F.A. Barbour, R.V. Barbour, and W.E. Haines. Paving Asphalts: Chemical Composition, Oxidative Weathering, and Asphalt-Aggregate Interactions: Part II. FHWA, Interim Rept. FHWA-RD-74-71, June 1974.
7. J.C. Petersen, E.K. Ensley, and F.A. Barbour. Molecular Interactions of Asphalt in the Asphalt-Aggregate Interface Region. TRB, Transportation Research Board Record 515, 1974, pp. 67-78.
8. H. Plancher, S.M. Dorrence, and J.C. Petersen. Identification of Chemical Types in Asphalts Strongly Adsorbed at the Asphalt-Aggregate Interface and Their Relative Displacement by Water. Proc., AAPT, Vol. 46, 1977, pp. 151-175.
9. E.K. Ensley. A Study of Asphalt-Aggregate Interactions and Asphalt Molecular Interactions by Microcalorimetric Methods: Postulated Interaction Mechanism. Journal of the Institute of Petroleum, Vol. 59, 1973, pp. 279-89.
10. H. Plancher, C. Chow, S.A. Holmes, and J.C. Petersen. Moisture-Induced Damage in Bituminous Pavement--A Study of Nitrogen Compound Interactions with Aggregates. Proc., International Symposium, Progressi Nella Technologia Dei Bitumi, Stazione Sperimentale per i Combustibili, Viale A. Gasperi 3, 20097 San Donato Milanese (Milano), Italy, 1981, pp. 263-273.
11. J.C. Petersen, R.V. Barbour, S.M. Dorrence, F.A. Barbour, and R.V. Helm. Molecular Interactions of Asphalt: Tentative Identification of 2-Quinolones in Asphalt and Their Interaction with Carboxylic Acids Present. Analytical Chemistry, Vol. 43, 1971, pp. 1491-1496.
12. H. Plancher, E.L. Green, and J.C. Petersen. Reduction of Oxidative Hardening of Asphalts by Treatment with Hydrated Lime--A Mechanistic Study. Proc., AAPT, Vol. 45, 1976, pp. 1-24.
13. H. Plancher, G. Miyake, R.L. Venable, and J.C. Petersen. A Simple Laboratory Test to Indicate the Susceptibility of Asphalt-Aggregate Mixtures to Moisture Damage During Repeated Freeze-Thaw Cycling. Canadian Technical Asphalt Association, Vol. 25, 1980, pp. 247-262.
14. S.M. Dorrence, F.A. Barbour, and J.C. Petersen. Direct Evidence for Ketones in Oxidized Asphalts. Analytical Chemistry, Vol. 46, 1974, pp. 2242-2244.
15. J.C. Petersen, F.A. Barbour, and S.M. Dorrence. Identification of Dicarboxylic Anhydrides in Oxidized Asphalts. Analytical Chemistry, Vol. 47, 1975, pp. 107-111.
16. J.C. Petersen, S.M. Dorrence, M. Nazir, and H. Plancher. Oxidation of Sulfur Compounds in Petroleum Residues: Reactivity-Structural Relationships. Division of Petroleum Chemistry, American Chemical Society, Preprints, Vol. 26, No. 4, 1981, pp. 898-906.
17. C.V. Chachas, W.J. Liddle, P.E. Peterson, and M.L. Wiley. Use of Hydrated Lime in Bituminous Mixtures to Decrease Hardening of Asphalt Cement. Materials and Test Division, Utah State Department of Highways, Salt Lake City, Final Rept., Dec. 1971. NTIS: PB 213 170.
18. F.A. Barbour, R.V. Barbour, and J.C. Petersen. A Study of Asphalt-Aggregate Interactions Using Inverse Gas-Liquid Chromatography. Journal of Applied Chemistry and Biotechnology, Vol. 24, 1974, pp. 645-654.
19. J.C. Petersen. An Infrared Study of Hydrogen Bonding in Asphalt. Fuel, Vol. 46, 1967, pp. 295-305.
20. H. Plancher, G. Miyake, and J.C. Petersen. Shale Oil Products as Replacements for Petroleum Counterparts in Pavement Applications. Proc., 13th Oil Shale Symposium, Colorado School of Mines, April 16-18, 1980, pp. 261-268.
21. E.K. Ensley. Multilayer Adsorption with Molecular Orientation of Asphalt on Mineral Aggregate and Other Substrates. Journal of Applied Chemistry and Biotechnology, Vol. 25, 1975, pp. 671-682.
22. J.W. Button, J.A. Epps, and B.M. Gallaway. Laboratory Evaluation of Selected Shale Oil Asphalts in Paving Mixtures. U.S. Department of Energy, Rept. LERC-3695-1, Jan. 1978.

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Silane Pretreatment of Mineral Aggregate to Prevent Stripping in Flexible Pavements

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Debonding of asphalt from mineral aggregates (stripping) was termed a problem as far back as 1938, yet it continues to plague the paving industry today. Commercial antistrip additives are available, but their long-term effects are not well understood. A silane coupling agent was compared with a well-known commercially available liquid antistrip agent (amine) in the immersion-compression and double-punch debonding tests on two Arizona mineral aggregate sources. The silane was used as a mineral aggregate pretreatment, whereas the amine was added to the asphalt. The results of this research are encouraging and indicate that the silane generally performed as well as the liquid antistrip agent or better. Further testing is recommended along with construction of experimental projects to evaluate field performance.

