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# Lignins As Stabilizing Agents for Northeastern Iowa Loess

By S. P. SINHA, D. T. DAVIDSON and J. M. HOOVER

## INTRODUCTION

One of the common problems in highway and airport construction faced by engineers all over the world is the scarcity of suitable materials for base and subbase construction. Lately, there has been such an increase in traffic on highways and airfields that the problem of suitable base and subbase design can no longer be ignored. Engineers have been striving hard to find ways and means to solve this problem. One solution lies in the utilization of locally available materials. Since soil is the most plentiful of available materials, it has been chosen by engineers and scientists to do the job. Unfortunately, not every soil in its original condition can be utilized as an engineering material without modification of its properties. For this reason, emphasis is being placed on finding ways to make suitable construction material out of unsatisfactory soil; the art and science of doing this is called "soil stabilization."

There are several methods of stabilizing soils. One of such methods is the use of admixtures. Admixtures such as Portland cement, bituminous material, lime-fly ash, lime, and lignins are a few of the possibilities. Since 1947 the Iowa Engineering Experiment Station has been conducting research on soil stabilization. Various kinds of admixtures have been investigated during this period. This report presents the work done to date with lignins as stabilizing agents for northeastern Iowa loess and loess derived soil.

## REVIEW OF LITERATURE

### MANUFACTURE OF LIGNINS

Lignin is the natural cement that binds the fibers of wood together in plants (37). Because the chemical structure of lignin is still unknown, it is difficult to define it appropriately. The term "lignin", therefore, cannot be considered the designation of a constitutionally defined compound, but is more a collective term for a group of high molecular, amorphous compounds which are chemically very closely related to other natural high molecular products (4).

Several processes are used at present to isolate lignin from pulping liquor, and the processes employed vary with the properties of lignin. The most common is the sulphite process. The lignin obtained by this process is called "lignosulfonate" and is water soluble. It forms a major constituent of solid residues of the so-called "spent sulphite liquor."

The lignin liquor comes from the digester in a solution having approximately 8 per cent solids. In this form the liquor is commonly used on "gravel" roads and streets near pulp mills to reduce dust and the effects of frost action. When used for such purposes the spent sulphite liquor may be referred to as Road Binder. Concentrated Road Binder can be prepared by condensing the dilute liquor, largely through evaporation, to a desired per cent of solids solution, usually 46 to 50 per cent solids (60). Complete drying and pulverization produces the powdered form of lignin.

The 113 sulphite pulp mills in the United States and Canada produce over 30,000,000 gallons of spent sulphite liquor a day. One ton of pulp wood produces about 950 pounds of cellulose used for paper while the remaining 1,050 pounds of solid is recovered in the spent sulphite liquor. This quantity of liquor contains about 750 pounds of lignins, 250 pounds of resins and other substances (50).

Since the development of uses for spent sulphite liquor is still in its infancy, the material is abundantly available and is very economical as a road construction material; the liquor being sold at about 6 cents per gallon in 55 per cent solids form at the mill (21). When diluted to 25 per cent solids at the railroad siding, the cost would be about two cents per gallon plus freight; the latter being six cents per gallon from Appleton, Wisconsin to Des Moines, Iowa. The northeastern Iowa area investigated for the possibility of the use of spent sulphite liquor is much nearer to the Wisconsin mills producing liquor than Des Moines, thus reducing the cost. The cost would be further reduced if the lignins were available in a more concentrated form, providing the cost of concentrating the liquor does not exceed the added cost of hauling the material at a lower concentration.

## DEVELOPMENT OF LIGNIN SOIL STABILIZATION

So called road binders made from a lignin base were first utilized in Sweden about 50 years ago (1, 28, 31). The factor encouraging its use was primarily economic. Since the local supply of road binding oils was inadequate, a large quantity of road binding oils had to be imported. In search for a cheaper road binding material, the Swedish Government started experimenting with the sulphite liquor available in large quantities throughout the country.

The use of spent sulphite liquor as a road material in the United States resulted from a search for methods of disposing of it. Fifteen years ago, this material had no value in the United States and was wasted by dumping into streams or rivers. This brought serious objections from the public because of its pollution effects.

In the early part of this century, several patents were issued in the United States, Germany, France, and in Sweden, on the use of spent sulphite liquor as a road binding material (6, 10-15, 22, 27, 31, 35, 39, 43, 44-46, 51, 55, 56). One of the most common uses of spent sulphite liquor in road construction has been as a dust palliative (3, 10, 15-19, 24, 28, 29, 33, 41, 44-47, 54-55, 60, 63). It has been used to a limited extent as a stabilizer for base and surface courses (3, 12, 33, 38, 61-63), and it has been found very successful in the prevention of frost heave (17, 23). Its action as a soil dispersant, and thereby its beneficial effects on such soil properties as density, compaction, optimum moisture content, capillarity, and permeability have been reported (12, 38). Spent sulphite liquor used in amounts ranging from 3 to 10 per cent of the soil dry weight together with a chromium salt such as potassium bichromate or sodium bichromate has been found to form a tough gel having binding and water proofing properties (22).

### *Work Done on Spent Sulphite Liquor in Europe*

Spent sulphite liquor has been used as a road construction material in Europe for nearly six decades, Sweden topping the list of the countries using it. Of the 55,000 miles of public roads in Sweden, 52,000 miles were gravel roads at the end of 1939. During that year, 33,000 miles of the gravel roads were treated with dust binding material (34). The work in the beginning was confined mainly to dust control (1). The Institute of Road Research at Stockholm found in their dust control experiments with sulphite liquor that it reacted well with dust and bound the particles together if the road surface was rich in clay. The Institute also studied the binding power of spent sulphite liquor (23), using a mixture of stone dust and the liquor. Molded specimens containing various percentages of liquor were tested for shearing strength. Good results were obtained when the amount of the liquor was 2 per cent of the dry weight of the dust. It was also observed that the use of lignin in dry solid form provided higher early strength, whereas the concentrated liquor when thoroughly dry gave the best results after 28 days curing.

Anger (2) investigated the effects of spent sulphite liquor on some soils which were susceptible to frost action. A mixture of sulphite liquor and sodium bichromate was used as a binder. Since the addition of sodium bichromate with spent sulphite liquor forms a gel, and acts as a waterproofing agent, it was observed that a solution of 9 per cent concentrated spent sulphite liquor (50 per cent solids) and 10 per cent bichromate solution (50 per cent $Na_2Cr_2O_7$) added to the soils in such proportions that the amounts of binder corresponded to 2, 4, 6, and 8 per cent of the dry soil weight reduced significantly the intake of capillary moisture. The use of 2 per cent of this binder was found to be economical as well as beneficial. Eight per cent of sulphite liquor used alone reduced capillary absorption of moisture considerably (2). During the 28-day testing period, one observation worth mentioning was that untreated soil continued to take up moisture even after 28 days, but the treated soil stopped such absorption long before the testing period was over. It was concluded that a frost susceptible soil can be made resistant to frost action through use of a binder of spent sulphite liquor and a bichromate.

