the results of this experimental evaluation and the published findings of other researchers, a mechanism by which stripping might occur was proposed. The observed relationships between the properties of an aggregate and its stripping potential were found to be consistent with the proposed stripping mechanism.

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TABLE 1 PHYSICOCHEMICAL PROPERTIES OF ASPHALT, AGGREGATE, AND MIXTURE INFLUENCING STRIPPING

Material	Physicochemical Property		
Asphalt	Viscosity Surface tension Volatility Relative fraction polar constituents Phenol group concentrations Carboxylic group concentrations Amine group concentrations		
Aggregate	Size and shape Pore volume and size Surface area Chemical constituents at surface Acidity and alkalinity Adsorption site surface density Surface charge or polarity		
Asphalt-aggregate mixture	Pore space fraction filled with asphalt Asphalt adsorption ratio Chemical constituents of adsorbed asphali		

EXPERIMENTAL WORK

Materials

Asphalt

An AC-20 asphalt cement supplied by the Alabama Highway Department was used in this study.

Aggregates

Five aggregates used in Alabama road construction were supplied by the Alabama Highway Department. These aggregates (granite, limestone, dolomite, quartz gravel, and chert gravel) were crushed and screened to ³/₈-in. to No. 4 mesh size.

Antistripping Additives

Three different antistripping additives were used in this study. Two of the additives (Additives 1 and 2) were commercial antistripping additives, and one of the additives was a hydrated lime. In each case, the additive was mixed with asphalt cement by first placing the additive in a glass beaker and then pouring the heated asphalt cement into the beaker. The mixture was then stirred with a hot spatula to ensure thorough mixing.

Procedure

Boiling Water Test

After having been soaked in distilled water for 24 hr and towel dried, 100 g of saturated aggregate was placed in a stainless steel bowl and kept in an oven maintained at 300°F for 1 hr. Next, 5.5 g of asphalt cement, with or without an antistripping additive, was heated at 275°F for 10 min and then poured onto the preheated aggregate. The asphalt content was found to have only an insignificant effect on the boiling water test result when it was above the minimum amount required to completely coat the aggregate (3 to 4 g). The asphalt and aggregate were mixed using a hot spatula for 2 min and then placed in an oven maintained at 300°F for 10 min. After it had cooled to room temperature, the completely coated mixture was placed in

boiling water (250 mL in a 400-mL beaker) over a hot plate. The water was allowed to boil slowly for 10 min, during which time it was stirred using a glass rod for 10 sec at 4-min intervals. The mixture was then kept in the water while it cooled to room temperature. After cooling, the water was drained from the beaker, and the mixture was placed on a paper towel and allowed to dry.

The amount of resultant stripping was determined visually and reported in terms of the observed percentage of asphalt coating retained on the aggregate. A rating board was developed that had 10 intervals representing from 0 to 100 percent of retained coating in order to standardize, as much as possible, the visual evaluation (16).

Pore Volume and Surface Area

The pore volume and surface area of each aggregate (3/8-in. to No. 4 mesh size) were measured using a mercury porosimeter (Autoscan-33 Porosimeter, QuantaChrome Co.). The surface area of an aggregate was calculated from a cumulative pore volume curve measured using the porosimeter and assuming that the aggregate had cylindrical pores open at each end. According to the Washburn equation (17), the surface area is

$$S = (1/\gamma \cos \theta) \int_{0}^{V} P dV \tag{1}$$

where P is compression pressure, V is pore volume, γ is the surface tension of mercury, and θ is the contact angle of mercury over the solid sample. Using the commonly accepted values of $\gamma = 480$ erg/cm and $\theta = 140$ degrees, surface area was calculated by graphically integrating the cumulative pore volume curve (17).

pH Measurements on Aggregate Powder

Powdered aggregate (10 g) was added to distilled water (100 mL) in a beaker and the pH of the water was measured using a pH meter with a glass/calomel electrode. The mixture was stirred with a magnetic stirrer bar, and the pH was recorded over time.

