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Contract Report S-68-5

Research Study on

SOIL TREATMENT MATERIALS FOR
DUST PALLIATION, SOIL WATERPROOFING AND
SOIL STRENGTHENING

by

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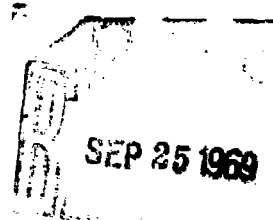
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FOREWORD

The work in this report was performed under Contract Number DA-22-079-eng-437 entitled "Research Study on Soil Treatment Materials for Dust Palliation, Soil Waterproofing and Soil Strengthening," dated June 15, 1965, between United States Army Waterways Experiment Station and the Ashland Chemical Company. This research was sponsored by the United States Army Materiel Command under Department of the Army Project DA-1-T-O-21701-A-046, "Trafficability and Mobility Research," Task 05, "Mobility Engineering Support (Dust Control, Southeast Asia)."

This report was prepared by Mr. C. N. Impola and Dr. D. A. Olsen of Ashland Chemical Company. This program was under the general supervision of Dr. W. J. McKillip of the Contract Research Group of Ashland Chemical Company.

The contract was monitored by Mr. G. R. Kozan, Chief, Stabilization Section, Expedient Surfaces Branch, under the general supervision of Mr. W. J. Turnbull, Chief, Soils Division, WES. Contracting Officers were COL J. R. Oswalt, Jr., CE, and COL Levi A. Brown, CE.

PART I: BACKGROUND

Introduction

Military operations require improved materials to suppress dust at airfields, helipads, operational bases, intermediate and cantonment areas as well as supply bases and roads. Dust in these areas has increased logistical problems greatly by reducing service life of machinery and equipment. For example, helicopter rotor blades have to be replaced after 200-300 hours rather than the expected 1100 hours. Also, the engines have to be replaced after 1/3 to 1/2 the normal usage period.

Present requirements for a potential dust palliative have been set forth in the Department of the Army Approved Qualitative Materiel Requirement (QMR) for Dust Control Material (August 1966). The following performance characteristics are sought:

1. Be effective and operationally usable within four hours after application to the surface of all types of soil.
2. Withstand, without failure or peeling, helicopter rotor downwash (10 psf disc loading) and C-130 aircraft propwash (100 mph air velocity).
3. Be effective, with only minor maintenance, for the following minimum time:
 - a. Six months in non-traffic areas.
 - b. Three months in areas subjected to infrequent traffic of ground vehicles or aircraft, such as shoulders or overruns of runways.
 - c. One month in areas trafficked by ground vehicles or aircraft.

In addition to the above performance requirements, the following usage requirement is requested:

Weight and volume characteristics of the material shall not exceed three pounds per square yard or 0.45 gallons per square yard of ground surface treated on trafficked areas.

Purpose and Scope

Initial screening of Ashland's resins for use as dust palliatives in non-trafficked areas as authorized under Contract Number DA-22-079-eng-437 was based on knowledge, technology and resins

developed for foundry core binders. The determination of surface free energies of soils, and correlation of wetting with compressive strengths of treated soils was also authorized under this contract.

Laboratory modifications of these resins and screening of other Ashland resins along with other commercially available organic and inorganic resin systems were done under Modification Number 1 of the subject contract.

Under Modification Number 2 of the subject contract, laboratory synthesis and evaluation of new and specialized resin systems were conducted to meet the broader requirements of the QMR which was issued during this period.

This final report, prepared in fulfillment of Contract Number DA-22-079-eng-437 and Modifications Number 1 and 2, covers the work done from June 15, 1965 to January 22, 1967.

PART II: MATERIALS TESTED AND TEST PROCEDURES

Materials

Soils

Three types of soil procured locally and 150 pounds of buckshot clay received from Waterways Experiment Station (WES), Vicksburg, Mississippi, were used in the screening of the resins as dust palliatives. The soils procured locally were dried and pulverized at 150 F. using a drum drier in the Ashland experimental station. Final water content was less than 2%. The sieve analyses obtained on these soil samples by the Ashland Control Laboratory are given below.

<u>Screen Size No.</u>	<u>Per Cent Passing</u>		
	<u>Sand</u>	<u>Clay</u>	<u>Silt</u>
10	99.1	--	100
20	--	92.4	93.2
40	88.5	72.2	72.9
60	65.1	48.2	53.7
80	--	51.4	--
100	30.7	47.4	35.9
200	10.4	38.5	27.0

The buckshot clay received from WES was not analyzed for size distribution.

Resins

The resins that were evaluated as dust palliatives were chosen on the basis of:

1. Experience with foundry core resins
2. Known resin properties such as ease of cure and physical strength
3. Ease of application
4. Availability
5. Cost

The following general classes of resin systems were evaluated:

- Epoxy
- Emulsified petroleum resin
- Gelatin
- Acrylamide

Acrylic
Phenol-formaldehyde
Aniline-furfurol
Polyurethane
Sodium silicate N
Plasticized sulfur
Unsaturated polyester
Alkyd

Test Procedures

Sample Preparation:

Primary evaluations were conducted by placing the loose dried soil into an 8-inch diameter by 1 1/2 inch deep disposable pie tin. The resin was then poured on the surface. Limited supplies of dried and pulverized soils required changing to 2-inch diameter sample size for initial screening. Samples were prepared using 2-inch ID rigid polyvinyl chloride pipe cut in 2 1/2-inch lengths. The test samples were prepared in three different ways:

1. Two inches of loose soil was placed in the mold and the calculated amount of resin to give 3 pounds per square yard was poured on and allowed to soak into the soil.
2. Soil and resin were prepared as in 1 above, but the samples were compacted after one hour using an American Foundry Service Rammer made by the Dietert Company as specified for foundry sample preparation. Compaction was effected by dropping a 14-pound weight attached to a two-inch diameter disk from a height of two inches three times to the 2-inch diameter surface.
3. Soil and resin thoroughly milled or admixed by mechanical stirring and then compacted as in 2 above.

Other test samples were prepared by mulling or admixing the soil and resin by mechanical stirring and then cast into a 4 x 8 x 1/2-inch mold, pressing out the entrapped air with a trowel and the surface troweled smooth.

Another method, used for spray application, was to pour the loose, dry soil into a 6 x 6 x 2-inch mold. The soil was leveled to the mold edge with a straight edge, then the resin was applied with either air atomizer or air-less spray gun.

To obtain physical properties on urethane elastomers, the elastomers were cast on a round one-gallon can cover treated with wax to prevent adhesion. The liquid urethane was poured on the cover and allowed to cure. After cure, the free film was removed from the cover and dumbbell-shaped specimens were cut for testing using a die according to ASTM Method D-412.

Tests Performed

The following tests were run on samples which had no visual defects on the surface, such as cracks:

1. Six by six-inch diameter soil specimens
 - a. Effect of air impingement was evaluated by subjecting the specimens to an air blast equivalent to 150 mph for one minute. Specimens were tilted at an angle of 10° in the direction of the air blast. The air blast was created by a blower with a rated capacity of 8000 cfm.
 - b. Samples that passed the air impingement test, i.e., where the surface skin was not broken, were checked for resistance to water erosion. The test specimens were mounted below a spray nozzle calibrated to give a 65° solid water cone at a rate of 1.1 gallons per minute at 40 psi water pressure. The samples were placed 4-1/2 inches below the nozzle tips so that 70% of the sample was sprayed with high velocity water droplets. The duration of the test was two hours initially. This was later reduced to one hour duration to conform to the tests run at Waterways Experiment Station, Vicksburg, Mississippi. Weight immediately after testing was recorded and compared to weight before testing to determine if any water had been absorbed through the resin surface.
 - c. Samples that passed the water spray test were again subjected to the air impingement test as in "b" above.
2. Two-inch diameter and 2 x 2 x 1 1/2-inch soil specimens
 - a. Water repellancy tests were run on the 2-inch diameter soil specimens by adding 0.5 ml water to the soil surface two hours after the resin application. The time required for the water to soak in was recorded. The test specimens were rated as follows:

Poor (P) - water soaked in within one minute; Fair (F) - water visible on the surface 1-5 minutes; Good (G) - water visible on the surface 5-10 minutes; and Excellent (E) - water visible on the surface more than ten minutes.

- b. Unconfined compressive strengths were run on fully cured (at least three days at ambient temperature) 2-inch diameter specimens and 2 x 2 x 1 1/2-inch samples cut from the 8-inch diameter specimens. Surfaces of the specimens were ground smooth with a belt sander before testing. Compressive strengths were run using a Model TT-C Instron Universal Testing Machine according to ASTM Method D-2166 with a head speed of 0.05 inches per minute.
 - c. Freeze-thaw resistance was evaluated by immersing the specimens for 16 hours in water at room temperature, pouring off the water and placing the wet specimens in a freezer for 8 hours at -10 F. Appearance and unconfined compressive strengths were recorded after 8 cycles.
 - d. Wet-dry resistance was determined by immersing the 2-inch diameter or 2 x 2 x 1/2-inch specimens cut from larger samples for 8 hours in water at room temperature, pouring off the water, and subjecting the specimens to heat for 16 hours in a forced draft oven at 140 F. The specimens were tested for unconfined compressive strength after 8 cycles.
3. Four-by-eight by one-half inch cast specimens
- a. Water repellant tests were run on the cast soil specimens after two hour cure according to the procedure in 2a.
 - b. Flexural strengths were determined by the standard ASTM Method D709 using a Model TT-C Instron Universal Testing Machine. Three 1 x 8 x 1/2 inch specimens were cut from the cast 4 x 8 x 1/2 inch specimens using an abrasive blade.
4. Cast urethane elastomers
- a. Tensile strengths and per cent elongations were determined for the elastomers by ASTM Method D-412 using a Model TT-C Instron Universal Testing Machine at a head speed of 20 inches per minute.

Some of those elastomers having elongation beyond the range of the Instron were tested on a Scott Model L-6 Tensile Tester with a head speed of 20 inches per minute.

PART III: TEST RESULTS

Preliminary Screening of Resins

Sixteen different resin systems, including organic and inorganic, were evaluated as dust palliatives and soil waterproofing agents in the initial screening. Each resin type is discussed below in the order of listing in table 1.

1. Bisphenol A Derived Epoxy Resins

Bisphenol A type epoxy resins are used where good adhesion and high tensile strengths are desired. Epon 828 from Shell Chemical Company was selected as representative of this group of resins. A low-cost, amine-terminated resin, Ashland Serial 1496, was selected as the curing agent because of its low toxicity, ready availability, and Ashland's prior experience with this material. Good penetration was obtained on sand and clay when the resin was diluted with high levels of Solox solvent. The cure times were slow (12-40 hours). The clay-epoxy specimen broke up easily.

2. Emulsified Petroleum Resins

Cohorex, an emulsified petroleum resin manufactured by the Golden Bear Oil Company, has been widely used as a dust palliative around military installations. Cohorex and a comparable Ashland experimental emulsion were evaluated on sand and clay. The penetration was good on sand but only minor penetration was obtained on clay. The treated surface did not form any skin and stayed moist. The Cohorex-treated specimens had only fair water repellency after two-hour cure.

3. Gelatin

15XPF gelatin was tested on dry sand with chromium sulfate, and formaldehyde as catalysts. The sample treated with chromium sulfate apparently gelled, preventing complete penetration. The formaldehyde-cured sample gave complete penetration in the sand and cured to a hard, but easily broken, brittle composite. The chromium sulfate-cured specimen was softer and rubbery.

4. AM-9

AM-9, a chemical grout marketed by American Cyanamid, is a blend of water-soluble acrylamide and diacrylamide

which polymerizes when properly catalyzed to give a void-filling substance for sealing leaks in dams, etc. When catalyzed with DMAPN-KFe*, AM-9 rapidly cures to a crumbly, soft gel that continues to cure over a period of several days to a hard, strong composite. Water repellency of the treated sand specimen was poor.

5. Acrylic Emulsions

Acrylic emulsions are relatively low-cost, water-dilutable resin systems and are widely used as paint vehicles, adhesives, fabric treatments, etc. Based on recommendations of the Ashland Resins Laboratory, a number of experimental emulsions were screened. EP8908-23, EP8908-122 and EP8908-129 are representative of acrylic emulsion resins that may be obtained commercially.

The resins as supplied, had a viscosity too high to penetrate the soil samples to any degree. However, they formed a heavy impervious surface skin on the clay and silt. The sand-treated specimen also had a surface skin; but the skin was slightly porous, testing only fair on water repellency .

Reducing the viscosity of the emulsions with water gave good penetration in sand and improved the penetration slightly in the silt and clay. However, the surface skin was more porous with the higher water content resins and the water repellency diminished considerably.

6. Chem-Rez 200

Chem-Rez 200 and phenol-modified Chem-Rez 200 are furfural based, rapid-setting resins developed by Ashland for foundry applications. These resins, admixed with foundry sand (3% level), give significantly higher strengths than aniline-furfural in foundry cores. When these resins were applied on sand and cured with phosphoric acid, they cured very slowly or not at all. Water repellancy was generally poor.

7. Aniline-Furfural

Attempts to use aniline-furfural resins as stabilizers for surface soil using either poured or admixing techniques were not very successful. Resin penetration was excellent but cures with 37% HCl required 12 to 24 hours. Erratic

*Dimethylamino-propionitrile-potassium ferricyanide-ammonium persulfate.

cures were obtained, particularly with clay and silt soils.

8. Emlon E-200

Emlon E-200 is a new epoxy resin manufactured by Stoner-Mudge, Pittsburgh, Pennsylvania, that is soluble in water. Cures of the Emlon E-200 were obtained in two hours or less with diethylene triamine, which is also water soluble. Reproducibility of cure and composite homogeneity appear to be problems based on our initial screening.

9. Polyurethane Elastomer

An attempt was made to use the dried sand as a filler for a urethane elastomer-type composite. Sand was blended with a 2000 molecular weight polypropylene glycol (Wyandotte P2010) and then with a low-cost crude diisocyanate (Allied Nacconate 4040). Cure was very slow even with substantial quantities of stannous octoate, and residual moisture in the sand caused extensive foaming of the composite.

10. Sodium Silicate N

Penetration of sodium hexafluorosilicate-cured sodium silicate N was low by the poured technique. Composites prepared by admixing cured in one hour to a hard, somewhat brittle composite. The water repellency and surface abrasion of the sodium silicate N sand specimens were poor.