### *Work Done in America*

The first published reports on the use of spent sulphite liquor in this country originated with the work done by the Raylig Division, Raynier Incorporated, on the West Coast (62). The report gives the methods of application of spent sulphite liquor for various end results.

The states of New Jersey, Washington, Maryland, and Idaho have made much use of spent sulphite liquor as a dust palliative, road stabilizer, and base treatment with improving wearing courses (38, 62). The state of New Jersey has used spent sulphite liquor for thirty years, primarily as a stabilizer preliminary to a bituminous treatment. Spent sulphite liquor was found to be beneficial in the prevention of spring break up of roads (62).

The state of Idaho in 1937 used concentrated spent sulphite liquor for surface treatments on several roads in the state. Inspection in the spring of 1938 revealed that the binder had afforded relief from dust and had conserved surface aggregate during the summer. It had slowly diffused downward, tending to produce a stabilized base course of increasing thickness (42).

The city of Spokane, Washington, reported in 1943 that diluted sulphite liquor had been used for twelve years as a dust layer on secondary city streets, and indicated the results obtained were very satisfactory (62). Mason County, state of Washington, has reported on the effects of heavy rainfall on sulphite liquor treated roads. Frequent applications of sulphite binder and grading of the road surface were found to be necessary during the rainy season. The sulphite liquor treated roads did not suffer from frost heave, but untreated roads in the same neighborhood suffered badly from frost heave.

The Quebec, Canada Department of Roads conducted some laboratory tests to determine the bearing capacity of raw gravel, clay stabilized gravel, and gravel treated with lignin extract (22, 26).

They concluded that the bearing capacity of gravel treated with 1.2 per cent lignosol was higher than that of the raw gravel and the clay stabilized gravel. Compression and absorption tests were also made. A definite increase in compressive strength was noted with the addition of 2 per cent of lignosol. The absorption of water through capillary action was noticeably reduced. It was observed that the unconfined compressive strength of the dried lignin treated gravel increased almost proportionately with the amount of lignosol used. Moisture-density relationship tests showed that an increase in the amount of sulphite liquor added to the soil increased the density and reduced the optimum moisture content.

In 1954, Lamb (30) reported that calcium lignosulfonate acts as a dispersing agent and has beneficial effects on soil properties. Smith and Hough at Cornell University, worked on chrome-lignin soil stabilization, and found that an admixture of spent sulphite liquor and bichromate increases the stability of soil and acts as a waterproofing agent (25).

## MECHANISM OF LIGNIN SOIL STABILIZATION

The binding effect of sulphite liquor appears to be due to the lignosulfonate, which is the major constituent of the solids in it. Lignin serves as a glue (38). It is believed that sulphite liquor has a marked influence on the surface properties of the fine particles of a soil system (26, 42). However, there is no definite proof as to how it alters the properties which improve soil stability.

Lambe (30) studied the effect of calcium and magnesium sulfonate, used in trace amounts with other additives, on the properties of soil. His studies indicated that lignin acts as a dispersing agent. Because of dispersion, the largest voids are destroyed, and since the breakdown of aggregates furnishes smaller particles that can fit into void spaces, greater density is achieved. It is also believed that the permeability of soil treated with spent sulphite liquor is decreased considerably. This helps in preventing frost heave. Research carried out at Ecole Polytechnique in Canada, under the sponsorship of the Department of Roads, Quebec, Canada (26), also showed that spent sulphite liquor has a dispersing action on the soil fines, and that it decreases the required moisture content to attain maximum density.

## LABORATORY INVESTIGATION

No standard method had been developed for evaluating the stability of lignin-soil mixtures. Previous studies, however, have shown that there are several variables which affect the stability. Figure 1 shows diagrammatically some of the more important ones. Time has not permitted an investigation of all the variables shown; however, an attempt has been made to study a few of them. The results to date are described in this report.

## PROPERTIES OF SOILS AND LIGNINS

### *Properties of Soils Used*

Three samples of northeastern Iowa loess and loess-derived soil[a] were used in this study. Loess deposits occur over an area of about 4,000 square miles in northeastern Iowa (34). It is believed that northestern Iowa loess for the most part was deposited during the Iowan and Tazwell substages of the Wisconsin glacial stage (34). Thicknesses of the loess are quite variable throughout northeastern Iowa. Along the Iowan drift border it is commonly 10 to 20 feet thick, but the thickness gradually decreases down to a few feet in the northeastern corner of Allamakee County.

The three soil samples used in this study, Nos. 207-1, 207-2, and 212-5, are from two northeastern Iowa counties. Samples 207-1 (A-horizon) and 207-2 (B-horizon) came from Allamakee County, and sample 212-5 (C-horizon) was obtained from Clayton County. The reasons for selecting these soil samples are twofold: the samples represent the variations in properties of the loess in northeastern Iowa, and the area is near to the mills producing spent sulphite liquors. The second reason is purely an economic one in that, if

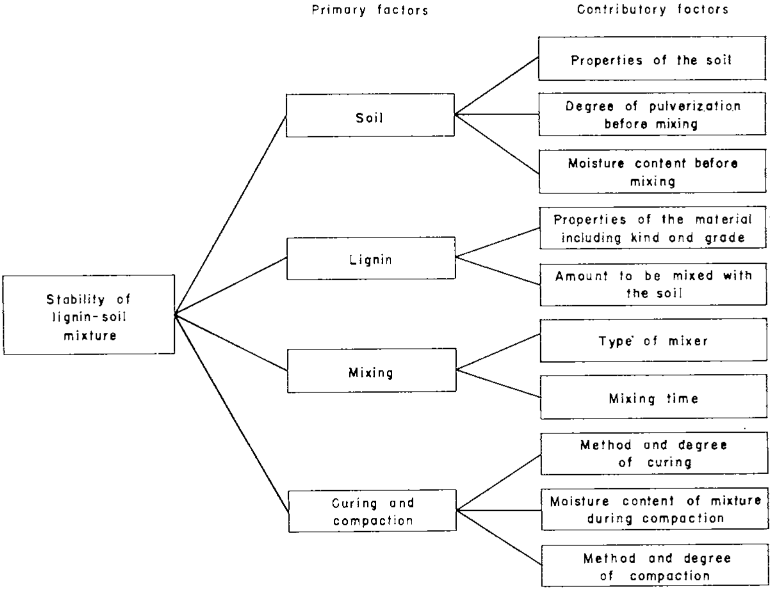

Figure 1. Variables affecting stability of lignin-soil mixture.