The aggregate powders for this experiment were obtained by first crushing and grinding the aggregates and then screening them to the No. 60 to No. 80 mesh size range in order to increase the surface area.

Surface Charge Measurements

The zeta potentials of the powdered aggregates in water were measured with a Lazer Zee Meter (Model 501, Pen Kem, Inc.). This meter measured electrophoretic mobility to determine the zeta potential of the aggregate particles in water. The sample for this measurement was a mixture of 10 g of aggregate powder (less than 325 mesh size) in 100 mL of distilled water.

DISCUSSION OF RESULTS

Five different aggregates were characterized by measuring their pore volumes and surface areas, the pH values of contacting water, and surface charges in water. The experimental results were evaluated with respect to the effect of (a) pore volume and surface area, (b) pH value of contacting water, and (c) surface charge of aggregate on the stripping propensity of the aggregate as measured by the boiling water test. The results are discussed in the following subsections.

Pore Volume and Surface Area

Five aggregates (granite, limestone, dolomite, chert gravel, and quartz gravel) were tested for surface area and porosity, and their stripping propensity was determined by the boiling water test (Table 2). A low pore volume or surface area suggests a smooth, crystalline surface with low surface roughness. On the basis of purely mechanical considerations—that is, the requirement for large areas of interfacial contact and surface roughness to have good adhesion and interlock—the low pore volume and surface area of granite should imply the existence of low adhesive bond strength with the asphalt cement and high moisture susceptibility.

TABLE 2 PORE VOLUMES AND SURFACE AREAS OF AGGREGATES AND THEIR STRIPPING PROPENSITIES AS DETERMINED BY THE BOILING WATER TEST

	Pore Volume	Surface	Average Pore Size	Percentage Coating After
Aggregate	$(\text{cm}^3/\text{g} \times 10^3)$	Area (m^2/g)	$(mm \times 10^4)$	Boiling
Granite	3.2	0.116	1.10	10
Dolomite	6.5	0.586	0.44	35
Chert gravel	23.0	2.09	4.40	55
Quartz				
gravel	5.4	0.052	4.15	65
Limestone	6.2	0.079	3.14	90

Note: Average pore size = 4 [(Pore volume)/(Surface area)].

A comparison of the effect of different pore sizes was provided by dolomite and limestone (Table 2). Dolomite and limestone were used for this comparison because of the similarity of their chemical and electrochemical properties. Although dolomite had a higher surface area than did limestone, it had a higher stripping propensity because of its smaller pore size. It should be noted that, even though the dolomite had nearly the same pore volume as the limestone, it had about seven times as much surface area, which meant that the dolomite had a smaller pore size. In Table 2, the average pore size for each of these aggregates was estimated by assuming that the pores had a cylindrical shape.

One possible reason for the observed adverse effect of pore size on the stripping propensity of the dolomite and limestone aggregates is that, when an asphalt cement coats a rough surface that has fine pores, air is trapped and the asphalt can hardly penetrate the fine pores. Consequently, only a fraction of the aggregate's apparent surface area might actually be in contact with the asphalt cement. In general, the depth of penetration of the asphalt depends on the size of the pore, as well as the viscosity and surface tension of the asphalt.

Overall, a correlation between the physical properties of an aggregate and its stripping propensity could not be established. The chemical properties of the aggregates considered varied significantly, and it was thought that this had an overriding

effect. As the data in Table 2 indicate, although limestone and crushed quartz gravel had nearly the same physical properties (pore volume and surface area), limestone had a better stripping resistance. The chemical properties of the limestone were much different from those of the quartz gravel, as will be discussed later.

pH of Contacting Water

In 1960 Hughes et al. (3) reported that the adhesion between asphalt and aggregate in the presence of water became weakened when the pH of the buffer solution was increased from 7.0 to 9.0. Scott (7) also reported the same observation for the effect of pH on stripping. In this study, some insight into the effect of pH changes was developed by considering the chemical and electrochemical properties of the aggregate surfaces.