11. Sulfur

Sulfur plasticized with E-13, a thioglycollate ester, gave hard, chemically-inert composites on cooling from the molten state (approximately 150 C) to room temperature. The plasticized sulfur and soils were preheated to 150 C, blends were prepared with sand and silt, and then the blends were poured into molds. The composites set to rigid solids on cooling, generally within ten minutes.

12. Unsaturated Polyester Resins

The low cost of polyesters (less than 25¢/lb in bulk) and the ability to use low-cost reactive solvent (styrene - less than 10¢/lb in bulk) prompted a rather extensive evaluation of these resins for soil treatment. Complete penetration of all three soil types (sand, clay, and silt) was readily obtained when high levels of styrene (up to 80%) were used to reduce resin viscosity to essentially the consistency of water. The cure times could readily be controlled over

wide limits by changing catalyst and/or promoter levels. Addition of 10% water to the soil had no observable effect on gel time. Excellent water repellency was obtained when higher resin solids unsaturated polyester resin was used.

13. Moisture-Cure Polyurethanes

Polyurethane resins that cure by reaction with moisture in the air are rapidly gaining in popularity as coatings and sealants. The urethane resins can be readily formulated to give plastics that are hard and brittle or strong elastomeric rubbers.

Initial results using Arothane 160, Arothane 156 and Arothane 170 as poured resins for soil stabilization were very encouraging. These resins, particularly Arothane 170 diluted with ethyl acetate to reduce viscosity, gave good cures as soon as one hour if properly catalyzed. These resins were outstanding in their ability to waterproof all three types of soils.

14. Water-Soluble Alkyds

Alkyd resins offer the unique advantage of reaction with oxygen in the air to give surface cures for dust palliation and soil waterproofing. The resin below the surface should remain in an uncured state to provide self-healing properties if the surface skin is broken. Arlon 110, Arlon 310, Arlon 363, and Arlon 580 were selected as representative resins for the early screening.

Penetration of these resins was unexpectedly poor. The resins tended to give the desired surface skin, but the surface strengths were poorer than other systems tested. Water repellency was generally only fair, presumably due to the presence of water solubilizing groups in the resin.

15. Solvent-Dilutable Alkyds

The solvent-dilutable alkyds would also be expected to give only surface cure for use as dust palliatives and waterproofing agents. Ashland has pioneered the development of rapid-setting alkyd resins, which permit the formulation of solvent resistant enamels that have short drying times. Aroplaz 6008, Aroplaz 6065, and Aroplaz 832 were selected as representative resins for this study.

As expected, these resins gave surface skins when applied by poured technique. These films were excellent water repellents for sand and clay. Penetration into sand was excellent. Only slight penetration of clay was observed.

16. Miscellaneous Systems

a. Aroflint 505

Aroflint 505 is a specially-epoxidized oil resin that gives hard, ceramic-like coatings on cement blocks, etc. This resin did not cure when applied to soil samples and water repellency was poor as the water soaked into the soil immediately.

b. Several newly-developed foundry resins (Lino-Cure, Lino-Cure A, Lino-Cure C, Lino-Cure 2125) were evaluated as soil treatment materials. Lino-Cure, Lino-Cure A and Lino-Cure 2125 did not cure when applied on sand containing 10% water. Lino-Cure C applied alone did not cure either but when Lino-Cure C was applied with ethylene glycol, a hard surface cure was obtained that had excellent water repellency.

c. Octadecyl isocyanate was evaluated as a noncuring soil waterproofing agent on sand and clay containing 10% water. Waterproofing of the soils was outstanding. Grinding the treated soils with a mortar and pestle did not reduce the water repellency. Water poured on the soil after the treatment remained in a spherical droplet. Blending the isocyanate with Arothane 170 gave soil treatment with excellent water repellency with some degree of cure to improve dust palliation properties.

d. An unsaturated polyester resin prepared in the laboratory, EP2379-60, blended with a small amount of octadecyl-isocyanate produced excellent water repellency when applied on sand. The cure, however, was slow and incomplete, producing a rubbery skin on the surface.

e. Several alternate polyurethane and free diisocyanate systems were examined briefly. Alpha-methyl glucoside polyether-toluene diisocyanate (AMG-TDI) prepolymers gave only limited penetration. The water repellency was good on sand and excellent on loose clay. A good abrasion resistant surface was obtained.

Hexamethylenediisocyanate alone applied on sand and clay containing 10% water gave hard composites but water repellency was poor. Most of the isocyanates are lachrymators when in the free state and not reacted on to the polymer chain.

- f. Siroc #1 is an inorganic siliceous grout from Diamond Alkali Company. Penetration on sand and clay containing 10% water was good. The samples did not cure and water repellency was poor.

Physical Testing

Poured or Admixed Applications

Table 2 lists the results of air impingement and water erosion tests, unconfined compressive strengths, and freeze-thaw and wet-dry cycling effects on the resins initially screened as soil treating materials. Each type of resin will be discussed in the order listed in the table.

1. Bisphenol A Type Epoxy Resins

The Epon 828-treated sand and silt specimens had good wind and water erosion resistance at the use levels tested. The lowest use level tested was 1.3 lbs/square yard resin plus 5.1 lbs/square yard solvent. Unconfined compressive strength was 1079 psi at a use level of 5.2 lbs/square yard resin and 5.1 lbs/square yard solvent. Using a level of 11 lbs/square yard resin plus 16.5 lbs/square yard solvent, the unconfined compressive strength was over 2200 psi. The Epon 828 treated sand specimens withstood freeze-thaw and wet-dry cycling without noticeable change. At lower levels, only surface penetration was obtained and a hard crust formed.

2. Emulsified Petroleum Resins

Coherex and Ashland experimental emulsion both withstood 60 mph air impingement test on sand (150 mph air impingement test was not used at this time). Both resin systems failed on loose clay and silt due to shrinkage cracks on the surface. Neither resin passed the water erosion test. Unconfined compressive strengths were not run since the specimens did not cure.

3. Gelatin

Sand treated with 15XPF gelatin cured with chromium

sulfate or formaldehyde passed both the 60 mph air impingement and water erosion test. However, the sample was flexible and easily broken after the water erosion test. Unconfined compressive strength was not run on these samples.

4. AM-9

AM-9 treated sand gave a hard, strong composite with an unconfined compressive strength of over 1700 psi. However, the cure time was long and resistance to freeze-thaw and wet-dry cycling was fair to poor as evidenced by the loss of 30 to 60% compressive strength.

5. Chem Rez 200

Chem Rez 200 applied to washed and dried foundry sand gave 354-610 psi unconfined compressive strength after 24 hour ambient cure based on results obtained by the Ashland Chemical Foundry Group. This resin, when applied on sand and cured with phosphoric acid under this test program, cured very slowly or not at all. Resistance to 60 mph air impingement was good, but water erosion was only fair with some surface erosion.

6. Aniline-Furfural

Unconfined compressive strengths on aniline-furfural treated sand was low (399 psi). Resistance to wind and water erosion, freeze-thaw, and wet-dry cycling was good to excellent. When applied on clay with a high level of solvent, a hard specimen was obtained after 24 hour cure. The unconfined compressive strength was very low (32 psi).

7. Emlon E-200

Emlon E-200 applied on sand and clay performed satisfactorily in the air impingement test. Some erosion of the treated soil, especially clay, is apparent from the loss in weight during the water erosion test.

The unconfined compressive strength was good (1388 psi). The treated specimen withstood the freeze-thaw cycling but split after the third wet-dry cycle.

8. Sodium Silicate N

Sodium silicate N sodium hexafluorosilicate system

added to sand by mulling formed a hard composite within one hour. Resistance to wind and water erosion was good to excellent. The unconfined compressive strength was fair (650 psi). Resistance to freeze-thaw and wet-dry cycling was poor as indicated by the loss of 50% of the unconfined compressive strength.

9. Unsaturated Polyester Resin

Aropol 7110 with high levels of styrene gave excellent unconfined compressive strengths when applied on sand by pour-on technique. The strengths varied 1173 psi with low resin level to 2300 psi with high resin level. The freeze-thaw and wet-dry cycling did not affect the unconfined compressive strengths. Resistance to air impingement and water erosion was excellent.

10. Moisture-Cure Polyurethane Resins

Arothane 170 in ethyl acetate gave satisfactory unconfined compressive strengths (700 psi) when applied on sand by pour-on. The resistance to freeze-thaw and wet-dry cycling was excellent.

11. Air-Dry Alkyds

Application of air-dry alkyds to sand by mulling gave hard but relatively weak composites. Arlons, the water-soluble alkyds, and Aroplazes, the solvent-dilutable alkyds, produced specimens with low compressive strengths (90 and 107 psi). Both specimens withstood the freeze-thaw cycling. On wet-dry cycling, the water-alkyd-treated specimen crumbled. The solvent-alkyd treated sample did withstand the wet-dry cycling although some spalling was noted on one specimen.

12. Acrylic Emulsions

Only one acrylic emulsion sample, prepared with EP8908-23, was considered satisfactory for testing. Resistance to air and water erosion and wet-dry cycling was good. Although the unconfined compressive strength (685 psi) was 1/2 to 1/3 of the epoxy and polyester systems, it was considerably stronger than many of the other acrylic systems tested. One of the acrylic emulsion-treated samples, EP8908-122, crumbled in the freeze-thaw and wet-dry cycling tests. The third emulsion system passed the freeze-thaw but crumbled during wet-dry cycling.

Sprayed Application

Table 3 lists the results of air impingement and water erosion tests of various resins applied on sand and/or clay by spray-on application. The polyester and Chem Rez resins were applied with an air atomizing spray. The rest of the resins were applied with an air-less spray gun. The results will be discussed as listed in table 3. These were tested as soil treatment materials intended for use in nontraffic areas at three pounds per square yard maximum use level, including solvent.

1. Asphalt Emulsions

Cationic asphalt emulsions at various solids content were tested on sand and clay. The sand samples passed the 150-mph air impingement test, both before and after the water erosion test. The surface did not fail during the water erosion test but small holes in the surface allowed some water seepage as indicated by the increase in weight.

When applied on loose clay, large surface cracks appeared due to wetting and subsequent shrinkage of the clay.

2. Epoxy Resins

Two different types of epoxy resins were tested on loose clay. Neither the Emlon E-200 nor the Epon 815 performed satisfactorily due to surface cracks from the shrinkage of the clay.

3. Acrylic Resin Emulsions

The acrylic emulsions also failed to pass the wind test on loose clay due to shrinkage cracks on the surface.

4. Polyurethane Resins

Arothane 170 failed the air impingement test on dry, loose clay due to poor wetting of the clay and cracking. Arothane 160 performed satisfactorily when applied on pre-wetted clay. The specimens passed the 150-mph air impingement tests and water erosion test. However, some water seeped through the surface in the water erosion test.

5. Air-Dry Alkyds

Aroplaz 6008, Aroplaz 6065, Aroplaz 832, and a blend of

Aroplaz 832-Aroplaz 6008 were sprayed on dry and pre-wetted clay and dry sand. The alkyds did not wet the dry clay sufficiently to cover the surface. However, after pre-wetting the clay, the coverage was good. The alkyds formed a surface skin on the clay that withstood the air impingement and was fairly impervious to water. When tested four hours after the application, the surface skin was not completely cured and tended to buckle slightly under the wind. The alkyds soaked into the loose sand but still formed a surface skin that held under the air impingement test.

6. Unsaturated Polyester Resin

Several commercial unsaturated polyester resins were evaluated as soil treatment materials. Most of the emphasis was placed on the less rigid Aropol 7510M and Aropol 7720M. Several levels of resin solids were used since the diluent, styrene, also cross-links with the resin and is, therefore, not just a diluent. The maximum solids content that was tested was 50%, and the minimum was 30%. The polyester-treated surfaces generally passed the air impingement tests. However, in many instances, the resin pulled away from the edge of the mold, forming a small crack. Even with the crack at the edge, the surface coating did not blow away although some of the soil was blown out. The surface also withstood the water erosion test, but some water entered the soil in most cases.

Blends of Aropol 7510M and Aropol 7720M at 50% resin solids performed satisfactorily on both sand and clay. Aropol 7720M alone performed very satisfactorily on pre-wetted clay. It passed the air impingement tests and gained only 95 grams in the water erosion test. Part of the 95 grams, as much as 50 grams, was water soaked up by the wooden forms used to hold the soil.

7. Chem Rez 200 and Chem Rez 300

Chem Rez 200 at 50% solids performed satisfactorily on both pre-wetted and dry, loose clay and dry sand. The treated specimens passed the wind impingement tests. The specimens also passed the water erosion tests with no apparent change. The weight increase after the water erosion varied from a low of 45 grams to a high of 207 grams.

Chem Rez 300, tested at varying solids content and as blends with epoxy resins (Dow's DER, Shell's Epon) passed the air impingement tests on loose, dry clay. The weight increase during the water erosion tests was considerably lower than with other systems generally. Epon 815 blended with the Chem Rez 300 produced some flexibility to the surface coating.

8. Epoxidized Oils

Admex 710, epoxidized soybean oil, and ELO, epoxidized linseed oil, both performed satisfactorily on sand and clay. However, the tear strength of the surface coating from these resins appeared low.

9. Miscellaneous Resin Systems

Several polyurethane resins were synthesized in the laboratory to attempt to improve the flexibility of the surface coatings on soil. Series 112B to 115B in table 4 are representative of these. These resins applied on clay and sand at 50-60% solids performed satisfactorily in the air impingement and water erosion tests. However, the flexibility of these systems was less than deemed necessary.

A styrene-butadiene latex emulsion tested on sand gave a slow cure but did pass the tests. The surface had poor flexibility. The same latex applied on clay produced several cracks on curing.

EP8911-41 is a low-cost experimental latex emulsion prepared by the Ashland resin group. This material, applied on clay, gave good coverage but the flexibility of the surface coating was only moderate and the tear strength was low.

Flexural Strength with Unsaturated Polyester Resin

Unsaturated polyester resins exhibited good physical strength properties in the early testing. A series of flexural strength specimens were prepared with Aropol 7510M milled with sand.

The test specimens were prepared by adding a weighed amount of sand to polyester resin containing catalyst and promoters and mixing with a mechanical stirrer for 20-30 seconds. The sand mixture was then poured into a 4 x 8 x 1/2-inch mold, packed to remove entrained air, and leveled with a spatula. Three 1 x 8 x 1/2-inch test specimens were cut from each

mold using a table saw with a carborundum blade. These cut specimens were tested for flexural strength on an Instron Model TT-C Universal Testing Machine using Standard ASTM D-709. The experiments were designed for a statistical study by factorial analysis to determine the interaction, if any, and the optimum level, for cobalt, dimethylamine (DMA) and hydrogen peroxide (H_2O_2). Three levels of each component were tested on dry sand using Aropol 7510M at 30% resin solids in styrene. Cobalt and DMA were tested at 0.5, 1.0 and 1.5% of the resin-plus-styrene weight and 50% aqueous hydrogen peroxide was tested at 1.0, 1.5, and 2.0% (same basis). Data is listed in table 4.