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[a] Hereafter, the term *soil* will be used in referring to both C-horizon and solum samples, following the common practice in engineering.

the stabilization with lignin proves successful, it would be economical to use in northeastern Iowa.

The sampling locations and a brief description of the soil samples are given in Figure 2 and Table 1; their important physical and chemical properties are summarized in Table 2.

### *Properties of Lignins Used*

Five types of lignins supplied by four different companies were used in this study. The lignins used were:

1. Spent sulphite liquor, supplied by the Kansas City Star Co., Flambeau Paper Division, Park Falls, Wisconsin.

2. and 3. Sulphite lignin grade A and sulphite lignin grade D, supplied by Lake States Yeast Corporation, Rhinelander, Wisconsin.

4. Bindarene flour, supplied by International Paper Company, New York.

5. Clarian extract, supplied by the New York and Pennsylvania Co., Inc., New York.

No standard specifications for the manufacture of lignins have

Table 1
Sampling Locations of Soil Samples

| Sample No. | County in Iowa | Section | Township and Range | Soil Series | Horizon Sampled | Sampling Depth Below Surface |
|---|---|---|---|---|---|---|
| 207-1 | Allamakee | NE¼ SE¼<br>Sec. 23 | Jefferson<br>T97N-R5W | Fayette | A | 0′-6″ |
| 207-2 | Allamakee | NE¼ SE¼<br>Sec. 23 | Jefferson<br>T97N-R5W | Fayette | B | 2′2″-2′8″ |
| 212-5 | Clayton | SE¼ SW¼<br>Sec. 27 | Lodomillo<br>T91N-R5W | Tama | C | 12′3″-12′9″ |

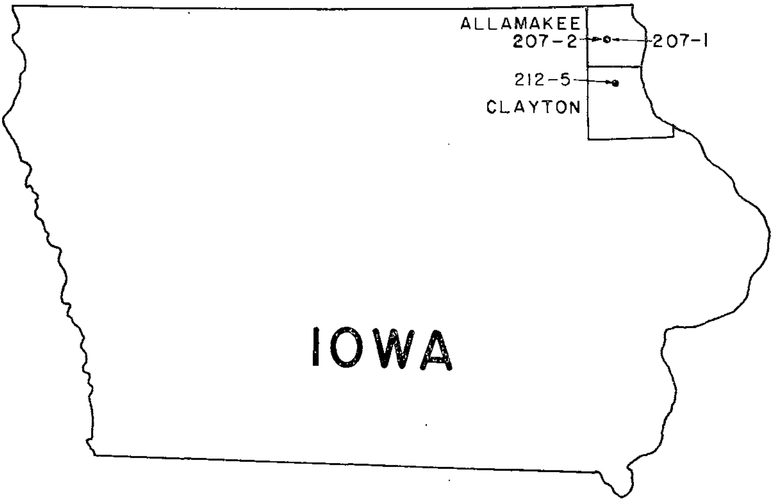

Figure 2. Sampling locations of loess and loess derived soil used in lignin stablization studies.

**Table 2**

Properties of Soil Samples

| | Sample No. | 212-5 (C-horizon) | 207-2 (B-horizon) | 207-1 (A-horizon) |
|---|---|---|---|---|
| Physical properties | L.L., % | 33.5 | 43.3 | 39.3 |
| | P.L., % | 21.5 | 24.0 | 29.0 |
| | P.I., % | 12.0 | 19.3 | 10.3 |
| | C.M.E., % | 16.9 | 23.3 | 23.2 |
| | F.M.E., % | 22.4 | 22.7 | 24.2 |
| | S.L., % | 20.4 | 16.3 | N.D. |
| Chemical properties | pH | 8.0 | 7.1 | 7.5 |
| | Chlorides, % | None | N.D. | N.D. |
| | Calcium carbonate, % | 16.6 | 6.8 | 1.3 |
| | Iron, % | 0.50 | 1.49 | N.D. |
| | Sulphate content, % | None | None | Trace |
| | Cation exchange capacity, m.e./100g. | 15.5 | 18.9 | 19.2 |
| | Organic matter, % | 0.1 | 0.36 | 4.77 |
| Textural composition,[a] % | Sand | 4.8 | 0.4 | 1.2 |
| | Silt | 71.4 | 62.9 | 70.4 |
| | Clay | 23.8 | 36.7 | 28.4 |
| | Colloidal Clay | 18.2 | 29.0 | 18.6 |
| Textural classification (B.P.R. system) | | Silty clay loam | Silty clay | Silty clay loam |
| Engineering classification | | A-6(9) | A-7-6(12) | A-6(8) |

[a] Sand—2.0 to 0.074 mm., silt—0.074 to 0.005 mm., clay—below 0.005 mm., colloidal clay—below 0.001 mm.

**Table 3**

Properties of Lignins

| Properties | Bindarene Flour[a] | Clarian Extract[b] | Sulphite Lignin Grade A[c] | Sulphite Lignin Grade D[d] | Spent Sulphite Liquor[e] |
|---|---|---|---|---|---|
| Lignin, % | 55.9 | N.D.[f] | N.D.[f] | N.D.[f] | N.D.[f] |
| Total sugars, % | 20.8 | N.D.[f] | 2.0 | 4 | N.D.[f] |
| Moisture, % | 4.0 | 50.0 | 50.0 | 6 | N.D.[f] |
| Iron, % | 0.02 | 0.25 | N.D.[f] | N.D.[f] | N.D.[f] |
| Magnesium oxide, % | 1.5 | | | | N.D.[f] |
| Calcium oxide, % | 4.9 | 6.0[g] | 4 | 7.5 | N.D.[f] |
| Sodium oxide, % | 0.3 | N.D.[f] | N.D.[f] | N.D.[f] | N.D.[f] |
| Sulfated ash, % | 17.1 | 8.0 | 5 | 9.4 | N.D.[f] |
| Sulfone $So_2$, % | 5.5 | N.D.[f] | N.D.[f] | N.D.[f] | N.D.[f] |
| Sulfur trioxide, % | 0.9 | N.D.[f] | N.D.[f] | N.D.[f] | N.D.[f] |
| Free sulfur dioxide, % | 0.2 | 1.0 | N.D.[f] | N.D.[f] | N.D.[f] |
| Total sulfur, % | 4.1 | 6.0 | N.D.[f] | N.D.[f] | N.D.[f] |
| Volatile acids, % | 3.9 | N.D.[f] | N.D.[f] | N.D.[f] | N.D.[f] |
| pH, average | 5.7 | 7.0 | 5.4 | 5.4 | N.D.[f] |
| Color | Light yellow powder | Dark brown viscous liquid | Dark brown viscous liquid | Light tan powder | N.D.[f] |
| Calcium lignosulfonate, % | 80. | N.D.[f] | 48. | 90 | N.D.[f] |
| Fe and Al as oxides, % | N.D.[f] | N.D.[f] | 0.05 | 0.10 | N.D.[f] |
| Specific gravity | N.D.[f] | 1.26 | 1.25 | N.D.[f] | |