An asphalt concrete pavement is inherently dependent on the cohesive and adhesive characteristics of the binder to hold it together. As a result, the bond between the asphalt binder and the mineral aggregate is of special importance. It is critical that a good bond be developed during construction and maintained for the life of the pavement. Any degree of loss of the asphalt-aggregate bond will result in a corresponding loss of pavement performance in one manner or another. The strength of an asphalt concrete mixture is a result of the cohesive resistance of the binder, the adhesive bond between the binder and the aggregate, the aggregate interlock, and the frictional resistance between aggregate particles.

Several methods have been used to limit the possibility of stripping. Some of the more common methods are as follows:

1. Addition of dry lime or portland cement in small percentages to the mix or lime-slurry treatment of mineral aggregate;
2. Precoating aggregates with bitumen or diluents prior to asphalt concrete production;
3. Addition of selected natural mineral fillers;
4. Disallowance of known hydrophilic aggregates;
5. Washing, wasting, or blending of aggregates; and
6. Addition of chemical antistripping agents.

All these methods, for one reason or another, are not always acceptable or economical in every situation.

Under certain circumstances, an asphalt binder will separate from the aggregate, a complex phenomenon known as debonding (commonly referred to as stripping). Debonding is a function of the environmental conditions, traffic loading, binder and aggregate characteristics, mixture properties, and more. However, it is generally agreed that the mechanism of debonding is the intrusion of water between the adherends. Even though a proper bonding of the asphalt to the aggregate may have taken place during construction, debonding is still possible. Water intrusion is the mechanism that will facilitate debonding by replacing the asphalt coating on mineral aggregates. Since water in one form or another will always be present in a pavement, stripping is always a possibility. An extensive study of debonding is available in the literature (1-16). The intent of this research project was to evaluate and compare an organofunctional silane as a mineral aggregate pretreatment with an amine liquid antistrip agent to determine whether the silane should be considered a practical antistrip treatment. It has been reported that amidoamine and imidoazoline antistrip compounds may actually increase "emulsion" formation at the aggregate-asphalt interface (1). These additives actually enable a better coating to be placed on the aggregate particles initially but could lead to accelerated stripping later. Emulsion formation is one mechanism by which stripping can occur; however, it is not an accurate description of the stripping mechanism. Although it is true that the term "emulsion" can be applied to any asphalt-water mixture, it may cause confusion when used to describe the stripping mechanism.

In 1976, the Arizona Department of Transportation (ADOT) undertook a limited in-house study to evaluate liquid antistrip additives as well as alternative methods of preventing debonding. It was apparent then, and still is now, that the long-term effects of these additives are not well understood. Many of our new pavements exhibited asphalt stripping 6-12 months after construction even though a commercial antistrip agent was used. A simple but significant test program was initiated. Three aggregate materials from different sources were tested with each of two different commercial antistrip compounds and Dow-Corning Z-6020 organofunctional silane in the immersion-compression test (AASHTO T165).

The commercial antistrip compounds tested were Edoco and Pavebond Special. Each was added to the asphalt in the amount of 1 percent by weight of the asphalt. The silane was applied to the mineral aggregates as a pretreatment and allowed an ambient cure of 24 h before mixing. Two different silane-in-water solutions were tested: 1.5 and 2 percent. These solutions were applied to the dry mineral aggregate at the rate of 3 percent by weight of the mineral aggregate.

The results were encouraging. The silane pretreatment solutions imparted a better retained strength in the immersion-compression test in every case but one. The complete results are shown in Table 1.

Based on these results, a second project was initiated and a consultant was hired to run further tests to confirm the effect that the silane solution had on the retained strength of an asphalt concrete mix. This time two different material sources were selected for testing. Furthermore, the double-punch test procedure (3) was to be used in addition to the immersion-compression test to see whether the results were test dependent. It was decided that Pavebond Special and Dow-Corning Z-6020 silane were to be tested. Pavebond Special was chosen because of its widespread use in Arizona.

Table 1. Immersion-compression test: preliminary data.

Compressive Strength (psi) Dry	Compressive Strength (psi) Wet	Retained Strength[a] (%)	Treatment
Aggregate Source 1: Pit 8567 Zuni			
353	118	33	None
	238	67	1 percent Edoco Anti-Strip
	326	92	1 percent Pavebond Special
	227	78	1.5 percent silane
	296	84	2.0 percent silane
Aggregate Source 2: Pit 8500 Globe			
287	119	41	None
	163	57	1 percent Edoco Anti-Strip
	237	83	1 percent Pavebond Special
	325	113	1.5 percent silane
	325	113	2.0 percent silane
Aggregate Source 3: United Metro No. 11 Yuma			
375	68	18	None
	125	33	1 percent Edoco Anti-Strip
	190	51	1 percent Pavebond Special
	207	55	1.5 percent silane
	264	70	2.0 percent silane

[a](Wet compressive strength/dry compressive strength) x 100 percent.