Figure 1 shows the changes in pH values caused by the addition of several different aggregate powders to water. Similar results have been reported for different aggregates by Scott (7). Apparently (Figure 1), limestone and dolomite, which are known to be basic aggregates, caused the pH of the water to rise to a relatively high value. Also, granite, which is known to be acidic, reacted with water, leading to a gradual increase in the pH of the system. The silicate lattice of the granite surface reacted with water to impart excess hydroxyl ions as follows:

$$- \text{Si} - \text{O} - \text{Na} + \text{H}_2 \text{O} \rightarrow - \text{SiOH} + \text{Na}^+ + \text{OH}^-$$
 (2)

Equation 2 illustrates a typical hydrolytic reaction of the salt of a weak acid.

To assess the sensitivity of stripping to changes in the pH of water in contact with the aggregate surface, a series of boiling water tests was performed using water of different pH values. The pH of the water was modified by adding HCl or NaOH solution. As shown in Figures 2 and 3, changes in the pH of the water had significant effects on stripping. Stripping became

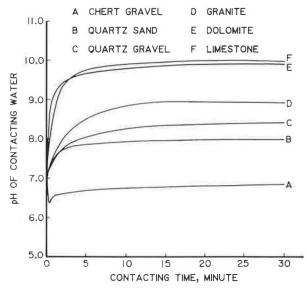


FIGURE 1 Changes in pH of water in which aggregates were immersed.

more severe as the pH value increased from 2.0 to 7.0 for the granite aggregate (Figure 2) and from 3.0 to 13.0 for the crushed chert gravel (Figure 3). This result indicates that a significant change in the pH of water in contact with the aggregate surface could cause stripping to occur.

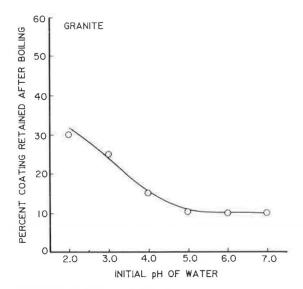


FIGURE 2 Effect of pH of water used in the boiling water test on granite.

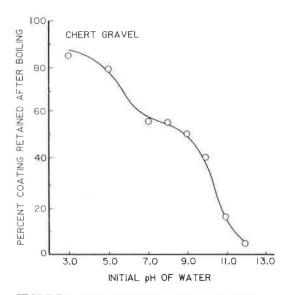


FIGURE 3 Effect of pH of water used in the boiling water test on chert gravel.

When an aggregate is being coated with asphalt, the aggregate selectively adsorbs some components of the asphalt, such as the more polar species of the asphalt, and hydrogen bonds or salt links are formed. The types and quantities of adsorbed components are thought to play an important role in adhesion and stripping (8). The presence of ketones and phenolics is thought, for example, to improve stripping resistance whereas carboxylic acids, anhydrides, and 2-quinolenes are thought to increase stripping sensitivity because of the high water susceptibility of their bonds with aggregate surfaces.

The water susceptibility of the hydrogen bonds (or some other dipole-dipole attractive bonds) and the salt links (i.e. the ionic bonds) between the adsorbed asphalt components and the aggregate surface would increase as the pH of the water present at the aggregate surface was increased. For this reason, stripping damage might be expected to occur with an aggregate that causes an increase (to a relatively high value) in the pH of any water present at the asphalt-aggregate interface.

The data shown in Figure 4 appear to agree with this suggestion. Granite, which imparted a high pH to contacting water, had a higher stripping propensity than did either crushed chert gravel or crushed quartz gravel, both of which imparted a lower pH to the contacting water. The pH values shown in Figure 4 were obtained by measuring the pH of the water used in the boiling water test of each aggregate after cooling.

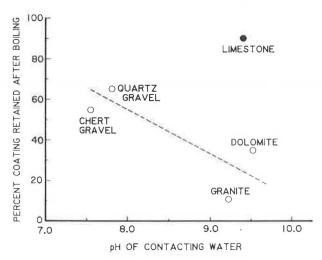


FIGURE 4 Comparison of pH of contacting water and stripping propensity as determined by the boiling water test.