[a] Contains 95% non-volatile material and 5% moisture; all percentages based on 100%.
[b] 50% solid and 50% moisture.
[c] 50% solid and 50% moisture.
[d] 94% solid and 6% moisture.
[e] 57% solid and 43% moisture.
[f] N.D., not determined.
[g] Magnesium oxides and calcium oxides are combined.

been developed. The information in Table 3 on the properties of the lignins used were supplied by the manufacturers.

## METHOD OF INVESTIGATION

The test methods used in this investigation to evaluate the effectiveness of the lignins as stabilizing agents for northeastern Iowa loess and loess-derived soil are of three general types: standard methods, methods in common usage but not yet standardized, and methods which have been developed and used in the soil stabilization research of the Iowa Engineering Experiment Station.

### *Preparation of Lignin-soil Mixtures*

The three soil samples used in this investigation were air-dried, pulverized, and passed through a No. 10 sieve prior to preparing the lignin-soil mixtures; the whole of each of the soils passed through this sieve. Since some of the additives were solid and some were liquid, a slightly different procedure for mixing each type of additive was followed.

The soil and powdered additives were mixed dry with a trowel; the amount of additive used being calculated on the basis of the oven-dry weight of soil. The resulting dry mixture of lignin and soil was then transferred to a mixing bowl. Mixing was done by a mechanical mixer at moderate speed. During the first minute of mixing, distilled water was added to bring the lignin-soil mixture to the optimum moisture content. The total time of mixing was five minutes.

The desired amount of liquid lignin was calculated on the basis of solids content and was diluted with enough distilled water to make the amount of liquid equal to the amount desired in the soil for compaction purposes. This additive was mixed with the soil sample as previously described.

### *Moisture-density Relationship Study*

The effect of amount and kind of lignin admixture on the optimum moisture content and maximum dry density of the soil was studied by using the compaction apparatus developed in the Iowa Engineering Experiment Station for molding 2 in. diameter by 2 in. high test specimens. This method of determining the moisture-density relationship of soils has been correlated with the standard Proctor method (ASTM Designation: D698-42T) and gives optimum moisture contents and maximum dry densities that are very close to those obtained by the standard method (7). With the soils and mixtures used in this investigation, five blows of a 5 pound hammer falling from a height of 12 in. on each end of the single layer of material being compacted in the mold was equivalent to standard Proctor

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[a] The mixer used was a Blakeslee Kitchen Mixer, Model C-20.

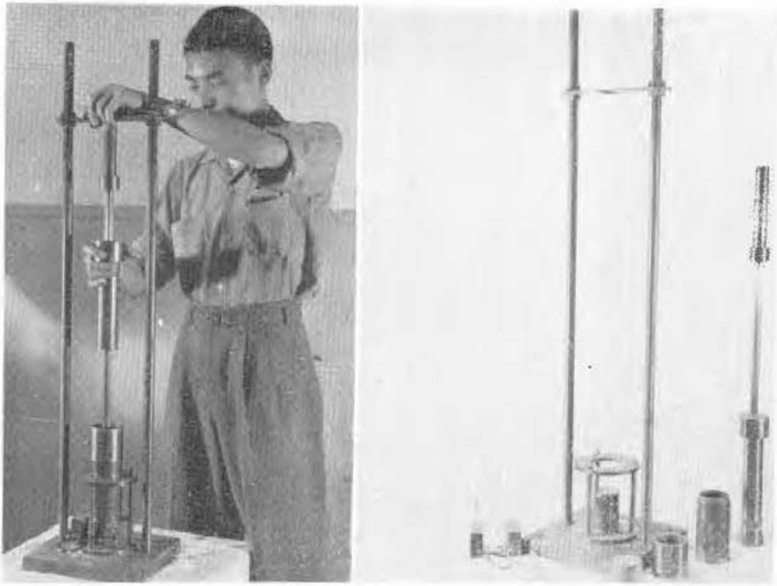

Figure 3. Apparatus for molding 2-inch diameter by 2-inch high test specimens. At left, drop hammer and molding cylinder in place. At right, drop hammer and molding cylinder detail.

compactive effort. The equipment is shown in Figure 3. The advantages of using this test method are in the great savings of time and materials as compared with the standard method.

The amounts of each lignin added to the soils were 3, 6, and 9 per cent of the dry soil weight.[a] The maximum dry density and the optimum moisture content values given in this report are the average of three determinations.

### *Unconfined Compressive Strength and Moisture Absorption Study*

The five lignins were used in varying amounts with the three soils to determine the effect of lignin admixtures on the unconfined compressive strength and capillary moisture absorption of the treated soils. Test specimens 2 in. in diameter and 2 in. high were molded, cured, and tested for unconfined compressive strength and moisture absorption by the methods described below. The amounts of each lignin added to the soils were 1, 3, 6, and 9 percent of the dry soil weight.

*Molding of specimens.* Molding of 2 in. diameter by 2 in high specimens was begun immediately after the completion of mixing. Approximately 200 grams of the soil mixture at optimum moisture

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[a] In the case of the liquid lignins, the dry solid content of the additives equaled these percentages.