THEORY

It should be noted that after considerable work by ADOT in testing silane as an antistrip agent, it was discovered that previous work involved the use of silane coupling agents in asphalt concrete mixes both as an antistripping additive and as an aggregate pretreatment and is documented in a patent held by Chevron. However, silanes are not now, nor have they ever been, marketed as antistrip agents.

The patent describes dramatic asphalt retention by aggregates even when treated with as little as one part per million. Furthermore, it was reported that a tenfold increase in retained asphalt (on the surface of the aggregate) was demonstrated after a water-immersion test of 180°F for eight days. This work verified our early work and encouraged further testing. Significant favorable results were achieved with the silane as a mineral aggregate pretreatment and as an asphalt additive. This report addresses only the use of silanes as a mineral aggregate pretreatment.

DESCRIPTION OF TESTING

In March 1980, a proposal was submitted to the Federal Highway Administration to compare Dow-Corning Z-6020 and Pavebond Special as antistrip agents. R.A. Jimenez of the University of Arizona was commissioned to prepare and evaluate 240 asphalt concrete specimens by two different test methods: the double-punch debonding test and the immersion-compression test.

Immersion-Compression Test

A measure of resistance to debonding was obtained with the immersion-compression test (AASHTO T165). The AASHTO procedure was followed except that enough material was mixed at one time to produce three specimens instead of one. This change was necessary to assure that all specimens of a set had received the same chemical treatment. Work in the laboratory has shown variability in density, and strength measurements have met the usual requirements when enough material is mixed at one time to produce these specimens. After mixing at approximately 140°C, enough mixture was taken to produce a specimen 101 mm by 101 mm after compaction at 121°C. Following compaction, the set of six specimens was placed in a 60°C oven for 24 h. After the 24 h of curing and then cooling, specimens were weighed in air and submerged in water for density calculations. The six specimens were divided into two groups of nearly equal average density. One group was submerged in hot distilled water at 60°C for 24 h and the second group was stored in a 25°C room. After the 24-h hot-water exposure, the three specimens were transferred to a 25°C water bath for 2 h prior to testing under unconfined-compression conditions. The other three dry specimens were then tested under similar conditions. The effect of the hot-water exposure is found by dividing the strength of the wet specimens by the strength of the dry specimens and expressing this ratio as the percentage of retained strength.

Double-Punch Debonding Test

The double-punch debonding test was developed by Jimenez of the University of Arizona. The procedure has been described and published (3). The sequence of steps for this method is similar to that of the immersion-compression procedure, with the following exceptions:

1. Mixing and compaction: Size of specimen is 101 mm by 63 mm and compaction is by a vibratory kneading compactor.
2. Wet exposure: Specimen is not cured for 24 h. After density determination, specimen is submerged in 50°C distilled water for a minimum of 45 min and is vacuum saturated during this time. Following saturation and submersion, the specimen is stressed by the application of a repeated pore-water pressure that varies from 34 to 207 kPa and cycles at 10 Hz (580 cpm) for 10 min at a temperature of 50°C.
3. Strength test: Strength of the wet or dry specimen is obtained by an indirect tensile test referred to as the double-punch test. Two steel punches 25 mm in diameter stress the specimen on the centers of the two flat surfaces. Load is applied at a displacement rate of 25 mm/min.

The silane pretreatment of the mineral aggregate varied as to solution concentration as well as aggregate surface moisture condition [oven-dried or approximate saturated surface dried (s.s.d.)]. The silane was applied in two different ways depending on this condition. In the oven-dried state, the aggregate was treated with 3 percent (by weight of the aggregate) of four different silane concentrations. In the approximate s.s.d. condition, the aggregate was treated with 1 percent (by weight of the aggregate) of the same four silane-in-water concentrations, namely, 0.25, 0.75, 1.00, and 1.50 percent. These two different aggregate surface moisture conditions were selected to determine whether the silane-aggregate reactivity is dependent on this characteristic as well as to simulate field conditions. Figure 1 depicts the variables and test methods. Two different aggregate sources were tested--Salt River and Agua Fria--both from the Phoenix area. Pavebond Special was added to the asphalt binder in the amount of 1 percent by weight of the asphalt, which is the same amount used in construction.

MATERIALS USED FOR TESTING

Mineral Aggregates

Two sources of aggregate were selected for use in the test program. Crushed Salt River and Agua Fria aggregate samples were obtained for testing from commercial stockpiles. Physical characteristics and mix design data for each source are included in Table 2. Both are stream deposits in the Phoenix valley area; both mix gradations approach the Fuller maximum-density curve. The sand equivalent values indicate the primary difference between the two sources: 32 for the Agua Fria as compared with 55 for the Salt River sources. The sand equivalent test is a very good measure of the portion of detrimental fine dust or claylike minerals in the mineral aggregate. It is logical to conclude that a low sand equivalent number will indicate a higher potential for asphalt-aggregate debonding and therefore proves to be a valuable test for aggregate evaluation. A good correlation between sand equivalent value and stripping has been established (3).

Asphalt

One asphalt was chosen for use throughout the test sequence. The asphalt used conforms to an aged residue grading classification of AR 2000, which was obtained from Sahuaro Petroleum, Phoenix. Edgington Asphalt in Long Beach, California, is the asphalt source.

Additives

Pavebond Special is the registered trade name of a product of the Carstab Corporation. It is marketed as an asphalt additive to prevent debonding.