The flexural data, obtained from these tests, were analyzed by computer. A definite interaction was found between cobalt and hydrogen peroxide. Based on these results, the optimum level of cobalt was found to be 1.35% and 1.27% the optimum hydrogen peroxide level. No clear interaction of DMA with cobalt and/or hydrogen peroxide could be determined by analysis of the data.

A second, shorter series of runs was conducted using Aropol 7510M at 30% resin solids in styrene on wet sand (10% water premixed into the sand). Three levels of cobalt, DMA, and hydrogen peroxide (50% aqueous) were used: 1, 2, and 3%. Using a simple Latin square statistical program, no conclusion as to interactions could be reached when analyzed by computer because of insufficient data. Significantly lower flexural strengths were obtained on wet sand as compared to dry sand (200-400 psi).

Flexural strengths were also run on the specimens after a seven-day age. (First flexural strengths were run after three days.) In general, a significant increase in strength was observed after the aging, indicating the cure continues over a period of several days.

Some of the flexural samples did not cure due to wrong proportions of promoter-to-catalyst. However, several high flexural strengths were obtained. For example, a flexural strength of 2250 psi was obtained using 0.5/0.5/1.5% ratio of cobalt, DMA and H_2O_2 . Other significant values were:

Ratio - Co/DMA/ H_2O_2	Flexural Strength psi
1/1/1%	2145
1.5/0.5/1%	2163
1.5/1.5/1.5%	2047

Unsaturated Polyester Resins

Storage stability of unsaturated polyester-styrene resin

systems in known to be limited at elevated temperatures unless high levels of inhibitors are used. Such high levels of inhibitors, however, will increase cure times and can even result in incomplete cures. Ashland has done extensive studies of type and level of inhibitor vs stability during the past five years. This has led to the development of several unsaturated polyester-styrene resin systems stable in excess of two years when stored at 70-80 F.

The unsaturated polyester resins exhibited high physical strength properties in the screening tests. Since the resins will be subjected to elevated temperatures in the field, stability tests were run at elevated temperatures using off-the-shelf type inhibitors. The effect of resin type, styrene level, and inhibitor type and level were checked. Test temperatures of 158, 130, and 110 F were used. The data is listed in table 5. The resins as listed are in the order of decreasing activity. That is, Aropol 7010, listed first, is the fastest curing and Aropol 7720M is the slowest.

Generally, the fastest reacting unsaturated polyester resin system had the least stability. Increasing the styrene level from 60% to 70% also reduced the stability noticeably. This is apparent with most of the resins tested. For example, using 0.25% dimethyl lauryl-amine-hydrochloride (DMLA-HCl) as the inhibitor, Aropol 7010 at 40% resin solids had 55-day stability at 130 F. Reducing the solids to 30%, the resin remained fluid for only 19 days at 130 F. The same trend was noted with Aropol 7510M. Using 0.5% of Adogen 464, a quaternary ammonium chloride, as the inhibitor, Aropol 7510M remained fluid 150 days at 130 F. When resin solids were reduced to 30%, the resin system remained fluid only 79 days at 130 F.

Temperature has a very pronounced effect on the stability: the polyester stability is 25 days at 158 F; whereas at 130F, 150-day stability was obtained with the same inhibitor. Some inhibitors provide less stability if too high levels are used, e.g., Aropol 7510M at 40% resin solids was evaluated with dimethyl lauryl amine hydrochloride as the inhibitor at 1, 0.75, and 0.5% levels. At 158 F, no difference was noted in stability. However, at 130 F, the resin with 1% DMLA-HCl gelled after 101 days; with 0.5% inhibitor, it gelled after 111 days; and with 0.25%, it was fluid for 130 days.

The best inhibitor in the test series was a blend of Adogen 464 with copper naphthenate. Aropol 7510M at 30% resin solids in styrene had 30-day stability at 158 F, and 160-day stability at 130 F using 0.4% Adogen 464 and 0.01% copper

naphthenate. The next best inhibitors were Adogen 464 alone and dimethylaurylamine hydrochloride which gave 150 and 130-days stability, respectively, at 130 F at 0.5% use level.

Many of the resin systems tested had not gelled at the termination of the test, but a solid formed within the container, generally on the cover above the resin layer. This was undoubtedly polystyrene. Previous testing by Dow Chemical has shown that styrene will vaporize and, consequently, polymerize in the vapor state in the absence of inhibitors. They have found that by using containers with various liners, such as Heresite, the styrene wets the liner and returns back into the solution without polymerizing.

Urethane Elastomers

The new QMR, which was issued August 1966, requires that the dust palliatives be effective for a minimum of one month in areas subjected to traffic by ground vehicles or aircraft. With the usage limit of three pounds per square yard or 0.45 gallons per square yard maximum, it was apparent that all previously screened resins would not withstand traffic at these usage levels without rupturing when applied on loose silt, clay or sand.

To bear traffic on loose sand at those usage levels, it is felt that the coating must be elastomeric with the following properties:

- . Elongation upwards of 1000-1500%
- . Tensile strength over 1000 psi

Urethane elastomers are known to possess these qualities. Published data show that urethane elastomers can be formulated with elongations of up to 1300% and tensile strengths more than 8000 psi. However, it is generally noted that the higher elongations are obtained at the expense of the tensile strength. Also, urethane elastomers with these properties are generally cured by vulcanization or post-cured at temperatures in excess of 100 C. This, of course, is impractical for the intended use as dust control agent.

Based on requirements of the Army QMR, research was redirected from screening and modifying off-the-shelf resins to synthesizing urethane elastomers that would have the high elongation properties, yet cure at ambient temperatures. Table 6 lists the tensile strength and elongation of some of the elastomers that have been prepared.

Several of the elastomers prepared initially had high elongation but low tensile strength, or high tensile strength but low elongation. One elastomer, a 2000 molecular weight polyether diol-toluene diisocyanate prepolymer at a 3.5/1 isocyanate to hydroxyl (NCO/OH) ratio, had a tensile strength of 2050 psi with an elongation of 870% when cured by atmospheric moisture. Other elastomers of this type, using various catalysts for moisture cure, had tensile strengths ranging from 825 to 1275 psi with elongations ranging from 880 to 950%.

Since curing by atmospheric moisture can result in erratic cure times dependent on atmospheric conditions, similar results were attempted by polyol cure. The polyol cure could be controlled more readily by catalysts. However, the results with the TDI prepolymers were discouraging because the proper balance of tensile strength and elongation could not be achieved.

To obtain higher elongations, emphasis was changed from the aromatic, TDI, to aliphatic diisocyanate-based prepolymers. With the aliphatic diisocyanates, aromatic amines can be used in the cure to obtain more rigidity, if desired. The aliphatic isocyanates are less reactive than the aromatic, and whereas the reaction of an aromatic isocyanate with an aromatic amine is instantaneous, the aromatic amine with an aliphatic isocyanate is slowed down enough so a complete mixing can be accomplished. Aliphatic amines are too reactive even with aliphatic isocyanates to obtain good mixing.

Two aliphatic diisocyanates were used: General Mill's DDI, dimer diisocyanate, and Mobay's HX, hexamethylene diisocyanate (HDI). Using DDI and a 2000 molecular weight polyether diol as the prepolymer, elongations of 800-900% were obtained with various aromatic diamine-diol blends as curing agents. However, the tensile strength was low at 200-300 psi.

Several HDI-2000 molecular weight polyether diol prepolymers were prepared, varying the reaction conditions (reaction time, temperature, additions, etc.). The isocyanate to hydroxyl ratios were also varied from 3/1, 2.5/1 and 2/1. At the higher NCO/OH ratios, the elongations were generally lower than desired (~500%). Very encouraging results have been obtained using an NCO/OH ratio of 2/1. Using varying ratios of aromatic diamine-diol blends for curing, urethane elastomers with over 2200% elongations and tensile strengths

over 1000 psi have been prepared. The maximum elongation and tensile strength could not be determined on many of the elastomers on the Instron Model TT-C Testing Machine because the elongation was beyond the limit of the machine. Some of these were subsequently tested on the Scott Model L-6 Tensile Tester to measure the ultimate elongation.

Using a blend of a 200 m.w. aromatic diamine and a 52 m.w. diol at an amine-OH molar ratio of 0.25/1, an elastomer with 620 psi tensile strength at 1150% elongation was obtained. The ultimate elongation (at break) was 1820%. Other elastomers tested: 810 psi tensile strength at 1290% elongation (ultimate elongation 1740%) when cured with an aromatic diamine-52 m.w. diol blend at an amine-OH molar ratio of 0.5/1; 825 psi tensile at 1250% elongation (ultimate 1880%) cured with amine-134 m.w. diol-amine-OH molar ratio 0.5/1; 690 psi tensile strength at 1230% elongation (ultimate 2240%) cured with amine-52 m.w. diol at amine-diol molar ratio 0.25/1.

Storage stability of isocyanate-terminated prepolymers is achieved by using trace amounts of benzoyl chloride. Benzoyl chloride acts as a non-aqueous buffer that maintains a slight acidic environment. This prevents an excessive degree of cross-linking which is obtained when catalyzed under basic conditions, thus affecting the tensile strength development to a slight degree, but ensuring a high elongation.

The last nine elastomers in table 6 can be used to compare the properties of the elastomer vs the benzoyl chloride level. Series #598-600 prepolymers were prepared with 0.04% benzoyl chloride. The next three (601-603) were prepared from a prepolymer with 0.02%; 0.01% benzoyl chloride was used to prepare series 604-606. Comparing physical strengths of the three prepolymer-based elastomers cured with the same curing agent (diamine-52 m.w. diol at 0.25/1 molar ratio) (#598, 601 and 604), it can be seen that the elastomer with the highest benzoyl chloride in the prepolymer (#598) had the lowest tensile strength, 630 psi. The difference in the tensile strength between the elastomer from the 0.02% benzoyl chloride prepolymer (#601) and the 0.01% benzoyl chloride prepolymer (#604) was not as noticeable (811 vs 895 psi). The same trend is noticeable when using a blend of diamine-90 m.w. diol (molar ratio 0.5/1) as curing agent (#600, 603, 606) where the tensile strength varies from 765 psi with the highest benzoyl chloride level to 1310 with the intermediate and 935 psi with the prepolymer containing the lowest benzoyl chloride level.

Several of the elastomers with the higher tensile strengths and elongations were applied on sand and loose, dry clay by spray-on application. The elastomers performed satisfactorily on sand when 1-2% Cab-O-Sil was used as a thixotroping agent. On loose, dry clay, however, the clay had to be pre-wet with water to effect a surface coating. After the spraying was complete, the clay surface formed shrinkage cracks which split the elastomer surface before the cure was sufficient to withstand the shrinkage. The problem was partially eliminated by using an aqueous amine solution for the pre-spray. Using this solution, there was no cracking of the surface, but the elastomer coating pulled away from the edges of the wooden form, forming small cracks along the edges.

However, wetting the soil very lightly with a fine water mist, not enough to cover the surface, and then spraying on the urethane resin with $1/3$ to $1/2$ theoretical level of curing agent, a very satisfactory surface coating was obtained. Occasionally, very small cracks formed on the edges. Those specimens without cracks passed the wind and water erosion tests.

PART IV: PHYSICAL CHEMISTRY STUDY

Estimation of Surface-Free Energies of Soils and Correlation of Wetting with Compressive Strength

In order to estimate the surface-free energy of a solid, the contact angles of several liquids must first be measured on the solid surface. The surface-free energy of the solid is then determined by graphical means using the cosine values of the contact angles and the surface tensions of the liquids. The work reported here consists of: 1) measurement of contact angles on sand, silt, and clay; 2) estimation of the respective surface-free energies; and 3) correlation of resin spreading with the compressive strengths of resin-soil samples.

Contact Angle Measurement

From previous work on the critical surface tension (i.e., an estimate of the surface-free energy) of glass, it was known that only liquid metals and aqueous solutions of inorganic salts such as potassium carbonate and calcium chloride would exhibit contact angles greater than zero on the soil samples.¹ The first phase of this work consisted of compressing the soil samples into slugs in a hydraulic press, removing the slug from the press, and measuring the observed contact angles of mercury and aqueous solutions of potassium carbonate as a function of applied pressure. It was anticipated that constant contact angles would be obtained with increasing applied pressure. Good results were obtained with mercury as shown in figure 1. The soil slugs were much too porous, however, for contact angle measurements using aqueous potassium carbonate solutions. Penetration was almost instantaneous and no reliable contact angles could be obtained.

An alternate method is to make use of the Washburn equation which predicts the rate of rise of a liquid into a capillary:²

$$\frac{dl}{dt} = \frac{2r \gamma \cos \theta - r^2 \rho g}{8\eta l} \quad (1)$$

1. D. A. Olsen and A. J. Osteraas, J. Phys. Chem., 68, 2730 (1964).
2. See, for example: J. T. Davies and E. K. Rideal, "Interfacial Phenomena," 2nd Ed., p. 423, Academic Press, New York (1963); L. I. Osirow, "Surface Chemistry," p. 277, Rheinhold Publishing Corp., New York (1962).

where: l = length of penetration in time t
 r = capillary radius
 γ = surface tension of the liquid
 θ = contact angle
 ρ = density of the liquid
 g = gravity
 η = viscosity of the liquid

For very small capillaries such as those in a bed of packed soil the second term is small and Washburn's equation reduces to:

$$\frac{dl}{dt} = \frac{r \gamma \cos \theta}{4 \eta l} \quad (2)$$

which may be integrated to:

$$l^2 = \frac{r \gamma t \cos \theta}{2 \eta} \quad (3)$$

Since the capillary radius, surface tension, contact angle, and viscosity are constant for a given liquid and solid, equation 3 reduces to:

$$l = k \sqrt{t} \quad (4)$$

and where:

$$k = \frac{r^2 \gamma^2 \cos^2 \theta}{4 \eta^2} \quad (5)$$

Thus, the penetration of liquids into the packed bed of the soil samples should be linear with root time. This prediction is shown to be true experimentally in figures 2, 3, and 4 for several liquids on sand, silt, and clay, respectively.