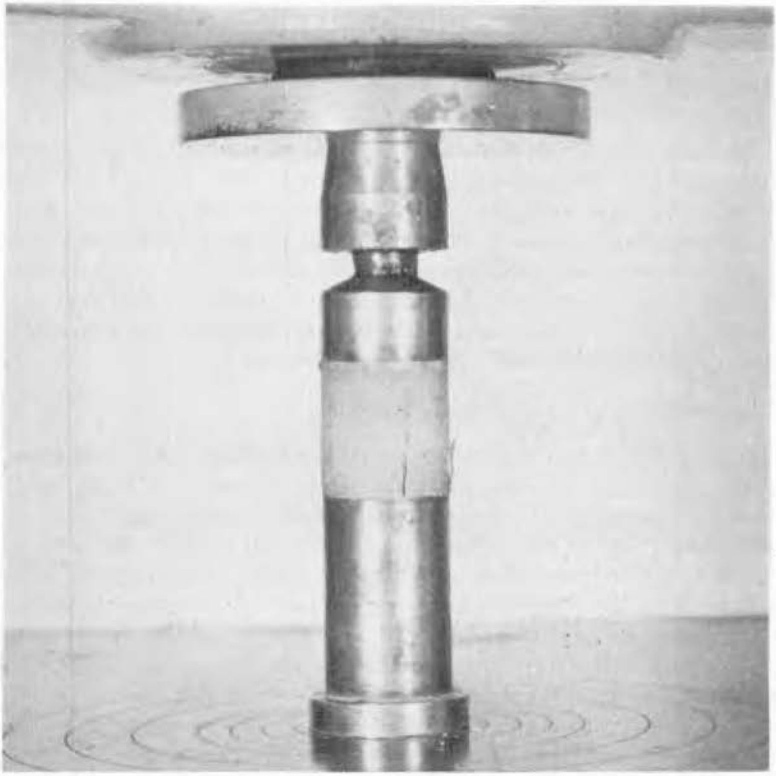

Figure 4. Two-inch diameter by 2-inch high specimen being tested for unconfined compressive strength.

content for maxjmum dry density was poured into the cylindrical mold shown in Figure 3 and was compacted as previously described. Immediately after molding, the specimens were weighed and measured to the nearest 0.1 gram and 0.001 inch, respectively.

*Curing of specimens.* The molded specimens were air-cured for 7 days. This time was chosen on the basis of a supplemental air-curing study. It was found that 2 in. by 2 in. lignin treated soil specimens reached approximately constant weight after seven days air-curing.

*Testing of specimens: After air-curing.* After seven days of air-curing, test specimens were weighed, measured, and tested for dry unconfined compressive strength. The unconfined compressive strength was determined with the apparatus shown in Figure 4. The rate of load application was 0.1 inch per minute. The maximum test load causing failure of the specimen was taken as its compressive strength. Compressive strength values in this report are the average of values for three specimens; the same is true for moisture absorption values.

*After capillary absorption.* The following procedure was used to determine the capillary moisture absorption and its effect on the compressive strength of test specimens. Both "as molded" and air-cured specimens were used in the test.

Half-inch thick felt pads were laid flat in the bottom of an air-tight cabinet. The water level in the cabinet was adjusted so that it remained just below the top of the pads. The specimens were then placed on the pads; filter paper being inserted between the specimens and the felt to prevent loss of soil particles from the bottom of the specimens. After three days, specimens that had not disintegrated were taken out of the cabinet, weighed, and tested for moisture absorption and compressive strength.

### *Consistency Limits and pH Study*

The standard ASTM tests for liquid limit, plastic limit, and plasticity index (ASTM Designations: D423-39 and D424-39) were used in experiments to determine the effect of lignin on these soil properties. The effect of lignin on the soil pH was studied with a Leeds and Northrop pH meter. Two in by 2 in. test specimens with and without lignin admixtures were molded and air-cured at room temperature for periods of 0, 1, 2, 4, and 7 days. At the end of each curing period, the specimens were broken and ground up by mortar and pestle to pass through the No. 10 sieve and then tested. Only one additive each of two Wisconsin lignins, sulphite lignin grade A (liquid) and sulphite lignin grade D (powder), were used. The percentage admixture of each lignin, selected on the basis of the results of density, compressive strength, and moisture absorption tests, was 6 percent of the dry soil weight.

### *California Bearing Ratio Study*

Two Wisconsin lignins, spent sulphite liquor and sulphite lignin grade D, were chosen for the evaluation of their effects on the C.B.R. values of the three soils; the amount of each lignin used was 6 percent of the dry soil weight. The C.B.R. test was performed essentially according to the procedure outlined by the U. S. Corps of Engineers (52). The raw soil and lignin treated specimens were compacted to standard Proctor density, and C.B.R. values for "as molded" and "soaked 4 days" were determined. The swelling and moisture absorption of the soaked samples were also determined.

### *Supplementary Curing Study Using Iowa Bearing Value Test*

A supplementary curing study using the Iowa Bearing Value test was made on a mixture of soil 212-5 (C-horizon) and 6 percent spent sulphite liquor. Test procedures followed were similar to those prescribed by Chu and Davidson (8). The apparatus used in the test is

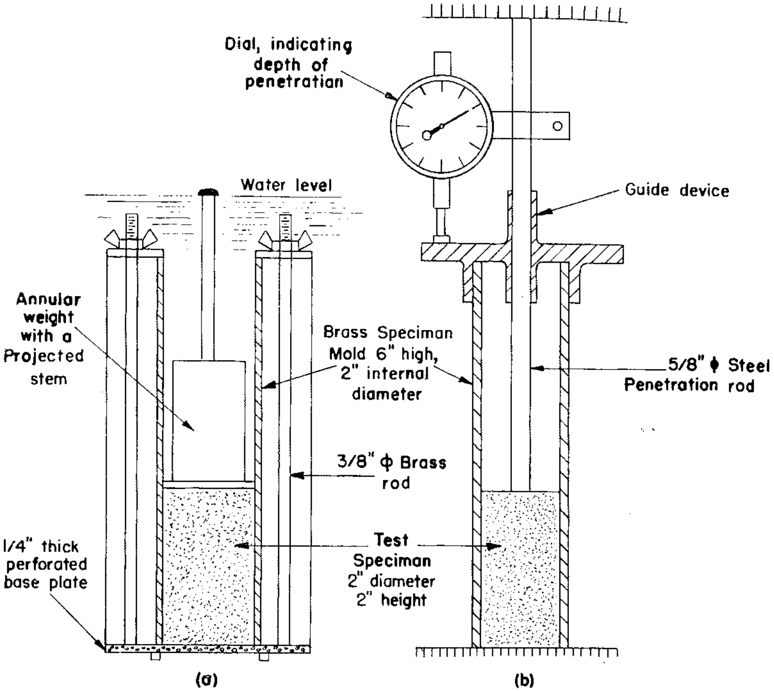

Figure 5: The Iowa Bearing Value Test Apparatus
(a) Immersion of a test specimen in water bath.
(b) Speciman in position for testing.

shown in figure 5. Specimens 2 in. in diameter and 2 in. high were molded at optimum moisture content to near standard Proctor density. The molding apparatus is shown in figure 3, except that specimens were molded in a brass cylinder having an internal diameter of 2 in. and a height of 6 in. The specimens were also cured and tested in the cylinders in which they were molded.