Silane coupling agents were first introduced to improve the water resistance of reinforced plastics. It was soon observed that they also imparted significant improvement to initial properties of laminates (2). Hydrophilic mineral surfaces were used in preparing composites with organic polymers; silanes were used to improve the bond. The similarity between the polymer-glass systems and the pavement materials was noted and it was felt that silanes may have the potential to increase the bond between asphalt and mineral aggregate surfaces.

Z-6020 Silane is a registered trade name of Dow-Corning. It is not marketed for the highway paving industry. It is primarily used as a coupling agent for the resin and plastic industry (2). It is a low-viscosity liquid aminoalkyl functional silane with the molecular formula $(CH_3O)_3SiCH_2NHCH_2CH_2NH_2$. It is only one member of one subclass of the much larger group of organofunctional silane coupling agents. The chemical name for Z-6020 is N-(beta-aminoethyl)-gamma-aminpropyltrimethoxysilane.

SUMMARY AND DISCUSSION OF RESULTS

The complete test results are included in Tables 3

Figure 1. Test program.

SILANE PRETREATMENT OF AGGREGATE

Prepare Four Silane-in-Water Solutions of 0.25%, 0.75%, 1.00% and 1.50%

MINERAL AGGREGATE OVEN-DRIED	MINERAL AGGREGATE SATURATED SURFACE DRY
Apply 3% (by weight of aggregate) to mineral aggregate of each of the 4 solution concentrations	Apply 1% (by weight of aggregate) to mineral aggregate of each of the 4 solution concentrations
Allow 24 hour ambient air cure	Allow 24 hour ambient air cure
Prepare asphalt concrete test specimens in triplicate for testing in both immersion-compression and double-punch tests	Prepare asphalt concrete test specimens in triplicate for testing in both immersion-compression and double-punch tests

PAVEBOND SPECIAL

Add 1% Pavebond Special to Asphalt and thoroughly mix.

Prepare asphalt concrete test specimens in triplicate for testing in both immersion-compression and double-punch tests.

NO TREATMENT

Prepare asphalt concrete test specimens in triplicate for testing in both immersion-compression and double-punch tests.

TEST MATRIX

TEST		DOUBLE PUNCH OR IMMERSION-COMPRESSION							
TREATMENT*		SILANE 3% O.D.		SILANE 1% SSD		PAVEBOND SPECIAL		NONE	
CONDITION **		DRY	WET	DRY	WET	DRY	WET	DRY	WET
CONCENTRATION	A	3	3	3	3	3	3	3	3
	B	3	3	3	3				
	C	3	3	3	3				
	D	3	3	3	3				

* O.D = OVEN DRY AGGR., SSD = SATURATED SURFACE DRY AGGR.
** WET OR DRY CONDITIONING OF SAMPLE
PAVEBOND SPECIAL 1% BY WEIGHT OF ASPHALT
3 REPLICATES OF EACH TEST

Table 2. Mineral aggregate characteristics.

Physical Characteristic	Salt River (% passing)	Agua Fria (% passing)
Sieve Size		
1 in	100	100
3/4 in	94	94
1/2 in	82	80
3/8 in	71	66
No. 4	50	50
No. 8	40	44
No. 16	32	36
No. 30	22	25
No. 50	15	14
No. 100	8	7
No. 200	4	4
Sand equivalent	55	32
Centrifuge kerosene equivalent oil ratio (%)	4.9	4.5
Effective specific gravity	2.67	2.69
Specific gravity with 5 percent asphalt	2.47	2.49

and 4. The results are also plotted in Figures 2 through 5. The retained strengths of the laboratory-prepared asphalt concrete specimens were calculated two ways, as shown in the tabular results. In this manner one can compare results of a particular treatment (wet soaked) versus an untreated unsoaked specimen or that same particular treatment versus the specimen in the dry state. Density, voids, and the differences in compressive strength between wet and dry specimens are also reported.

The results indicate that silane pretreatment of mineral aggregate is effective in preventing debonding as indicated by the two different test procedures. Furthermore, it appears to be more effective on the Agua Fria mineral aggregate, which is a much "dirtier" source than the Salt River aggregate, as shown by the low sand equivalent value of 32. Densities of the silane mixtures are consistently higher with lower void contents. This may have contributed to the increased retained strengths of the silane-treated mixes, since it is well known that mixes with low void content are more resistant to stripping than are mixes with high void content.

It is significant that the silane-treated specimens exhibited higher dry strengths than untreated specimens in all but three cases. Pavebond Special increased the dry strengths in every case; however, part of the increase may be a result of an increase in binder viscosity caused by the asphalt additive. It is known that amine additives facilitate better coating of aggregates, and this may also contribute to higher dry strengths.

Silane concentration, amount of silane solution applied, and application method all affect mix-strength retention as shown in Figures 2 through 5. For example, for the same given quantity of silane treatment (75 ppm), the retained strengths are substantially different in every case. This phenomenon indicates that aggregate surface condition (specifically, whether the surface is dry or approximately s.s.d.) has a definite impact on retained strength. Surface moisture may be the vehicle by which the silane is uniformly distributed over the aggregate surface area and, as such, could be an important factor influencing reactivity.