Aggregate surface properties, other than pH changes, must also be considered. The different types of metal ions involved in the interaction between the asphalt and the aggregate can also play an important role in stripping. As shown in Figure 4, for example, although limestone imparted a relatively high pH to contacting water, it had a high stripping resistance. Alkaline earth metals in limestone associate strongly with asphalt components such as carboxylic acids to form alkaline earth salts, and the bonds formed are not dissociated easily in water even at a high pH; that is, in this case the adsorption is strong because of the insolubility of the alkaline earth salts formed between the limestone and the bitumen acids.

Surface Potential

The responses of stripping propensity to differences in the surface electrical charge of the aggregates are shown in Figure 5. The interfacial activity occurring between charged surfaces of the mineral aggregates and asphalt cements can be of fundamental importance to the stripping of asphalt from aggregate (18). That is, the surface charge of the aggregate can be as important as are specific chemical interactions; mineral aggregates possess distinctive polarities or electrochemical properties.

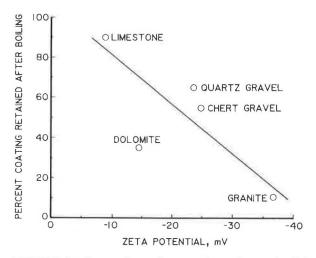


FIGURE 5 Comparison of aggregate surface potential and stripping propensity as determined by the boiling water test.

The functional groups of an asphalt that are adsorbed on an aggregate surface come mainly from the acid fraction of the asphalt. For instance, one of the acid molecules is represented by carboxylic acids (R-COOH). In the presence of water, the acid molecules are separated into two ions, the carboxylate anion (R-COOT) and the proton (H[†]), causing the asphalt surface to have a negative polarity at the interface. The increase in pH of water present at the aggregate surface increases the extent of dissociation of the acid molecules.

The aggregates with water present are negatively charged to varying degrees (Figure 5). As a result, a repulsive force develops between the negatively charged aggregate surface and the negatively charged asphalt surface at the interface, which causes the separation of the asphalt from the aggregate surfaces (stripping) (7). Solid surfaces in contact with water usually acquire charges through chemical reactions at the solid surface and adsorption of complex ions from the solution (19). For instance, metal oxide surfaces in water are hydrolyzed to form hydroxyl groups,

which, subsequently, dissociate according to the reactions

$$-M-OH(s) + H_2O \leftrightarrow -M-O^-(s) + H_3O^+(aq)$$
 (4)

$$-M-OH(s) + H_2O \leftrightarrow -M-OH_2^+(s) + OH^-(aq)$$
 (5)

to generate surface charges. The extent to which each of Reactions 4 and 5 proceeds depends on the pH of the solution and the type of mineral. A high pH value of the water in contact with the mineral surface would therefore cause the surface to be negatively charged.

The intensity of the repulsion developed between the asphalt and the aggregate depends on the surface charge of both the asphalt and the aggregate. As shown in Figure 5, granite, which had a high stripping propensity, possessed a relatively high surface potential whereas limestone, which had a high stripping resistance, had a relatively low surface potential (as determined by zeta potential measurements). The general observed trend was that the aggregates that had a relatively high surface potential in water were more susceptible to stripping.

From the foregoing discussion, it appears that, although physical properties of the aggregate are important, chemical and electrochemical properties of aggregate surfaces play an even more important role in stripping.

It should be noted that the values of pH and surface charge indicated in this study are only true for the sample tested because these values will be changed with variation of the mineral source and its aging history.

Antistripping Additives

Antistripping additives are substances added to asphalt cements to promote adhesion of the asphalt cement to the aggregate surface and thus to improve resistance to stripping. When antistripping additives were incorporated into the asphalt cement, the effectiveness of each additive varied considerably as the pH of the contacting water was changed, as shown in Figure 6. Here, the pH of water used in the boiling water test (contacting water) was changed by adding HCl or NaOH solution. Granite was used as a standard aggregate in this set of experiments. Some additives lost their effectiveness as the pH of the contacting water increased; the extent of effectiveness lost varied for each additive.