Specimens were cured by two different methods. In the first, specimens were air-cured at room temperature for a period ranging from 1 to 28 days. At the end of each curing period, four specimens were weighed. Two of them were tested immediately, and the other two were immersed in water for 7 days before being tested. Before immersion, the molds were clamped to a frame (figure 5a). An annular weight, weighing 1.1 lbs. in water, which has a stem projecting above the brass specimen mold for supporting an Ames dial to measure swelling and a perforated disc at the bottom for the passage of water through it, was placed on the top of the specimens. The water level was kept as shown in figure 5a. At the end of seven days immersion, the specimens in their brass cylinders were taken out of water, drained, weighed, and tested. The testing procedure was the same for both air-cured and soaked specimens. Figure 5b shows the testing apparatus and illustrates the method of testing. The rate of penetration was 0.05 inch per minute. The test load in pounds at 0.2 inch penetration was taken as the strength value of the specimens. Moisture absorption and swell during immersion were also determined. All values derived from this test are the average of at least two specimens.

Specimens were moist-cured using the other method for a period ranging from one to twenty-eight days at a relative humidity of 90 to 95 percent and a temperature of about 70° F. Testing of specimens with and without immersion, after each moist curing period was the same as in the first method.

The objectives of this study were to determine the effect of method and amount of curing on the bearing capacity of lignin treated soil. The Iowa Bearing Value test was used mainly because the large number of specimen molds required for the study and the moist cabinet space available, ruled out the use of the C.B.R. test. Other advantages of the IBV are the saving of time and materials. Studies in progress in the Iowa Engineering Experiment Station indicate that with fine-grained soils, the results of the IBV can be correlated with those of the C.B.R. test.

## PRESENTATION AND EVALUATION OF TEST RESULTS

### *Effect of Lignin on Moisture-Density Relationship*

Work done in the past has shown that the compacted density of a soil is increased by additions of lignin (26). This is a desirable property change, since an increase in density is usually an indication of an increase in strength.

The test results indicate clearly that the use of lignin has a bene-

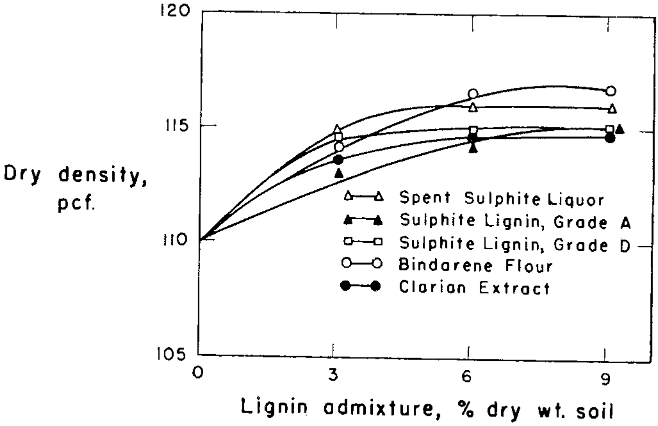

Figure 6. Variation of maximum dry density of lignin-treated C-horizon loess (soil sample 212-5) with the amount and kind of lignin.

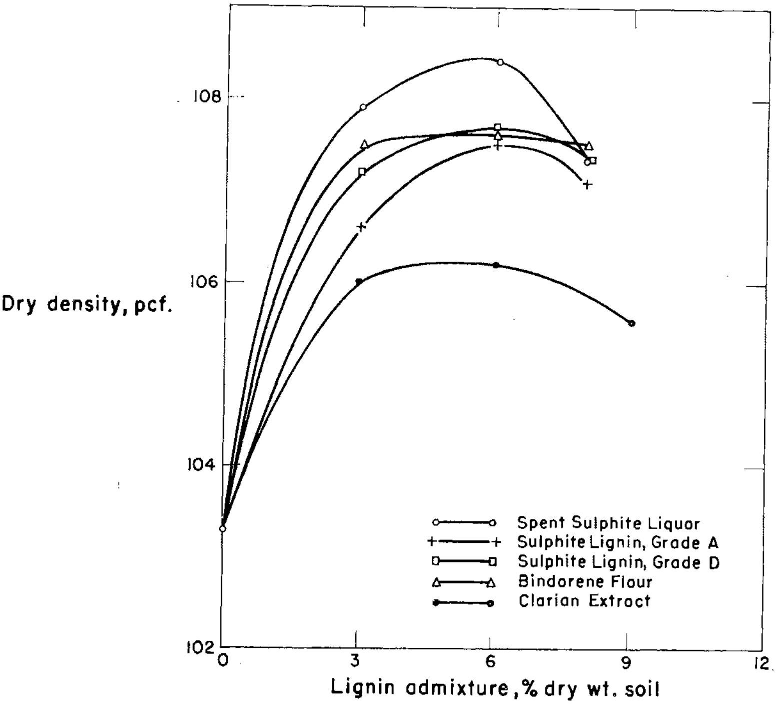

Figure 7. Variation of maximum dry density of lignin-treated B-horizon loess (soil sample 207-2) with the amount and kind of lignin.

ficial effect on the compacted density of soil, though no correction has been made in the dry density values for the amount of lignin present in the soil. From the curves shown in Figures 6, 7, and 8, it appears that the optimum amount of lignin to produce maximum dry density for the compactive effort used lies somewhat between 6 to 9 percent of the dry soil weight. In most cases there was no significant increase in density above 6 percent. Though it is difficult to single out the lignin having the best effect on density, it appears that spent sulphite liquor and bindarene flour may give a slightly higher density.

From Figures 9, 10, and 11, which illustrate the effect of the kind and amount of lignin on the optimum moisture content of the soils, it is shown that all the additives reduce the optimum moisture content; this reduction being of significance when the amount of lignin used was between 6 and 9 percent.