In summary, it appears that the silane pretreatment of mineral aggregates does improve resistance to debonding at least as well as Pavebond Special. Some retained-strength values are lower, but many more silane retained strengths are higher than those with Pavebond treatment. Further work must be conducted to optimize treatment methods and silane concentrations to accomplish the most economical application that yields the best results as well as to determine long-term results.

CONCLUSIONS AND RECOMMENDATIONS

1. In general, the silane pretreatment of two mineral aggregates improved the resistance to debonding of asphalt from mineral aggregate in the immersion-compression and double-punch tests.
2. The silane treatment appeared to have a more pronounced effect on the Agua Fria aggregate source. As a result, dirty aggregate sources (those

Table 3. Agua Fria aggregate: immersion-compression and double-punch test results.

Treatment	Density (pcf)	Voids (%)	Strength (psi) Dry	Wet	Difference[a]	Retained Strength A	B
Immersion-Compression Test							
Asphalt only	142.5	8.3	391	90	301	23	23
1 percent Pavebond Special	142.0	8.4	423	182	241	43	47
Silane concentration (%) applied to oven-dried aggregate							
0.25	143.0	8.0	439	158	281	36	40
0.75	143.5	7.5	511	276	235	54	71
1.00	142.5	8.1	464	269	195	58	69
1.50	144.0	7.5	510	352	158	69	90
Silane concentration (%) applied to s.s.d. aggregate							
0.25	144.5	7.0	478	220	258	46	56
0.75	144.5	6.8	514	288	226	56	74
1.00	144.5	6.8	454	268	186	59	69
1.50	144.5	6.2	477	329	148	69	84
Double-Punch Test							
Asphalt only	149.0	3.8	185	120	65	65	65
1 percent Pavebond Special	149.5	3.5	204	155	49	76	84
Silane concentration (%) applied to oven-dried aggregate							
0.25	150.5	3.0	204	145	59	71	78
0.75	152.5	1.9	230	214	16	93	116
1.00	152.0	2.2	238	238	0	100	129
1.50	151.5	2.4	230	230	0	100	124
Silane concentration (%) applied to s.s.d. aggregate							
0.25	151.0	2.8	158	136	22	86	74
0.75	152.0	2.1	193	176	17	91	95
1.00	150.5	3.1	151	139	12	92	75
1.50	152.0	2.2	169	159	10	94	86

Note: A = [wet strength (treated)/dry strength (treated)] x 100 percent; B = [wet strength (treated)/dry strength (untreated)] x 100 percent.
[a]Dry strength minus wet strength.

Table 4. Salt River aggregate: immersion-compression and double-punch test results.

Treatment	Density (pcf)	Voids (%)	Strength (psi) Dry	Wet	Difference[a]	Retained Strength A	B
Immersion-Compression Test							
Asphalt only	142.0	8.1	321	151	170	47	47
1 percent Pavebond Special	142.0	8.1	409	327	82	80	102
Silane concentration (%) applied to oven-dried aggregate							
0.25	142.0	8.1	325	312	13	96	97
0.75	142.5	7.8	379	292	87	77	91
1.00	142.5	7.8	428	321	107	75	100
1.50	143.0	7.2	337	283	54	84	88
Silane concentration (%) applied to s.s.d. aggregate							
0.25	144.5	6.4	498	478	20	96	149
0.75	143.0	7.5	419	335	84	80	104
1.00	145.0	5.9	537	537	0	100	167
1.50	143.5	7.1	447	384	63	86	120
Double-Punch Test							
Asphalt only	149.0	3.6	140	113	27	81	81
1 percent Pavebond Special	148.5	3.9	163	140	23	86	100
Silane concentration (%) applied to oven-dried aggregate							
0.25	151.0	2.3	165	160	5	97	114
0.75	151.5	2.0	181	181	0	100	129
1.00	151.0	2.1	157	143	14	91	102
1.50	151.5	1.7	167	167	0	100	119
Silane concentration (%) applied to s.s.d. aggregate							
0.25	151.0	2.2	172	136	36	79	97
0.75	151.5	1.9	207	143	64	69	102
1.00	151.5	2.0	200	200	0	100	143
1.50	150.5	2.4	181	179	2	99	128

Note: A = [wet strength (treated)/dry strength (treated)] x 100 percent; B = [wet strength (treated)/dry strength (untreated)] x 100 percent.
[a]Dry strength minus wet strength.

with low sand equivalent values) may benefit most from silane pretreatment. Furthermore, marginal aggregate sources may be allowed if testing with silane verifies that minimum retained-strength values can be obtained.

3. Silane concentration, application method, and aggregate surface moisture condition influence retained-strength values in both test methods. Therefore, a more detailed examination of these factors is the next logical step in future research endeavors.

4. Research should continue with silane chemicals as debonding preventives. In addition, silanes should be tested as additives in asphalts to compare with pretreatment of mineral aggregates as examined in this research effort. Preliminary tests by ADOT Research Center have shown the chemical to be effective down to 0.05 percent by weight of asphalt (the equivalent of approximately 25 ppm by weight of aggregate) as an antistripping agent.

5. It is recommended that experimental projects that use silane as an antistripping agent be con-

Figure 2. Agua Fria aggregate: immersion-compression test results.