Since the values for the various radii of the capillaries in the packed beds cannot be determined, a tortuosity factor, τ , must be included in equation 3 to give:

$$(\tau l)^2 = \frac{r \gamma t \cos \theta}{2} \quad (6)$$

or

$$l^2 = \frac{r}{\tau^2} \cdot \frac{\gamma t \cos \theta}{2} \quad (7)$$

For a given packing the bed constant r/r^2 will be constant. The bed constant can be determined by using liquids which wet the solid, i.e., $\cos \theta = 1$. For this case, equation 7 becomes:

$$l^2 = \frac{r}{r^2} \frac{t}{2\eta} \quad (8)$$

For evaluating contact angles from the experimental data of figures 2, 3, and 4, the factor r/r^2 can be eliminated from equations 7 and 8 to give:

$$\cos \theta_1 = \frac{(\text{slope}_1)^2 \eta_1 \sqrt{2}}{(\text{slope}_2)^2 \eta_2 \sqrt{1}} \quad (9)$$

where the subscripts 1 and 2 refer to the aqueous solutions and absolute ethanol, respectively. Absolute ethanol is assumed to completely wet the soil samples (i.e., $\cos \theta = 1$). The values calculated for the various contact angles are given in table 7. As can be seen from figures 2 and 4, the data for ethanol, water, and 2M calcium chloride have very similar slopes. Since the packing of the columns was undoubtedly non-uniform, any attempt to calculate contact angles from the small differences in these slopes is subject to considerable error. Such data were not used to estimate surface-free energies.

Estimation of Surface-Free Energies

Once contact angles have been obtained, the normal procedure is to plot $\cos \theta$ versus the liquid surface tensions and extrapolate to zero contact angle and thus obtain the critical surface tension³ (i.e., an estimate of the surface-free energy). Such a plot is not feasible here because of the widely separated surface tension values of the calcium chloride solutions and mercury. It is possible, however, to use the Gibbs-Autonoff equation,⁴ viz:

$$\cos \theta = \frac{2\gamma_s}{\gamma_L} - 1 \quad (10)$$

3. See, for example: W. A. Zisman, Advan. Chem., Series 43, 1, (1964).

4. V. R. Gray, Chemistry and Industry, p. 969, June 5, 1965.

which predicts a linear plot of $\cos \theta$ versus $1/\gamma_L$ with a slope of $2\gamma_S$ where γ_S is the surface-free energy of the solid. Alternatively, the graph may be extrapolated to $\cos \theta = 1$ where $\gamma_S = \gamma_L$. Such a graph for the contact angle data obtained on the soil samples is shown in figure A5. The value obtained for the surface-free energy of sand is 68 dynes/cm, of clay 60 dynes/cm, and of loam 49 dynes/cm. The value for sand is very close to the critical surface tension obtained for glass, viz 72 dynes/cm¹, and thus is reasonable. Since silt presumably contains the largest amount of organic materials, the lowest value is expected for silt, as was the case, with an intermediate value for clay.

Correlation of Spreading Coefficients with Compressive Strengths

After the surface-free energies of the various soils have been determined as above, it then is possible to determine whether spreading of a resin on a soil sample will also occur. The spreadings of a liquid on a solid is governed by:⁵

$$S = \gamma_S - \gamma_L - \gamma_{LS} \quad (11)$$

where γ_S , γ_L , and γ_{LS} are the surface-free energies (i.e., surface tensions) of the solid, liquid, and interface, respectively. For spreading (i.e., wetting): $S > 0$, and for non-spreading: $S < 0$. An approximation can be derived from equation 11 for the case of an organic liquid spreading upon a low energy solid surface, since it is then reasonable to assume that γ_{SL} is negligibly small in comparison with γ_{LV} . Therefore:

$$S = \gamma_S - \gamma_{LV} \quad (12)$$

and

$$\gamma_S > \gamma_{LV} \text{ for spreading.} \quad (13)$$

Hence, in all such systems, when spreading occurs, the specific surface-free energy of the liquid is usually less than that of the solid.

5. See, for example, A. W. Adamson, "Physical Chemistry of Surfaces," p. 264, Interscience Publishers, New York, (1960).

Scharpe and Schonhorn⁶ have stated explicitly that the criteria for spreading and for strong adhesion bear a one-to-one correspondence to each other and that it is, therefore, possible to decide, a priori, whether a liquid will adhere strongly to solid surface if the specific parameters in equation 13 are known. Equation 13 is the fundamental relationship that determines whether strong adhesion will occur.

Thus, for the case of resins on soils, if the surface tension of the resin is known, it should be possible to predict whether the resin will spread on a given soil sample and also, if the resin will adhere.

Several resin systems were chosen and the surface tension of the resin and/or its major components were determined using a duNouy tensionmeter. The results are shown in table 8 along with predictions of spreading and adhesion. The spreading predictions follow from equation 12, where if the value of the spreading coefficient S increases the tendency of the resin to spread on the particular soil (i.e., wetting) also increases. The adhesion predictions were made from the relative magnitude of the spreading coefficients.

After the spreading coefficients were determined, the results were correlated with actual tests of compressive strength from another portion of this study. The compressive strength is presumed to be an indication of the degree of adhesion. Both sets of data are shown below and the correlation is shown in figure 6.

Resin System	Spreading Coefficient dynes/cm			Compressive Strength psi		
	Sand	Clay	Silt	Sand	Clay	Silt
Aropol 7110	+36	+28	+17	2070+	1653+	1151+
Styrene				230-	114-	551-
Sodium Silicate N	- 9	-17	-28	322	76	30

From figure 6 it is seen that a good correlation was obtained between compressive strength and the spreading

6. L. H. Scharpe and H. Schonhorn, Advan. Chem. Series, 43, 189 (1964).

coefficients. Two possible explanations for this correlation are: (1) the criterion of spreading will predict adhesion as discussed at length above, and (2) the soil slugs used in the compression tests were made by pouring resin over the soil and allowing it to penetrate into the soil. Thus, the effect observed in figure 6 results from fortuitous differences in the rate of penetration as predicted by equation 8.

More work would be necessary to analyze the correlation of figure 6. If the spreading coefficient does indeed predict the compressive strength, then the effectiveness of new resins could be predicted simply by a determination of its surface tension and a knowledge of the surface-free energy of the soil on which it is to be applied.

PART V: CONCLUSIONS AND RECOMMENDATIONS

Conclusions

Based on the resins screened for dust palliatives, soil water-proofing agents and soil stabilizers, the following conclusions can be drawn:

1. Maximum unconfined compressive strength (>1000 psi) on soils was obtained with epoxy resins and unsaturated polyester resins. However, high use levels (11 and 6 pounds per square yard, respectively) were required to obtain 1 1/2 to 2-inch penetration by pouring the resin on the soil.
2. Intermediate levels of soil strengthening (200-1000 psi) were obtained with urethane, alkyd, acrylic, aniline-furfural, sodium silicate and plasticized sulfur resin systems.
3. Most of the resins had adequate resistance to water erosion. Notable exceptions were emulsion types such as Coherex, which washed away; the 15XPF gelatin sample, which resisted erosion but became flexible and easily broken presumably due to absorption of water; the aniline-furfural sample, which did not change but absorbed water; and Chem Rez samples, which exhibited some surface erosion.
4. Excellent resistance to freeze-thaw and wet-dry cycling was exhibited by composites prepared from epoxies, unsaturated polyesters, polyurethanes, and solvent dilutable alkyds. Samples prepared with sodium silicate, acrylic emulsions, and water soluble alkyds crumbled or spalled during one or both of the cycling tests. Reductions in unconfined compressive strength were observed with AM-9, sodium silicate, aniline-furfural and Emlon E-200. Some of these reductions in strength may not be real, but due to poor sample preparation.
5. Maximum water repellency was obtained with moisture-cured urethanes, octadecyl isocyanate, phenol-modified Chem Rez 200, and solvent system alkyds. The unsaturated polyester resins gave "fair" to "good" water repellency.
6. Based on economic considerations as well as performance results, the unsaturated polyesters with high levels of styrene and moisture-cure urethanes appear the most promising for soil treatment providing sufficient quantities can be used to develop strength. For water repellency without

dust palliation and soil strengthening, octadecyl isocyanate is the most effective. Areas not subjected to traffic in any form can be effectively treated with air-dry alkyds.

7. Although many of the other resin systems tested could be effective for soil stabilization and/or dust palliation and soil waterproofing, they were found unsuitable due to slow cure times in excess of four hours or to erratic cures.
8. Unsaturated polyester resins exhibited high flexural strengths when milled with sand. Values of over 2200 psi flexural strength were obtained using 20% of sand weight of a 30% resin solids Aropol 7510M in styrene.
9. Maximum elevated temperature storage stability on unsaturated polyester-styrene resin systems was obtained with a blend of 0.4% Adogen 464, a quaternary ammonium chloride, and 0.01% copper naphthenate (percentages based on resin weight).
10. Based on observations of field tests and on the requirements in the QMR issued August 1966, it is felt that for the resin to be effective for use in traffic areas at the low use level, the resin must have high elongation over 1000% and high tensile strength over 1000 psi.
11. Several urethane elastomers have been prepared with elongations over 2200% and tensile strength over 1000 psi.
12. Sand treated with the urethane elastomers passed the 150-mph air impingement and water erosion test. A flexible surface coating was formed.
13. When the urethane was applied on loose, dry clay, coverage was poor and shrinkage cracks formed in all the resins tested.
14. Wetting the surface with a very low level of water, not enough to cover the surface completely, and then applying the urethane resin with only 1/2 to 1/3 of the theoretical amount of curing agent resulted in a very satisfactory surface. These specimens passed the 150-mph air impingement and water erosion tests.

Recommendations

Urethane elastomers have shown excellent promise as treatment materials for dust palliation and soil waterproofing. Formulation changes can be made to achieve high elongation and high tensile strength. Research should be conducted to obtain elongation of over 2000% with tensile strength of 2000 psi. Several factors, which affect both elongation and tensile strength, should be evaluated. Among these are:

- Type of isocyanate used
- Type of polyol used
- Reaction conditions during polymer preparation
- Curing agent used