### *Effect of Lignin on Unconfined Compressive Strength and Moisture Absorption*

The test results of the unconfined compressive strength study indicate that the 7-day air-dried strength of the B- and C-horizon soils

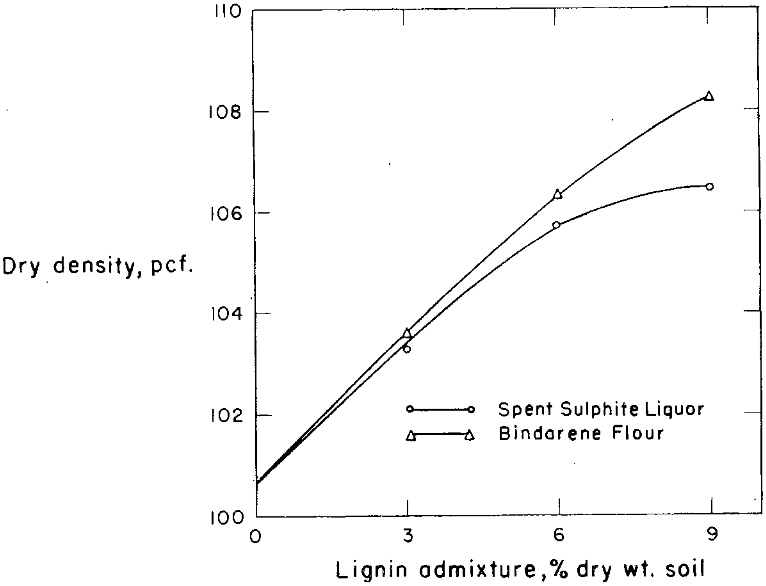

Figure 8. Variation of maximum dry density of lignin-treated A-horizon loess (soil sample 207-1) with the amount and kind of lignin.

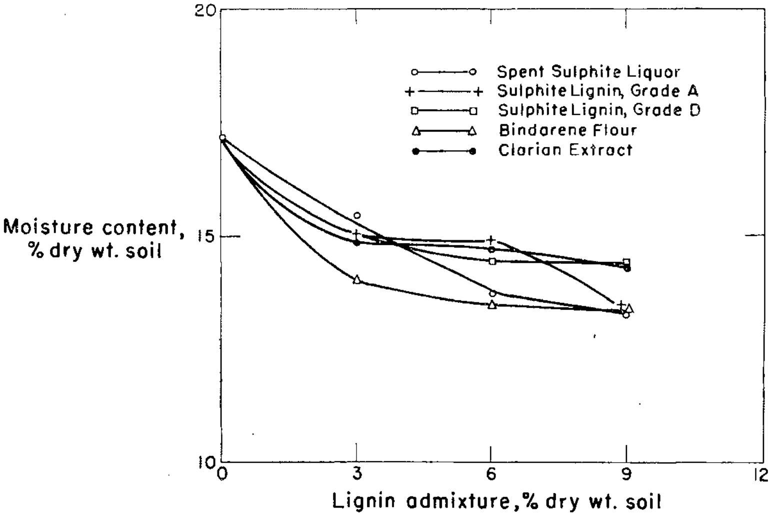

Figure 9. Variation of optimum moisture content of lignin-treated C-horizon loess (soil sample 212-5) with the amount and kind of lignin.

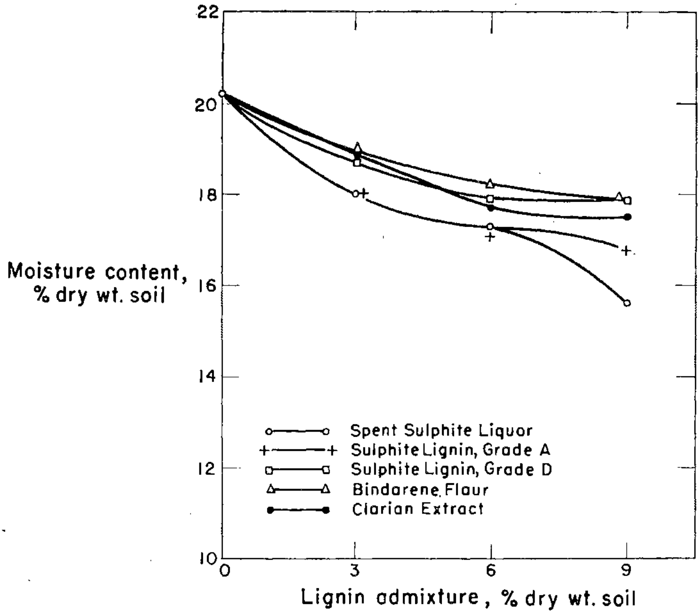

Figure 10. Variation of optimum moisture content of lignin-treated B-horizon loess (soil sample 207-2) with the amount and kind of lignin.

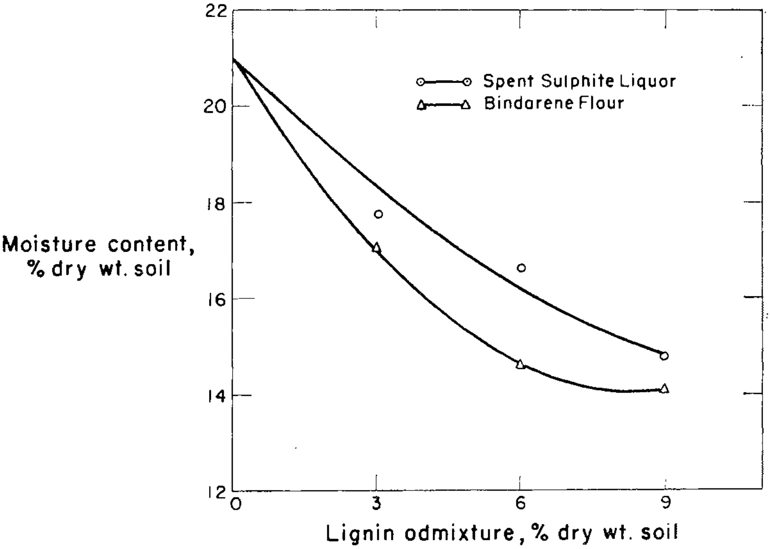

Figure 11. Variation of optimum moisture content of lignin-treated A-horizon loess (soil sample 207-1) with the amount and kind of lignin.

were decreased by the addition of lignins. This trend was not found with the A-horizon soil, 207-1, whose strength was a maximum with about 1 percent of lignin additive. The reason for the decrease in compressive strength seems to be that the lignin treated soil retains more moisture than the raw soil. Comparisons of the effect of the different lignins on the air-dried compressive strength of the three soils are shown in Figures 12, 13, and 14. It is difficult to rate the lignins on the basis of this data, but since it is desirable to have as little reduction of strength as possible, bindarene flour and sulphite lignin grade D might be rated slightly higher than the others.

The effect of the amount of additive on moisture retention after seven days air drying can be seen from the test data in Figures 15, 16, and 17. An increase in the amount of lignin additive increased the moisture retention capacity of the B- and C-horizon soils. The different effect of the lignins on the A-horizon soil is undoubtedly related to the organic matter content, and no attempt at further explanation will be made.