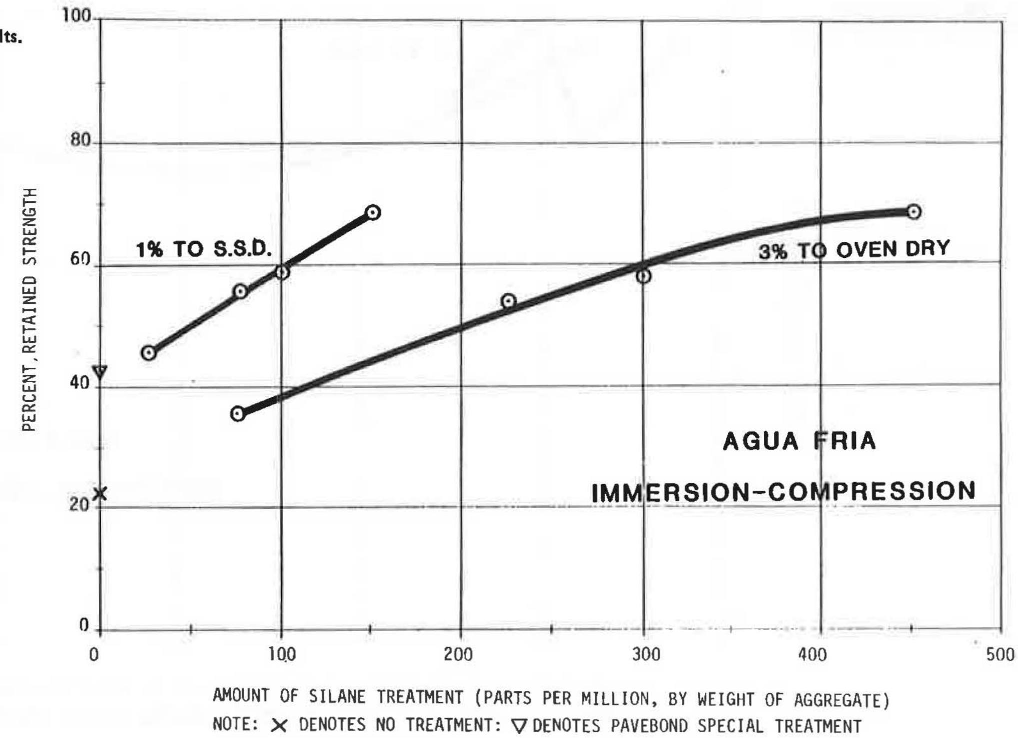
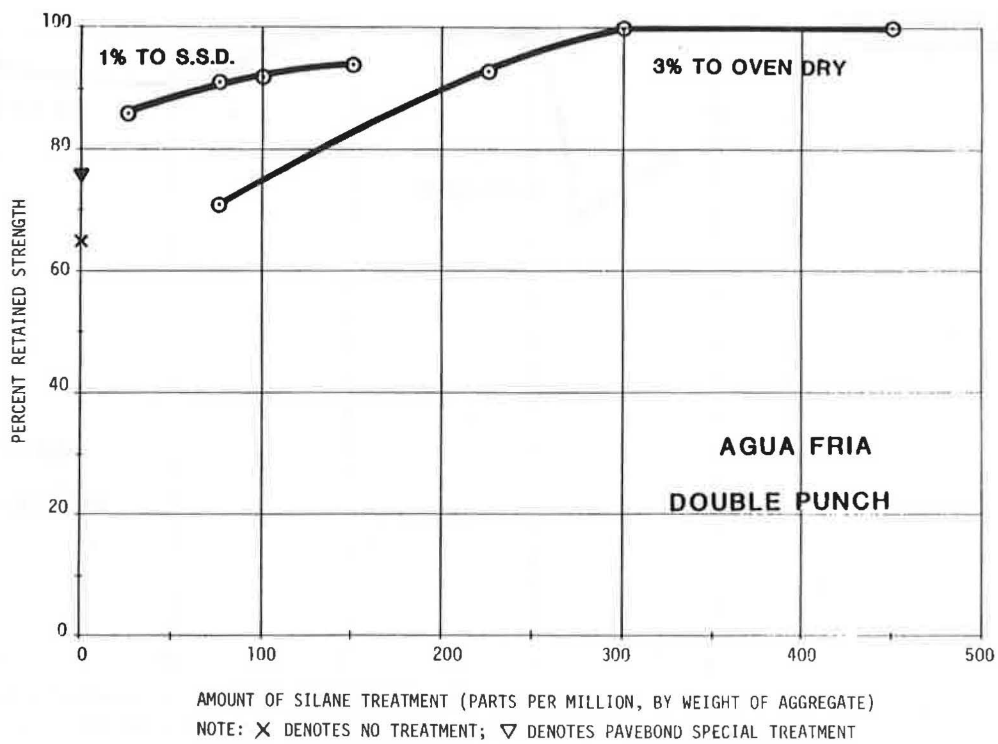


Figure 3. Agua Fria aggregate: double-punch test results.



structed to determine actual field effectiveness as well as long-term effects on asphalt concrete mixes.

6. Additional testing of aggregate pretreatment with silane solutions is warranted to compare the results of this research with various other aggregate types and characteristics, since this research was limited to testing of two local aggregate sources. In addition, other asphalt types and sources should be tested for the same reason.