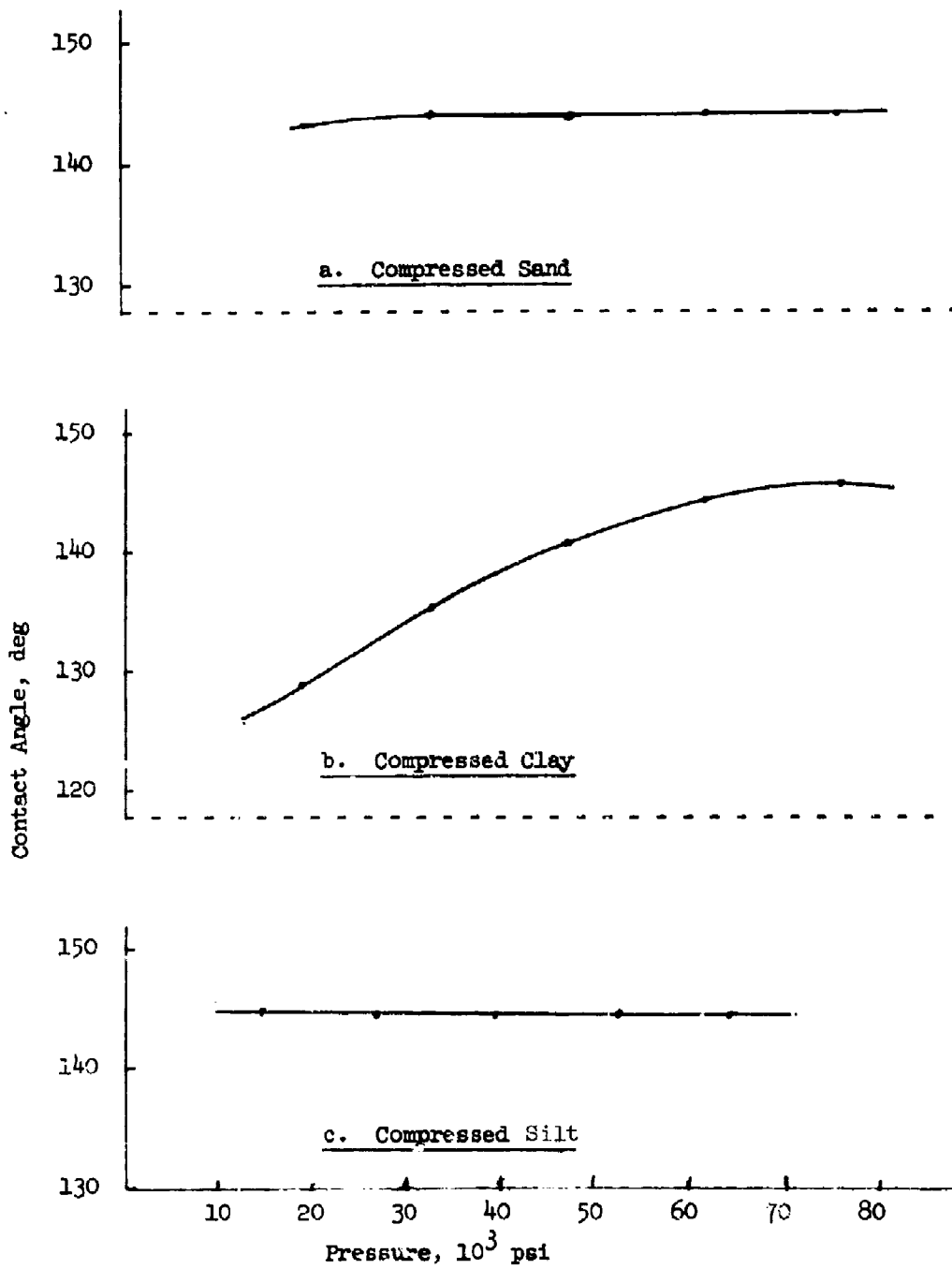


Figure 1. Contact Angles of Mercury

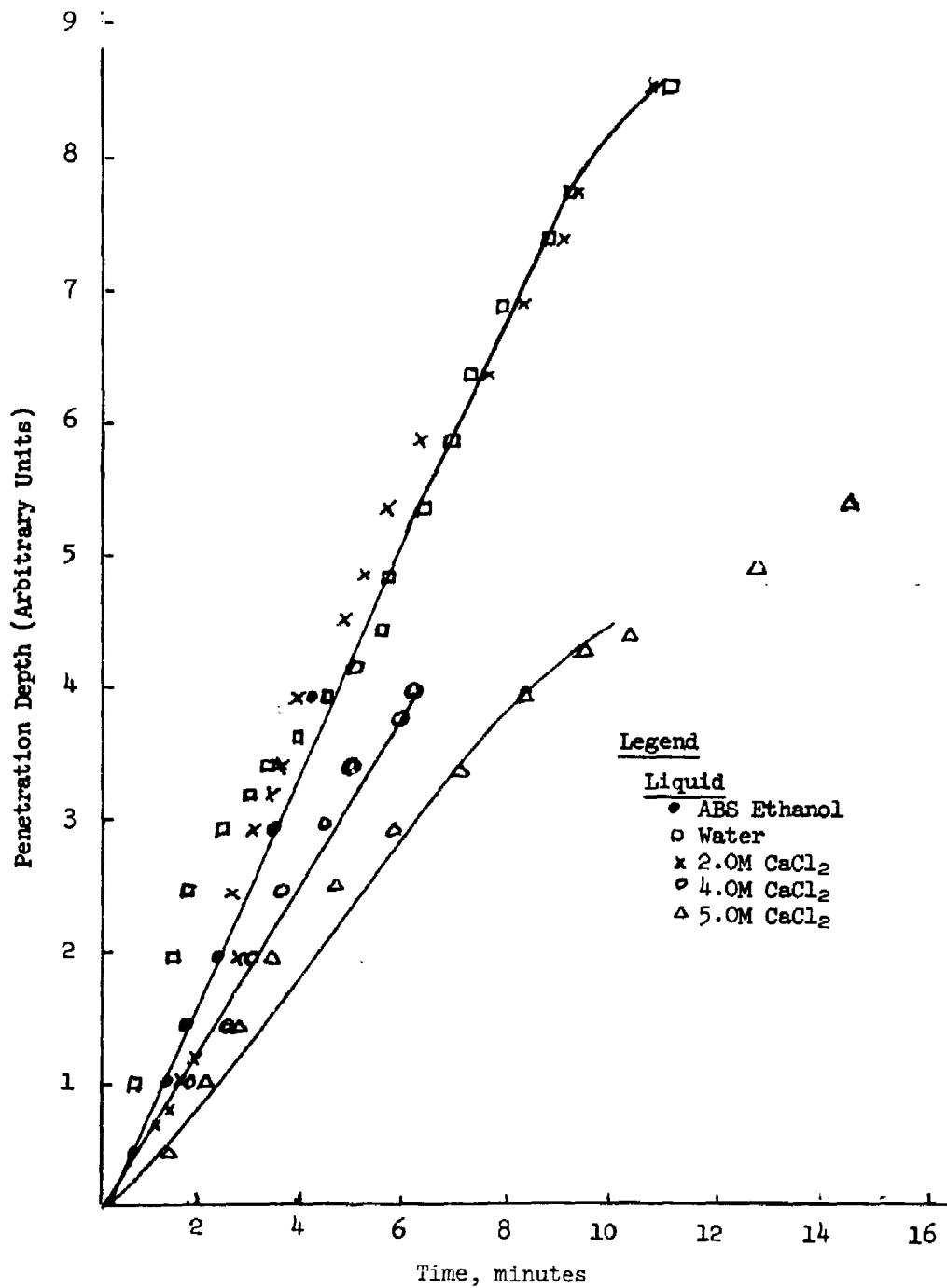


Figure 2. Liquid Penetration into Sand

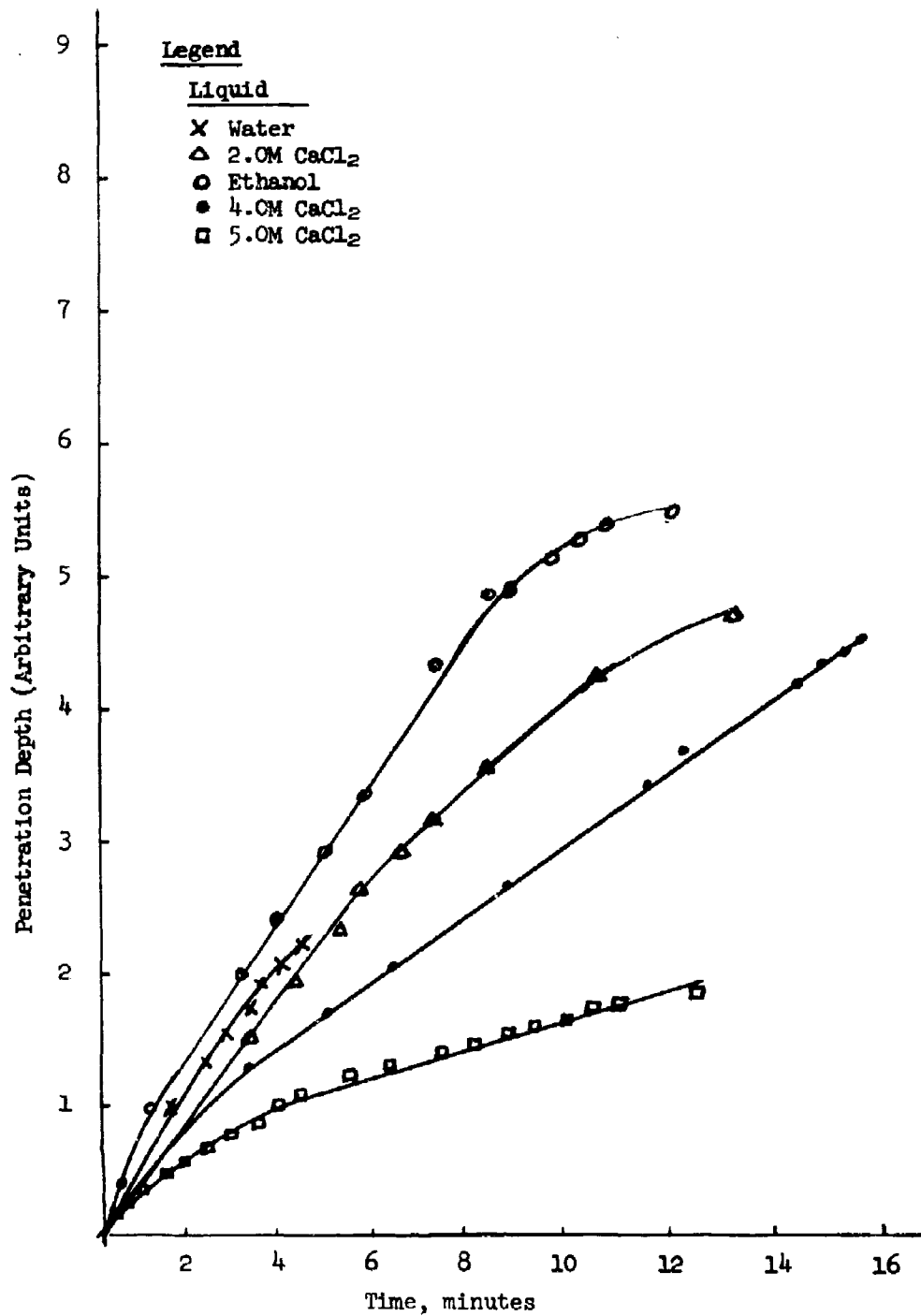


Figure 3. Liquid Penetration into Silt

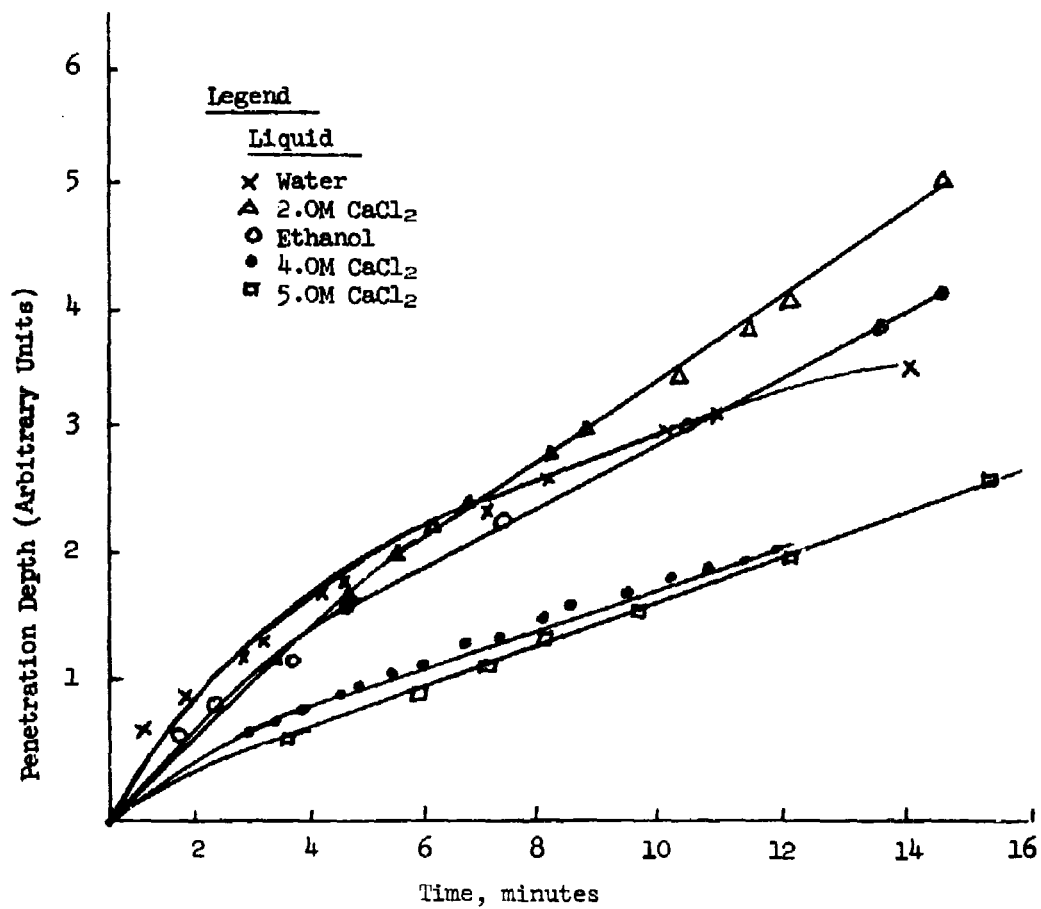


Figure 4. Liquid Penetration into Clay

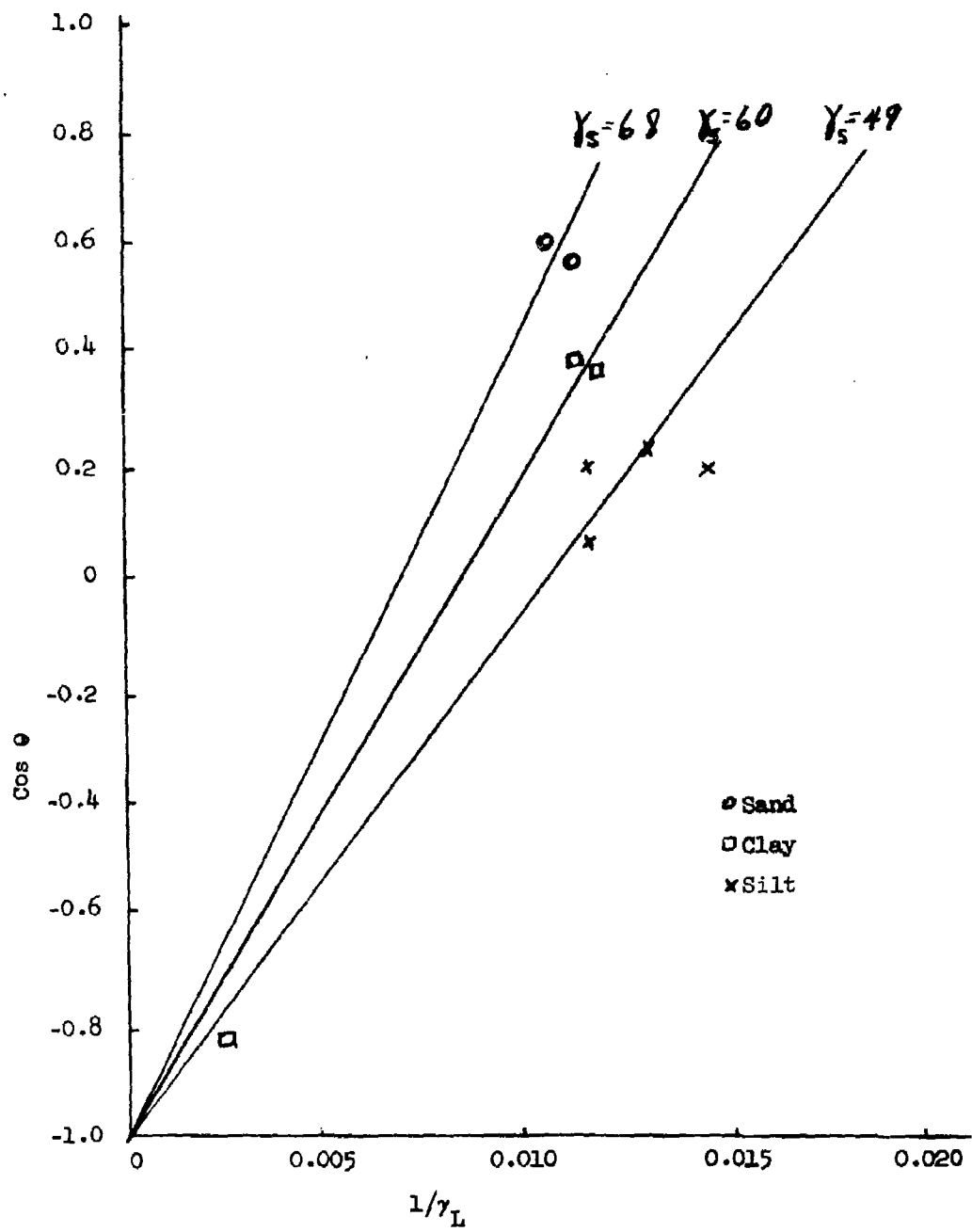


Figure 5. Estimation of Surface-Free Energies

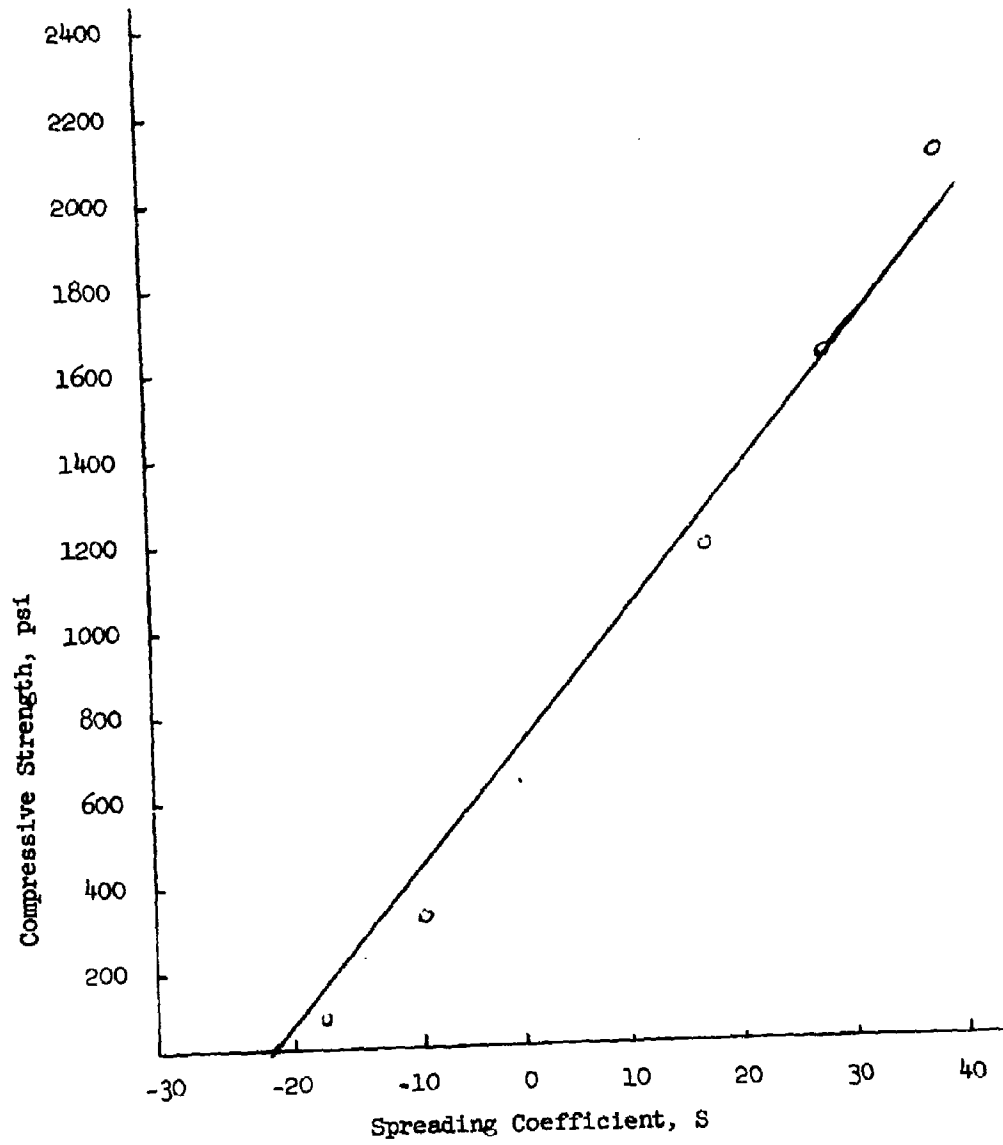


Figure 6. Correlation of Spreading Coefficient and Compressive Strength

Table 1
Resin Systems Evaluated in Initial Screening

Run No.	Type	Resin ^a Application		Solvent		Soil		Water Content, %	Curing Time, Hr	Penetration In. C	Water Repellency ^d	Remarks
		Rate, lb/yd ²	Catalyst	Type	Application Rate, lb/yd ²	Type	Compaction					
4A	Epon 828	1.3	Ashland S-1496	Solox	5.1	Sand	Loose	Dry	40	0.25	F	Crust crumbly
1A		5.2	Ashland S-1496	Solox	14.4	Clay	Loose	Dry	24	1.5	F	Hard case
17		5.5	Ashland S-1496	Solox	35.5	Clay	Loose	10	12	2.0	F	Break up easily
16		11.0	Ashland S-1496	Solox	12.7	Sand	Loose	Dry	12	2.0	F	Very hard
7A	Coherex:	2.9	None	Water	8.8	Sand	Loose	Dry	1.5	1.5	F	Did not harden, stayed moist
129		4.6	None	Water	4.6	Sand	Loose	10	2.0	0.25	F	Did not harden, stayed moist
130	Ashland Emul	4.6	None	Water	4.6	Clay	Loose	10	0.25	1.5	F	Did not harden, stayed moist
10A		2.9	None	Water	8.8	Sand	Loose	Dry	1.5	0.5	F	Did not harden, stayed moist
19A	15 XFF Gelatin	1.3	Cr ₂ (SO ₄) ₃	Water	11.8	Sand	Loose	Dry	1.0	1.5	F	Rubbery - 2/3 absorbed
11A	AM-9	4.3	Formaldehyde	Water	17.6	Sand	Loose	Dry	1.0	1.5	F	Hard, flex, easily broken
11A	AM-9	2.1	DMAP-42e	Water	8.8	Sand	Loose	Dry	2+	1.5	F	Flexible-porous
302	AM-9(3.0%) ^e		DMAP-42e	Water		Sand	Compact	10				Crumbly, soft
31	EP 8908-23	9.1	None	None	5.1	Sand	Loose	10	0.25	0.5	F	Surface skin
32		9.1	None	None	14.4	Clay	Loose	10	0.25	1.1	E	Surface skin
33		9.1	None	None	35.5	Silt	Loose	10	0.25	1.1	E	Surface skin
37		9.1	None	Water	4.6	Sand	Loose	10	2+	1.25	F	Surface skin
38		9.1	None	Water	4.6	Clay	Loose	10	2+	0.5	F	Surface skin
39		9.1	None	Water	4.6	Silt	Loose	10	2+	0.5	F	Surface skin
29	EP 8908-122	9.1	None	None	4.6	Clay	Loose	10	0.1	0.25	E	Surface skin
30		9.1	None	None	11.8	Silt	Loose	10	0.1	0.25	E	Surface skin
265	EP 8908-122 (6.6%) ¹		None	None		Sand	Compact	10	2+	1.5	F	Hard crust, crumbly
296	EP 8908-122 (8.2%) ¹		SO ₂	None		Sand	Compact	10	2+	1.5	F	Surface skin, no strength
34	EP 8908-129	9.1	None	Water	9.1	Sand	Loose	10	2+	2.0	F	Surface skin
35		9.1	None	Water	9.1	Clay	Loose	10	2+	1.0	F	Surface skin
36		9.1	None	Water	9.1	Silt	Loose	10	2+	1.0	F	Surface skin
17A	Chem Rez 200	2.3	H ₂ O ₄	Solox	5.4	Sand	Loose	Dry	2+	1.5	F	Soft, partly eroded by water
16A		4.6	H ₂ O ₄	Solox	3.6	Sand	Loose	Dry	2+	1.5	G	Soft, partly eroded by water
63	Phenol Mod	3.6	H ₂ O ₄	Solox	9.1	Sand	Loose	10	2+	2.0	G	Stayed wet, did not harden
64	Chem Rez 200	3.6	H ₂ O ₄	Solox	9.1	Clay	Loose	10	2+	2.0	P	Stayed wet, did not harden

a. Application rate of resin includes catalyst.

b. Water content based on dry weight of soil prior to addition of resin.

c. Depth of penetration of resin into soil. Null refers to blending resin into soil.

d. Approximately 0.5 ml of water added to surface 2 hr after resin was applied. Effectiveness of resin rated as follows:

E = Excellent, water as droplet after 10 min.

G = Good, water visible on surface after 5 to 10 min.

F = Fair, water visible on surface after 1 to 5 min.

P = Poor, water absorbed in 1 min or less.

e. Percentage based on dry weight of soil. Catalyst weight included in percent resin.

Table 1 (continued)

Run No.	Resin ^a			Solvent			Soil			Water Content, %	Curing Time, Hr	Penetration In.	Water Repellency ^d	Remarks
	Type	Application Rate, lb/yd ²	Application Rate, lb/yd ²	Type	Catalyst	Type	Type	Compaction	Content, %					
18A	Aniline-Purifural	3.0	3.0	Solox	17% HCl	Solox	Clay	Loose	Dry	2+	1.5		Hard crust	
14		3.1	4.1	Solox	17% HCl	Solox	Clay	Loose	Dry	2+	2.0		Hard crust	
15		4.7	3	Solox	17% HCl	Solox	Silt	Loose	Dry	2+	1.25		Hard crumbly edges	
285	Aniline-Purifural			Solox	17% HCl	Solox	Sand	Compact	10	2+	null	F	Thin surface shell	
	(6.7%) ¹			(1.3%) ¹										
24A	Eaton EPO	6.9	10.4	Water	DETA	Water	Sand	Loose	Dry	1.5	1.5		Absorbed slowly, cracked when dry	
53A		7.0	19.0	Water	DETA	Water	Clay	Loose	Dry	2.0	0.5		Upper 1/2 inch dried and separated from rest	
1		10	16.4	Water	DETA	Water	Sand	Loose	Dry	1.0	2.0		Very hard	
2		10	30.0	Water	DETA	Water	Clay	Loose	Dry	1.5	2.0		Hard, bottom crumbly	