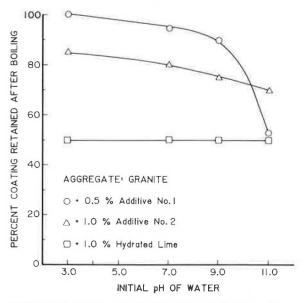


FIGURE 6 Effect of pH of water used in the boiling water test when using additives.

The effectiveness of Additive 1 was more sensitive to the pH of the contacting water than was the effectiveness of either of the other two additives. Additive 1 was a type of polyamine. This implied that, when the pH of the water was increased to a high value, the adsorption bond between amine-type additives and aggregate surfaces was weakened. As a result, water could more easily displace the asphalt from the aggregate surface. On the other hand, the performance of hydrated lime remained

constant, independent of the pH of the contacting water. The effectiveness of hydrated lime apparently was not as susceptible to environmental changes as was that of the polyamine-type additive.

It was found that by storing the asphalt-aggregate mixture for a few hours at 300°F (i.e., curing the mix), the effectiveness of some additives improved considerably even at a high pH value of contacting water (Figure 7). The formation of a film of polymerized asphalt (coke) on the aggregate surface was thought to be the primary cause of the observed curing effect. Some evidence in support of this postulation is provided in Figure 8. A granite was precoated with phenanthrene, which is known to have a high coking propensity. The result was an accelerated curing effect; that is, there was a faster response in stripping resistance to curing. It should also be noted that an aggregate surface can be conditioned by precoating with cokeforming compounds (e.g., phenanthrene, coal liquids) to enhance stripping resistance. More work related to curing mixtures to enhance their stripping resistance is to be published soon.

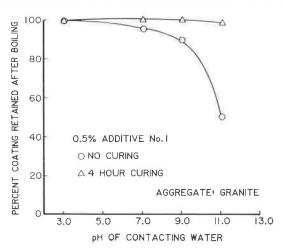


FIGURE 7 Effect of pH of water used in the boiling water test with and without additives.

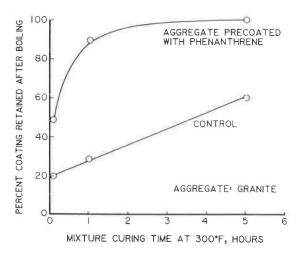


FIGURE 8 Acceleration of curing effect on stripping resistance by a high coking propensity additive, phenanthrene.

SUMMARY

Physicochemical properties of the asphalt, the aggregate, and the asphalt-aggregate mixture that might influence stripping are summarized in Table 1. It was found that, although the physical properties of an aggregate affected stripping, there was no strong correlation between the physical properties of the aggregate, such as pore volume and surface area, and the stripping propensity of the aggregate. Chemical and electrochemical properties of the aggregate surface in the presence of water were observed to have a significant effect on stripping.

All mineral aggregates tested in this study had distinctive pH values when immersed in water and had distinctive electrochemical surface properties that could be measured in terms of their zeta potential. It was found that aggregates that had relatively high surface potentials in water or that imparted relatively high pH values to water, or both, were more susceptible to stripping.

These results were thought to be due in part to the existence of electrical repulsive forces between the asphalt and the aggregate surface. When the pH of the contacting water was high, the negative surface potential of the asphalt in contact with the aggregate was also high because of the dissociation of acidic components in asphalt. Most aggregates would have a negative charge of varying intensity when in contact with water. The electrical repulsion between the two negatively charged surfaces, that is, the asphalt and the aggregate surface, can cause the asphalt to separate (or strip) from the aggregate surface.

It was found that the effectiveness of some additives could be sensitive to the pH of water in contact with aggregate. Storing the asphalt-aggregate mixture for a few hours at 300°F caused the effectiveness of some additives to improve considerably. The curing rate was accelerated when the aggregate was precoated with phenanthrene or coal liquids.

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