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Figure 4. Salt River aggregate: immersion-compression test results.

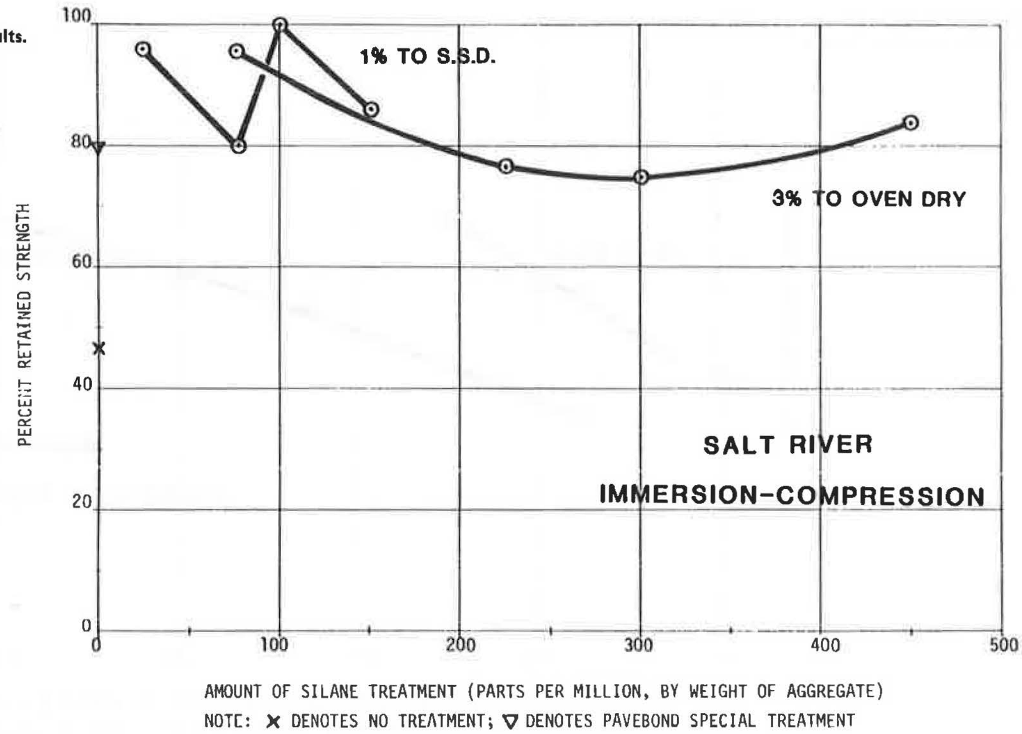
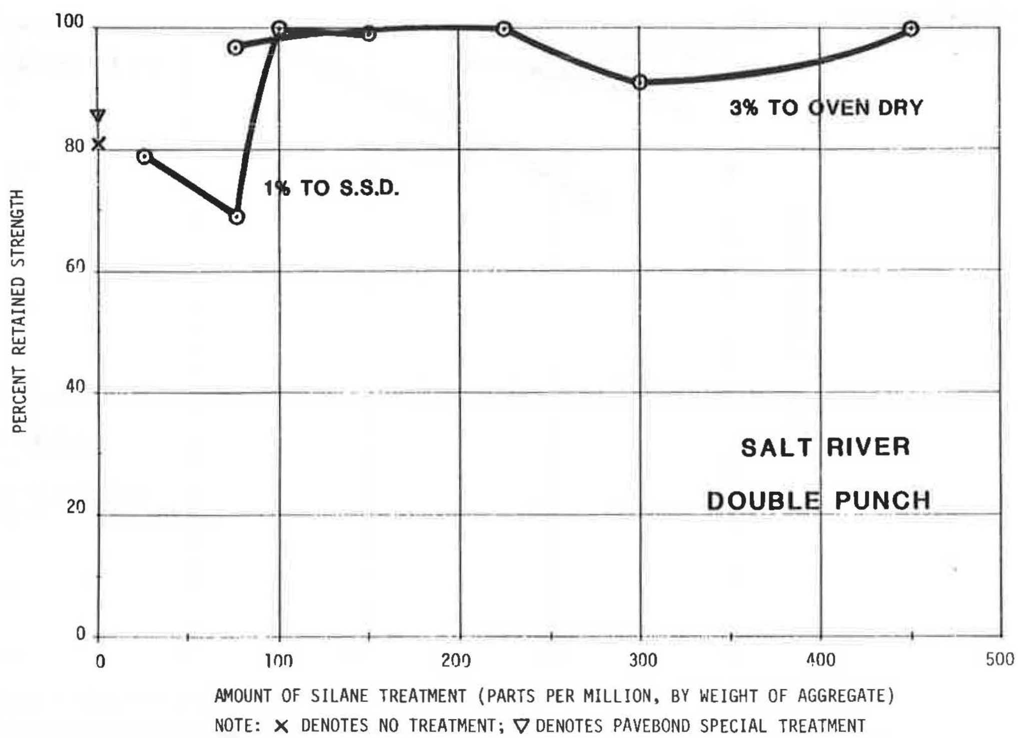


Figure 5. Salt River aggregate: double-punch test results.