3		10	23.6	Water	DETA	Water	Silt	Loose	Dry	1.5	1.5		Incomplete cure, foamed	
48A	P-2010(22.5%) ¹			None	Macomate	None	Sand	Loose	Dry	1.0	0.5	P	Incomplete cure, foamed	
47A	P-2010(33.5%) ¹			None	Macomate	None	Sand	Loose	Dry	1.0	0.5	P	Surface hardened, porous	
71	Na Silicate N	12	4.6	Water	NaSiF ₆	Water	Sand	Loose	10	1.0	0.25		Surface Hardened, porous	
72		12	4.6	Water	NaSiF ₆	Water	Clay	Loose	10	1.0	0.25		Very hard	
4	Na Silicate N			Water	NaSiF ₆	Water	Sand	Loose	Dry	1.0	Null		Very hard	
	(21%) ¹													
5				Water	NaSiF ₆	Water	Clay	Loose	Dry	1.0	null		Hard, crumbly edges	
6				Water	NaSiF ₆	Water	Silt	Loose	Dry	1.0	null		Breaks up readily	
19	Sulfur (16%) ¹			None	E-13	None	Sand	Loose	Dry	0.1	null		Very hard	
20				None	E-13	None	Clay	Loose	Dry	0.1	null		Hard, edges crumbly	
21				None	E-13	None	Silt	Loose	Dry	0.1	null		Very hard	
31A	Sulfur(23.6%) ¹			None	E-13	None	Sand	Loose	Dry	2+	1.5		Solidified on contact with sand	
35A	Aropol 7110	3.6	15.3	Styrene	BPO-DMA	Styrene	Sand	Loose	Dry	2+	0.5		Very hard, 15 min to penetrate	
43A		3.7	11.5	Styrene	BPO-DMA	Styrene	Clay	Loose	Dry	2+	0.5		Very hard	
34A		3.9	20	Styrene	BPO-DMA	Styrene	Silt	Loose	Dry	2+	1.5		Very crumbly edges, 5 min to penetrate	
30	Aropol 7110			Styrene	BPO-DMA	Styrene	Sand	Compact	10	2+	null	P	Very crumbly	
	(2.0%) ¹													
257	Aropol 7110			Styrene	BPO-DMA	Styrene	Sand	Compact	10	2+	null	F	Hard surface shell	
	(4.0%) ¹													
40	Aropol 7720M	7.3	7.3	Styrene	BPO-DMA	Styrene	Sand	Loose	10	2+	2.0	E	Very hard	
41		7.3	7.3	Styrene	BPO-DMA	Styrene	Clay	Loose	10	2+	2.0	F	Very hard	
42		7.3	7.3	Styrene	BPO-DMA	Styrene	Silt	Loose	10	2+	2.0	F	Hard	
60		7.3	3.6	Styrene	BPO-DMA	Styrene	Clay	Loose	10	1.0	1.5	E	Hard	
43	Arothane 160	7.3	7.3	Ethyl	None	Ethyl	Sand	Loose	10	2+	2.0	G	Very hard	
				Acetate	None	Acetate	Clay	Loose	10	2+	2.0	F	Very hard	
44		7.3	7.3	Ethyl	None	Ethyl	Silt	Loose	10	2+	2.0	E	Very crumbly	
45		7.3	7.3	Acetate	None	Acetate	Silt	Loose	10	2+	2.0	E	Very crumbly	

Table 1 (continued)

Run No.	M c s i l n Application		S o l v e n t Application		Type	Compaction	Water Content, % ^b	Curing Time, Hr	Penetration In. c	Water Repellency ^d	R e m a r k s
	T. J. P. e	Rate, lb/yd ²	Catalyst	Type							
73	Arothane 156	3.1	DBTDA	Ethyl Acetate	Sand	Loose-Compact	10	2+	2.0	E	Hard but crumbly
74		3.1	DBTDA	Ethyl Acetate	Clay	Loose-Compact	10	2+	1.0	G	Hard but crumbly
154	Arothane 170	1.1	TMBDA	Acetone	Silt	Compact	10	2+	0.2	E	Surface penetration only, skin
157		1.1	TMBDA	Acetone	Clay	Compact	10	2+	0.2	E	Surface penetration only, skin
140		2.0	None	Acetone	Sand	Loose-Compact	10	2+		F	
143		2.0	None	Acetone	Sand	Compact	10	2+		E	
146		2.0	None	Acetone	Sand	Compact	10	2+	0.25	E	Surface skin
151		2.0	TMBDA	Acetone	Clay	Compact	10	2+	0.15	E	Surface skin
162		3.0	None	Acetone	Sand	Loose	5	2+	0.15	E	Hard surface
141		3.0	None	Acetone	Sand	Loose	5	2+	0.25	E	Very hard throughout, surface abrasion fair to good
171		3.0	TMBDA	Acetone	Silt	Loose	5	2+	null	E	Hard surface, crumbly beneath
173		3.0	None	Ethyl Acetate	Sand	Compact	10	2+	null	E	Hard surface, slow cure, crumbly
280	Arothane 170 (4%)		None	Ethyl Acetate	Silt	Compact	10	2+	null	E	Crumbly skin
258	Arothane 170 (5%)		None	Ethyl Acetate	Clay	Compact	10	0.5	1.0	F	Sl hardening, crumbly
257		9.1	None	Styrene	Sand	Loose	10	0.5	0.15	F	Surface hardens
51	Arlon 110	9.1	None	Water	Clay	Loose	10	1	1	F	Poor cure
52		9.1	None	Water	Clay	Loose	10	2+	0.75	C	Hard surface, wet beneath
79	Arlon 310	4.6	None	Water	Sand	Loose	10	2+	0.5	F-G	Hard surface, wet beneath
80		4.6	None	Water	Clay	Loose	10	2+	1.25	G	Crumbly
55	Arlon 363	9.1	None	None	Sand	Loose	10	0.5	1.5	F	Very hard
56		9.1	None	None	Sand	Loose	10	2+	0.5	E	
327	Arlon 580	4.6	None	Water	Sand	Loose	10	2+	0.5	E	
82		4.6	None	Water	Sand	Loose	10	2+	0.5	E	
53		9.1	None	Water	Sand	Loose	10	2+	0.5	E	
105	Aroplaz 832	3.7	None	Ethyl Acetate	Sand	Loose-Compact	10	2+	0.5	E	
106		3.7	None	Acetate	Clay	Loose-Compact	10	2+	0.5	E	
340		4.5	None	Acetate	Sand	Loose	10	2+	0.5	E	Hard surface, uncured beneath
347		4.5	None	Acetate	Sand	Loose	10	2+	0.5	E	Hard surface, uncured beneath
83	Aroplaz 6008	4.6	None	Acetate	Sand	Loose	10	1.5	2	E	Did not harden
84		4.6	None	Ethyl Acetate	Clay	Loose-Compact	10	2+	0.75	E	Did not harden
107	Aroplaz 6065	3.7	None	Ethyl Acetate	Sand	Loose-Compact	10	2+	2	E	Very hard
108		3.7	None	Ethyl Acetate	Sand	Loose-Compact	10	2	0.5	E	Very hard
115	Arothane 170-C ₁₈ NEO	5.0	TEA	Ethyl Acetate	Sand	Loose-Compact	10	2	2	E	Slow cure
116		5.0	TEA	Ethyl Acetate	Clay	Loose-Compact	10		1.25	E	Hard

Table 1 (continued)

Run	Resin		Solvent		Catalyst		Application		Type	Compaction	Water Content, %	Curing Time, Hr	Penetration, In. C	Water Repellency ^d	Remarks
	1, 2, 3, 4	5, 6, 7	Type	Rate, lb/yd ²	None	Rate, lb/yd ²	None	Rate, lb/yd ²							
11	PP	305	Styrene	3.0	None		3.0	None	Sand	Loose	10	2+	2	P	No cure
12	PP	305	Mineral Spirits	2.5	None		2.5	None	Sand	Loose	10	2+	2	C	Stayed wet, no cure
13	PP	305	Mineral Spirits	2.5	None		2.5	None	Sand	Loose	10	2+	2	P	Stayed wet, no cure
14	PP	305	Ethyl Acetate	2.0	None		2.0	None	Sand	Loose	10	2	2	P	Stayed wet, no cure
15	PP	305	Ethyl Acetate	2.0	None	Ethylene Glycol	2.0	None	Sand	Loose	10	2	2	E	Surface hard, rest loose
16	PP	305	Ethyl Acetate	2.0	None		2.0	None	Sand	Loose	10	2+	2	P	No cure
17	PP	305	Ethyl Acetate	2.0	None		2.0	None	Sand	Loose	10	2+	2	E	Did not harden
18	PP	305	Ethyl Acetate	2.0	DBTDA		2.0	DBTDA	Clay	Loose	10	2+	1	E	Did not harden
19	PP	305	Ethyl Acetate	2.0	DBTDA		2.0	DBTDA	Clay	Loose	10	2+	1	E	Did not harden
20	PP	305	Ethyl Acetate	2.0	BPO-DMA		2.0	BPO-DMA	Sand	Loose	10	2+	0.5	E	Rubbery skin
21	PP	305	Ethyl Acetate	2.0	None		2.0	None	Sand	Loose	10	2+	0.75	C	
22	PP	305	Ethyl Acetate	2.0	None		2.0	None	Clay	Loose	10	2	0.5	E	
23	PP	305	Ethyl Acetate	3.0	None		3.0	None	Sand	Loose	10	2+	1	P	Very hard
24	PP	305	Ethyl Acetate	3.0	None		3.0	None	Clay	Loose	10	2+	0.75	P	Very hard
25	PP	305	Water	2.0	Stroc 2-3		2.0	Stroc 2-3	Clay	Loose	10	2+	1	P	Soft, no cure
26	PP	305	Water	2.0	Stroc 2-3		2.0	Stroc 2-3	Sand	Loose	10	2+	2	P	Soft, no cure

Table 2
Physical Test Data

Run No.	Resin		Solvent		Application		Soil		Sample Weight, gm		Water Erosion		Unconfined Compressive Strength, psi	
	Type	Rate, lb/yr ²	Type	Rate, lb/yr ²	Initial	After Wind Test	Type	Initial	After Wind Test	Initial	Dry	Initial	Thaw 8 Cycles	Wet Dry 8 Cycles
4A	Epon 828	1.3	Solox	5.1	351	344	Sand	351	344	360	314	1723	735	1180
3A		2.6	Solox	5.1	450	430	Sand	450	430	481	433			
2A		5.2	Solox	5.1	1600	1600	Sand	1600	1600	1675	1612			
55A		6.7	Solox	10.4	1762	1743	Sand	1762	1743	1765	1747			
56A		6.7	Solox	10.4	1622	1614	Loam	1622	1614	1642	1502			
16		11	Solox	16.4	1653	1622	Sand	1653	1622					
7A	Coherex	2.7	Water	8.6	1755	1746	Sand	1755	1746					
5A		11.5	Water	5.4	1671	1671	Sand	1671	1671					
10A	Ashland Exp Emul	2.3	Water	8.8	1694	1675	Sand	1694	1675					
7A		5.8	Water	5.4	1733	1705	Sand	1733	1705					
8A		11.5	Water	11.8	1786	1739	Sand	1786	1739					
19A	15 XFF Gelatin	4.3	Water	17.6	1155	1155	Sand	1155	1155					
11A	AM 9	2.1	Water	8.8	1031	1028	Sand	1031	1028					
17A	Chem Rez 200	2.3	Solox	5.4	1073	1071	Sand	1073	1071					
16A		4.6	Solox	3.6	1049	1047	Sand	1049	1047					
18A	Amaline-Purifural	3.0	Solox	9.1	1738	1738	Sand	1738	1738					
13		4.7	Solox	16.4	1369	1360	Sand	1369	1360					
14		4.7	Solox	16.4	1830	1823	Sand	1830	1823					
24A	Exlon E-200	6.9	Water	10.4	1762	1762	Clay	1762	1762					
23A		6.9	Water	10.4	1799	1796	Clay	1799	1796					
47A	Ms Silicate H- Mag-Sil 6	21.6%	Water	3%	1768	1762	Sand	1768	1762					
30A	Arpopol 7110	2.6	Styrene	15	1764	1764	Sand	1764	1764					
29A		6.0	Styrene	11.8	1814	1814	Sand	1814	1814					
44A		6.5	Styrene	15.5	1890	1890	Sand	1890	1890					
42A		8.7	Styrene	15.4	1890	1890	Sand	1890	1890					
289	Arachne 170	4.3%	Buyl Acetate	3%	706	738	Sand	706	738					

a. Withstood freeze-thaw and wet-dry cycling.
b. Not subjected to water erosion.
c. Washed away in 10 minutes.
d. Some surface erosion.
e. Not subjected to water erosion, weight after air dry.
f. Broke after third cycle.
g. Crumbled when immersed in water.
h. Surface spalled when immersed in water.
1. Per cent based on dry weight of soil.

Table 2 (concluded)

Run No.	Pest In Application		Solvent		Soil Type	Sample Weight, gm		Unconfined Compressive Strength		
	Type	Rate, lb/yd ²	Type	Rate, lb/yd ²		After Wind Test	Water Erosion	Initial	Thaw 8 Cycles	Wet-Dry 8 Cycles
321	Arlon 580	3.3%	Water	0.7%	Sand	Initial	90	62	8	
326	Arlon 310	3.3%	None		Sand	After Wind Test	122	193	8	
329	Arlon 110	3.3%	None		Sand	Initial	26		8	
362	Arlon 363	3.3%	None		Sand	After Wind Test	91	166	8	
364	Aropiaz 6008	3.3%	None	0.7%	Sand	Initial	107	363	189	
376	Aropiaz 6065	3.3%	JP-4	0.7%	Sand	Initial	109	150 ^h	162 ^h	
381	Aropiaz 839	3.3%	JP-4	0.7	Sand	Initial	255	382	363	
15A	EP 8508-23	7.7%	None		Sand	Initial	685		530	
309	EP 8508-122	6.6%	None		Sand	After Wind Test	1080		153	
342	EP 8508-129	3.3%	None		Sand	Initial	1107	1004	946	

Table 3
 Spray-On Application of Resins for Dust Palliation and Soil Waterproofing

Run No.	Type	Application Rate, lb/yd ²	Soil Type ^a	Pretest ^b	Appearance After Cure	Wind Test ^c	Water Test, gm inc. wt.	Wind After Water	Remarks
1F	Cat. Asphalt Fml.	25	Sand	no	Good surface	P	+231	P	
1B		32.5	Sand	no	Good surface	P	+226	F	
2B		2.6	Sand	no	Good surface	P	+230	F	
3B		2.6	Sand	no	Good surface	P	+254	P	
4B		2.4	Clay	no	Large shrink cracks	F			
5B		2.4	Clay	no	Shrink cracks	F			
6B		3.1	Clay	no	Shrink cracks	F			
7B		3.1	Clay	no	Shrink cracks	F			
8B		2.8	Clay	no	Many cracks	F			
9B		2.8	Clay	no	Shrink cracks	F			
10B		1.4	Clay	no	Shrink cracks	F			
11B	Epon 815	4.0	Clay	no	Shrink cracks	F			
12B	Epon 820	4.0	Clay	no	Shrink cracks	F			
13B	EP 8 23-122	3.0	Clay	no	Shrink cracks	F			
14B	EP 8 60-23	4.0	Clay	no	Shrink cracks	F			
15B	Arothane 170	3.0	Clay	no	Poor wet and cure	F			
16B		2.1	Clay	no	Poor wet and cure	F			
17B		3.6	Clay	no	Slight foaming	F	+142	P	
18B		3.0	Clay	yes	Good wetting, slight foam	P	+238	P	
19B		3.0	Clay	yes	Good wetting, slight foam	P			
20B		3.0	Clay	yes	Slight foaming	F	+142	P	DABCO-Co.
21B		2.8	Clay	yes	Good wetting	F			
22B		2.4	Clay	no	Good wetting, discarded	F			
23B	Aroplaz 6008	3.5	Clay	yes	Good wetting, physically spread	P	+ 47	P	Surface skin buckled in wind
24B	Aroplaz 6065	3.5	Clay	no	Good wetting	P	+238	P	Surface skin buckled in wind
25B	Aroplaz 832	3.5	Clay	yes	Good wetting	P	+127	P	Surface skin buckled, split
26B		3.5	Sand	no	Soaked in	P	+267	P-F	
27B		2.4	Clay	yes	Good wetting, soaked in	P	+1 P	P	
28B	Aroplaz 832-Aroplaz 6008	3.5	Sand	no	Good wetting	P			
29B		3.0	Clay	yes	Good wetting	F	+236	P	
30B		2.9	Sand	no	Soaked in	P			
31B	Aropol 7510H	3.1	Clay	no	Small cracks	P	+263	P	
32B		4.0	Clay	no	Resin rich surface	P			
33B		3.0	Clay	no	Soaked in, small cracks in edges	P	+21C	P	
34B		3.0	Sand	no	Crack on edge	P			Soil blew out of edge but resurfaced.
35B		2.4	Clay	yes	Crack on edge	P			

a. Sand used was bluff sand, clay used was dried, pulverized, Mississippi buckshot clay.
 b. Pretest with 1/2 lb/yd² water.
 c. Subjected to 150 mph wind one minute. P = passed, F = failed
 d. Subjected to forced water spray one hour.

Table 3 (concluded)

Run No.	Resin		Application Rate, lb/ft ²	Soil Type ^a	Pretest ^b	Appearance After Cure	Wind Test ^c	Water Test ^d gm inc. wt.	Wind After Water	Remarks
	Type	Solid								
13B	Arapol 7510M	50	3.0	Clay	yes	Sl. crack on edge	P	+225	P	1% Cab-O-Sil
14B		50	2.6	Clay	yes	Good surface	P	+151	P	
15B		50	2.4	Clay	yes	Good surface	P	+186	P	
102B		50	2.9	Clay	yes	Good surface	P	+256	P	Crack on edge
103B	Arapol 7510M/7720M	50	3.0	Sand	no	Good surface	P	+191	P	
107B		50	3.0	Clay	yes	Good surface	P	+95	P	
51B	Arapol 7720M	50	4.3	Clay	no	Good surface	P	+207	P	23% of 5% H ₃ PO ₄
52B	Chem Rez 200	50	3.2	Clay	yes	Good surface	P	+45	P	23% of 70% H ₃ PO ₄
53B		50	3.1	Clay	no	Good surface	P	+101	P	23% of 70% H ₃ PO ₄
54B		50	3.1	Sand	no	Good surface	P	+136	P	23% of 70% H ₃ PO ₄
55B		50	3.2	Clay	yes	Good surface	P	+164	P	23% of 70% H ₃ PO ₄
56B		50	2.0	Clay	no	Good surface	P	+97	P	13.6% H ₃ PO ₄ , 3.8 cw-1000
62B	Chem Rez 300	50	3.1	Clay	no	Good surface	P	+11	P	20% H ₃ PO ₄
63B		72	3.4	Clay	no	Good surface	P	0	P	16% H ₃ PO ₄ , 12.5% DER 741
64B		50	2.5	Clay	no	Good surface	P	8	P	15.5% H ₃ PO ₄ , 12% CW 200
65B		85	3.4	Clay	no	Good surface, some flex	P	10	P	14% H ₃ PO ₄ , 20% Epon 812
66B		70	3.0	Clay	no	Good surface	P		P	20% of 70% H ₃ PO ₄
57B	Chem Rez 200	47.5	3.5	Clay	no	Good surface	P	+31	P	
97B	Arapol 832 (30%) Chem Rez 300	75	2.6	Clay	yes	Good coverage	P	+271	P	H ₃ PO ₄
99B	DER 736 (20%) Chem Rez 300	75	3.0	Clay	no	Poor cure, soaked in	F		P	H ₃ PO ₄
100B	Epon 812 (20%)	75	1.5	Clay	yes	Good surface	P	+250	P	Tested 1/2 hr after appl. H ₃ PO ₄
106B	Admex 710	100	2.8	Clay	yes	Good surface	P	+108	P	Very flex, poor tear strength
109B	EL0	100	3.0	Clay	yes	Good surface	F	+82	P	Sl flex, fair tear strength
110B		100	3.0	Sand	no	Good surface	P	+72	P	Sl flex, fair tear strength
112B	2482-72-2	50	2.9	Clay	no	Good cover, soaked in	P	+102	P	Poor flex
113B	2343-61	50	3.0	Sand	yes	Good cover, soaked in	P	+173	P	Poor flex
114B	2482-72-1	60	2.8	Clay	no	Good cover, soaked in	P	+27	P	
115B	2343-61	50	3.0	Sand	no	Good cover, part soaked in	P	+50	P	1% Cab-O-Sil
116B	2482-72-1 Styrene-butadiene latex	50	3.0	Sand	no	Soaked in	P	+153	P	10 hr cure, poor flex
117B	EP6911-41	50	3.7	Clay	no	Several cracks	F		P	
118B		50	3.1	Clay	no	Good cover, part soaked in	P	+150	P	Fair flex