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REFERENCES

1. H.J. Fromm. The Mechanism of Asphalt Stripping from Aggregate Surfaces. Proc., AAPT, Vol. 43, 1974.
2. E.P. Plueddemann. Mechanism of Adhesion of Coatings Through Reactive Silanes. Journal of Paint Technology, March 1970.
3. R.A. Jimenez. Testing for Debonding of Asphalt from Aggregates. TRB, Transportation Research Record 515, 1974, pp. 1-17.
4. J.M. Rice. Relationship of Aggregate Characteristics to the Effect of Water on Bituminous Paving Mixtures. Symposium on Effects of Water on Bituminous Paving Mixtures, American Society for Testing and Materials, Philadelphia, PA, ASTM Spec. Tech. Publ. 240, 1958.
5. K. Majidzadeh and F.N. Brovold. State of the Art: Effect of Water on Bituminous-Aggregate Mixtures. HRB, Special Rept. 98, 1968.
6. P. Hubbard. Adhesion of Asphalt to Aggregates in the Presence of Water. Proc., HRB, 1938, pp. 238-249.
7. Proc., International Symposium on Porous Asphalt, Amsterdam, Netherlands, 1976.
8. R.P. Lottman. Predicting Moisture-Induced Damage to Asphaltic Concrete: Field Evaluation Phase. NCHRP, Project 4-8(3)/1, Interim Rept., Feb. 1978.
9. J.A.N. Scott. Adhesion and Disbonding Mechanisms of Asphalt Used in Highway Construction and Maintenance. Proc., AAPT, Vol. 47, 1978.
10. F.C. Sanderson. Methylochlorosilanes as Anti-stripping Agents. Proc., HRB, 1952, pp. 288-300.
11. L. Clplijauskas, M.R. Piggot, and R.T. Woodhams. Chemically Modified Asphalts for Improved Wet Strength Retention. American Chemical Society, I&EC Product Research and Development, June 1979.
12. M.C. Ford, P.G. Manke, and C.E. O'Bannon. Quantitative Evaluation of Stripping by the Surface Reaction Test. TRB, Transportation Research Record 515, 1974, pp. 40-54.
13. R.P. Lottman, R.P. Chen, K.S. Kumar, and L.W. Wolf. A Laboratory Test System for Prediction of Asphalt Concrete Moisture Damage. TRB, Transportation Research Record 515, 1974, pp. 18-26.
14. T.Y. Chu. Investigation of Roadway Drainage as Related to the Performance of Flexible Pavements. Univ. of South Carolina, Columbia, Aug. 1974.
15. J.G. Chehovits and D.A. Anderson. Upgrading of Marginal Aggregates for Improved Water Resistance of Asphalt Concrete. Pennsylvania State Univ., University Park, Jan. 1980.
16. J.C. Peterson and others. Paving Asphalts: Chemical Composition, Oxidative Weathering and Asphalt-Aggregate Interactions, Part II. FHWA, Rept. FHWA-RD-74-71, June 1974.

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Evaluating Asphaltene Settling Test and Relating Results to Physical Properties of Paving Asphalts

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The asphaltene settling test was designed to measure the compatibility of the components of an asphalt cement by measuring the settling rate of the asphaltene for a mixture of asphalt in hexane. The results are reported as the length of time required for the asphaltenes to settle out of a solution of hexane. The main premise on which the settling test is based is that the asphaltenes with the longer settling times are better dispersed in the hexane-maltene phase and that longer settling times indicate that the mixtures have more compatible components. The purposes of this study were (a) to evaluate the settling test in terms of test repeatability and the effect of varying selected parameters of the procedure on test results, (b) to determine the settling time of a series of asphalt cement specimens in order to investigate potential relationships between settling time and asphalt characteristics, and (c) to evaluate the effectiveness of asphalt modifiers such as softening agents and antistripping agents. Based on the results, it is felt that additional work is required if the test is to have a practical value. Nevertheless, the findings indicate that (a) the test has a fair repeatability; the coefficients of variation of settling times for 262 asphalts ranged from 2.8 to 9.4 percent; (b) the settling time of asphalt is sensitive to the test temperature; (c) no well-defined relationship was found between settling time and specification-type asphalt characteristics (penetration, viscosity, specific gravity, flash point); (d) the major factor that affected test results was the asphalt producer; and (e) the addition of antistripping agents reduced the settling time with respect to the virgin asphalt, whereas the effect of adding a softening agent was inconsistent, although there was a tendency for reduced settling times. A modified procedure to simplify the procedure, to reduce the time and cost of performing the test, and to improve its repeatability is presented.

The objective of the study summarized in this paper was to evaluate the asphaltene settling test, which has been suggested as a means to rapidly evaluate asphalt durability and the compatibility of asphalts and to determine how effective asphalt-softening agents, which have been proposed for use in recycled asphalt mixtures, are in redistributing the molecular agglomerates present in aged asphalts.

The test is based on previous work (1,2) describing relationships between asphaltenes and durability that led to the development of the settling test, which, according to Plancher and others (3), was developed by Hoiberg and Suhaka for the Asphalt Roofing Manufacturers Association. Later modifications by Plancher [comments in review of report by Kennedy and Lin (4)] adapted the test for paving-