Table 4

Flexural Strength Data

Aropol 7510 M (30% solid in styrene, 6 wt % resin, 14 wt % styrene) and Dry Sand

Run No.	Catalyst, % ^a		Water Repeilency ^b	Flexural Strength, psi		Remarks
	Co	IMA/H ₂ O ₂		3-day Cure	5-day Cure	
106B	.5	.5/1.5	E	1719	2250	
108B	.5	1/1	E	1087	1654	
111B	.5	1.5/1	G-E	809	1248	
112B	.5	1.5/1.5	E	1302	1500	
113B	.5	1.5/1.5	E	1510	1923	
114B	1	.5/1	E	1380	1902	
115B	1	.5/1.5	L	1296	1780	
116B	1	.5/2	E	1482	1872	
117B	1	1/1	E	1690	2145	
118B	1	1/1.5	E	1513	1702	
119B	1	1/2	E	1534	1769	
120B	1	1.5/1	E	1413	1736	
121B	1	1.5/1.5	E	1680	2017	
122B	1	1.5/2	E	1351	1595	
123B	1.5	.5/1	E	1700	2163	
124B	1.5	.5/1.5	E	1302	1831	
125B	1.5	.5/2	E	1482	1782	
126B	1.5	1/1	E	1361	1920	
127B	1.5	1/1.5	E	1208	1780	

a. Based on weight of resin + styrene

Co = 6% cobalt naphthanate, IMA - dimethyl analine, H₂O₂ - 50% hydrogen peroxide.b. Approx 0.5 ml water added to surface 2 hours after resin application. Effectiveness rated as follows:
E = excellent, water as droplet > 10 min; G = good, water visible 5-10 min; F = fair, water visible 1-5 min; and P = poor, water soaks in < 1 min.

Table 4 (concluded)

Run No.	Catalyst, % ^a Co/IMA/K ₂ O ₂	Water Repellency ^b	Flexural Strength, psi		Remarks
			3-day Cure	5-day Cure	
128B	1.5/1/2	E	1468	1831	10% water in sand
129B	1.5/1.5/1	E	1184	1553	10% water in sand
130B	1.5/1.5/1.5	E	1768	2047	10% water in sand
131B	1.5/1.5/2	E	1351	1663	10% water in sand
132B	1/1/1		321	302	10% water in sand
133B	1/2/2		263	301	10% water in sand
134B	1/3/3		220	260	10% water in sand
135B	2/1/2		400	374	10% water in sand
136B	2/2/3		315	312	10% water in sand
137B	2/3/1		378	412	10% water in sand
138B	3/1/3		232	215	10% water in sand
139B	3/2/2		292	284	10% water in sand
140B	3/3/1		354	343	10% water in sand

Table 5

Storage Stability of Unsaturated Polyester Resin at Elevated Temperatures

Ref	Unsaturated Polyester		Inhibitor ^b	Per Cent	Stability - Days to Gel at	
	Type	Solids, % ^a			158 F	130 F
1	Aropol 7010	75	--	0.01	6	
1A		75	Ashland Proprietary Inhib		12	18
5		40	--	0.5	4	46
57		40	Copper naphthanate		11	27 ^c
58		40	Ferric naphthanate		13	64 ^c
59		40	Adogen 464		20 ^c	42
105		40	DMIA-HCl	0.75	23	55 ^c
68		40	DMIA-HCl	0.50	16	85
103		40	DMIA-HCl	0.25	4	63
9		30	--	-	15	21
9A		30	Ashland Proprietary Inhib	0.01	19	130
88		30	DMIA-HCl	0.25	4	
22	Aropol 7110	75	--	0.01	12	
22A		75	Ashland Proprietary Inhib		11	52
69		50	Adogen 464	0.5	4	
23		45	--	-	4	
24		40	--	-	4	
26		35	--	-	4	18
26A		35	Ashland Proprietary Inhib	0.01	14	
27		30	--	-	4	
34	Aropol 7240 MC	50	--	-	6	20
34A		50	Ashland Proprietary Inhib	0.01	20	
35		25	--	-	4	
35A		25	Ashland Proprietary Inhib	0.01	16	
36	Aropol 7410 M	70	--	-	32	
36A		70	Ashland Proprietary Inhib	0.01	32	
37		30	--	-	6	
37A	Aropol 7420	30	Ashland Proprietary Inhib	0.01	20	25
77		50	--	-	6	
78	Aropol 7510 M	40	Adogen 464	0.5	18	16
38		75	--	-	11	

a. Per cent plastic in resin system, balance is styrene monomer

b. Inhibitor used in addition to 100 ppm hydroquinone: Copper naphthanate - 5% copper naphthanate; ferric naphthanate - 6% ferric naphthanate; Adogen 464 - quaternary ammonium chloride; DMIA-HCl - dimethyl lauryl amine hydrochloride; BHA - butylated hydroxy anisole.

c. Polystyrene formed on inside cover of container.

Table 5 (concluded)

Ref	Unsaturated Polyester		Inhibitor ^b	Fw Cent	Stability - Days to gel at		
	Type	Solids, % ^a			158 F	130 F	110 F
38A	Aropol 7510 M	75	Ashland Proprietary Inhib	0.01	14	21	25
80			--	0.5	5	150	
79			Adogen 464	1.0	25	101	
110			DMLA-HCl	0.75	25	111	
111			DMLA-HCl	0.5	25	130	
112			DMLA-HCl	0.4	25	55	
116			DMLA-HCl	0.04	33	53	
92			BHA	0.1	11	53	
113			BHA	0.25	11	53	
114			BHA	0.5	12	53	
115			BHA	0.25	15	115	
96			Adogen 464	0.5	19	79	
95			Adogen 464	1.0	45	160 ^c	
94			Adogen 464	0.4/0.01	30	22 ^c	
97			Adogen 464/Cu Naph	0.5	38	22 ^c	
98			DMLA-HCl	0.25	15	22 ^c	
42	DMLA-HCl	0.01	13	24			
42A	Ashland Proprietary Inhib	0.01	9	31			
81	--	0.5	24	34			
82	Adogen 464	0.01	11	35			
46	Ashland Proprietary Inhib	0.01	11	24			
46A	Ashland Proprietary Inhib	0.01	18	35			
47	Adogen 464	0.5	24	42			
83	Aropol 7010/7510 M	40	Adogen 464	0.5	34	52	
84	Aropol 7420/7510 M	40	Adogen 464	0.5	24	42	
85	Aropol 7510 M/7720 M	40	Adogen 464	0.5	34	89 ^c	

Table 6

Physical Properties of Urethane Elastomer Resins

Run No.	Resin	MOI/OH ^a Ratio	Curing Agent ^b	Ratio	Catalyst ^c	Tensile Strength psi	Elongation, %	100% Elong- ation, psi	Tensile Strength 300% Elong- ation, psi	Ultimate Elongation Per Cent
2348-65-39	UCAR latex 131	3-5/1	Moisture		DABCO	1330	195	920		
43	Diol-TDI	3-5/1	Moisture		DABCO	2090	870	750		
45		3-5/1	Moisture		DABCO	1127	970	382		
64		3-5/1	Moisture		DABCO	825	880	345		
47		3-5/1	Moisture		DABCO- Co Naph	1275	950	470		
48		3/1	Moisture		DABCO- Co Naph	375	1125	210		
63		3/1	Moisture		DABCO	775	750	325		
79		3-5/1	Triol		Sn Octo	1577	65	50	56	
80		3-5/1	Polyester diol		Sn Octo	58	1000	140	528	
212		4/1	Moisture		DABCO	710	832			
287	Polyester Amide Diol-TDI	3-5/1	Glycerine		DABCO	1630	130	1305		
326	Polyester Diol TDI Polyether Diol TDI	3-5/1	Polyester Diol		DABCO	1100	475	475	635	
432	2000 MW Diol-HDI	3/1	Arom dia-		DABCO	1085	590	370	570	
476		2/1	Polyether Diol		DABCO	410 ^d	1200 ^d	120	160	
485		2/1	425 MW Diol		DABCO	260 ^d	1200 ^d	80	130	
502		2/1	Dia 200-Diol 134	5/1	DABCO	690 ^d	1150 ^d	165	230	1820
498		2/1	Dia 200-Diol 300	25/1	DABCO	265 ^d	1380 ^d	55	95	
568		2/1	Dia 200-Diol 425	25/1	DABCO	1020 ^d	1350 ^d	205	290	
569		2/1	1/2 theo use level		DABCO	565 ^e	1330 ^d	110	160	
513		2/1	Dia 200-Diol 52	5/1	DABCO	810 ^d	1290 ^d	145	225	1740
332		2/1	Dia 200-Diol 425	5/1	DABCO	825 ^d	1640 ^d	85	130	
333		2/1	Dia 200-Diol 134	5/1	DABCO	415	1250 ^d	125	165	1880
570		2/1	Dia 200-Diol 134	5/1	DABCO	650 ^d	1180 ^e	100	135	2040
598		2/1	Dia 200-Diol 52	25/1	DABCO	630 ^d	1230 ^d	110	165	2240
600		2/1	Dia 200-Diol 52	25/1	DABCO	635	1240 ^d	83	133	
601		2/1	Dia 200-Diol 90	5/1	DABCO	765	1130	113	192	
602		2/1	Dia 200-Diol 52	25/1	DABCO	810	1100	198	377	
603		2/1	Dia 200-Diol 52	25/1	DABCO	610	1080	97	157	
604		2/1	Dia 200-Diol 90	5/1	DABCO	1310	1170	220	312	
605		2/1	Dia 200-Diol 52	25/1	DABCO	895	1160	110	171	
506		2/1	Dia 200-Diol 52	5/1	DABCO	640	1020	117	200	
506		2/1	Dia 200-Diol 90	5/1	DABCO	935	950	217	314	

a. Refers to molar ratio of isocyanate to hydroxyl in prepolymer.

b. Numbers refer to molecular weight. Ratio refers to molar ratio of diamine to diol.

c. Catalyst used to promote cure: DABCO - tertiary amine; Co Naph - 6% cobalt naphthenate; Sn Octo - 8% stannous octoate; and DABTL - dibutyltin dilaurate.

d. Elongation beyond maximum of machine, did not break.

e. 65% in MeCl₂.

Table 7

Date Used in Evaluation of Cosine θ

<u>Soil</u>	<u>Liquid</u>	<u>Surface Tension of Liquid</u>		<u>Slope</u>	<u>Slope</u>
		<u>dynes/cm</u>	<u>Relative to H₂O</u>		
Sand	EtOH	22.27	1.19*	1.80	1.0
	H ₂ O	72.3	11.0 *	‡	‡
	2M CaCl ₂	78.9	1.85**	‡	‡
	4M CaCl ₂	86.9	4.4**	1.33	0.519
	5M CaCl ₂	90.4	8.5**	1.029	0.577
Silt	EtOH	22.27	1.19*	0.592	1.0
	H ₂ O	72.3	11.0 *	0.500	0.178
	2M CaCl ₂	78.9	1.85**	0.432	0.231
	4M CaCl ₂	86.9	4.4**	0.267	0.193
	5M CaCl ₂	90.4	8.5**	0.125	0.0786
Clay	EtOH	22.27	1.19*	0.35	1.0
	H ₂ O	72.3	11.0 *	‡	‡
	2M CaCl ₂	78.9	1.85**	‡	‡
	4M CaCl ₂	86.9	4.4**	0.178	0.372
	5M CaCl ₂	90.4	8.5**	0.167	0.393

* Data from Lang's Handbook.

** Data from International Critical Tables

‡ Not calculated - see text.

Table 8

Prediction of Spreading of Resin Systems on Soil Samples

Resin System	γ_L dynes/ cm	Spreading Coefficient S(equation 12)* dynes/cm			Adhesion		
		Sand	Clay	Silt	Sand	Clay	Silt
Aropol 7110	45						
Styrene	32	+36	+28	+17	Good	Good	Fair
Arothane 170	29						
C ₁₈ NCO	31	+37	+29	+18	Good	Good	Fair
Chem Rez A-200	41	+27	+19	+ 8	Good	Fair	Fair
Sodium Silicate "N"	77	-19	-17	-28	Poor	Poor	Poor

Note:

* γ_S values are correlated with lowest γ_L value of resin systems since components with low γ_L tend to be surface active.

$$\gamma_S (\text{Sand}) = 69 \text{ dynes/cm}, \gamma_S (\text{Clay}) = 60 \text{ dynes/cm}, \gamma_S (\text{Loam}) = 49 \text{ dynes/cm}$$

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13. ABSTRACT The object of this study was to evaluate various commercially-available resin systems, both organic and inorganic, to determine if they could be utilized as dust palliatives, soil waterproofing agents, and soil stabilizers. The screening tests revealed that many of the resin systems would perform as dust palliatives or soil waterproofing agents for nontraffic areas. However, none of these used on loose soil at the low use level of 3 lb/sq yd had enough strength or flexibility to perform satisfactorily under traffic. At two or three times this rate (6-9 lbs/sq yd), sufficient strength could be obtained with epoxy and unsaturated polyester resins to withstand some traffic. The emphasis was then changed to laboratory-synthesized urethane elastomers with over 2000% elongation and over 1000 psi tensile strength. When applied on loose sand, these elastomers gave a tough, flexible surface coating. Some problems with surface cracking were encountered on loose, dry clay, due to shrinkage. However, it was possible to eliminate or minimize these cracks by formulation changes of the elastomer and its curing agent, and by a very light prewetting of the soil with water. The discussion of the test results in the main body of the report is in chronological order. The Appendix covers the work done under the Physical Chemistry studies early in the contract period.			

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