When test specimens were air-dried for seven days and then subjected to capillary absorption of moisture for three days prior to testing for unconfined compressive strength, a different trend in the compressive strength data was obtained.

The strength data are plotted in Figures 18, 19, and 20. Specimens containing no lignin additive fell apart during the capillary

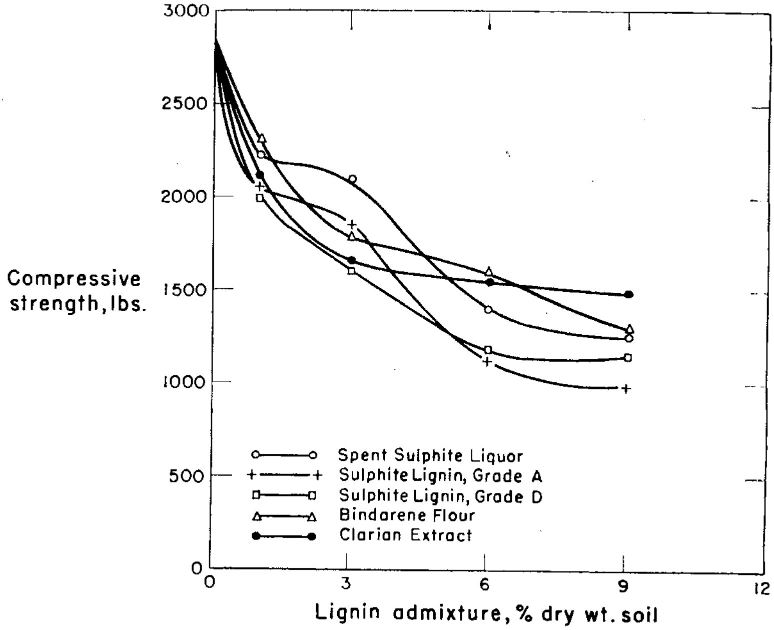

Figure 12. Variation of 7-day air-dried unconfined compressive strength of C-horizon loess (soil sample 212-5) test specimens with amount and kind of lignin admixture.

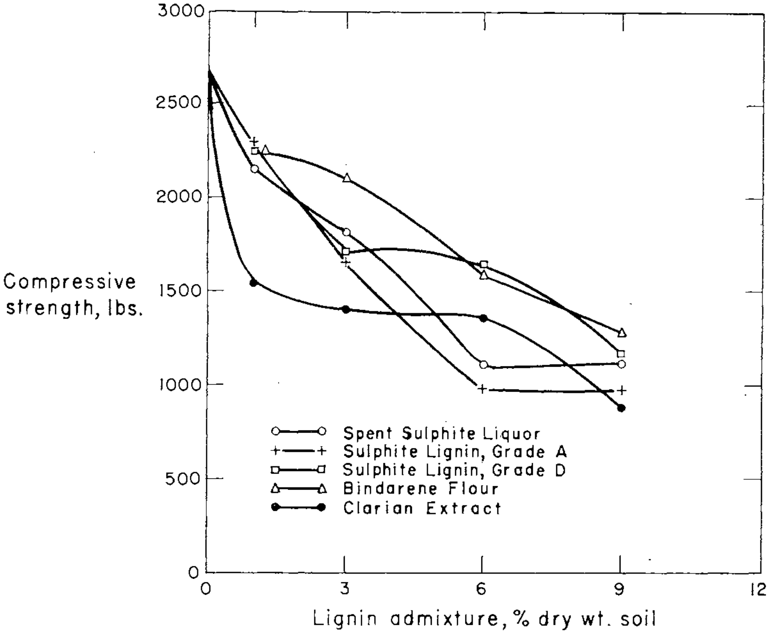

Figure 13. Variation of 7-day air-dried unconfined compressive strength of B-horizon loess (soil sample 207-2) test specimens with amount and kind of lignin admixture.

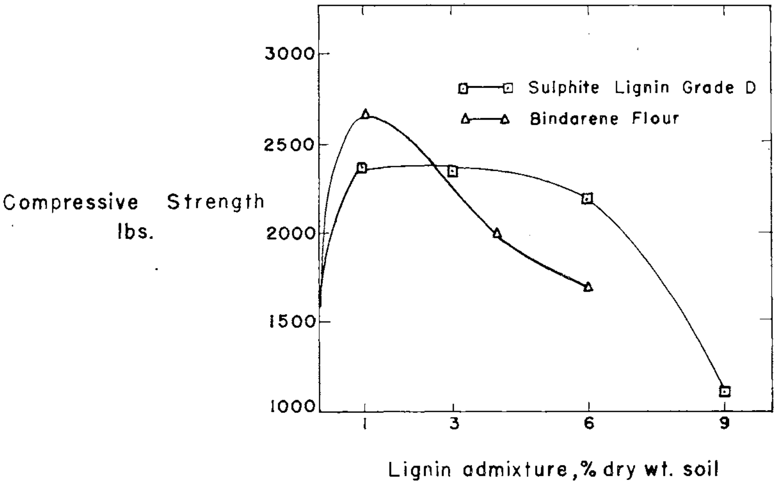

Figure 14. Variation of 7-day air-dried unconfined compressive strength of A-horizon loess (soil sample 207-1) test specimens with amount and kind of lignin admixture.

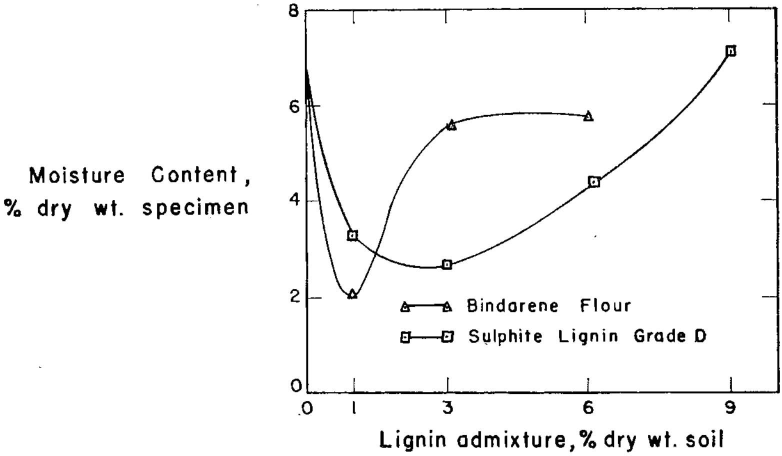

Figure 15. Effect of amount and kind of lignin admixture on moisture retention of C-horizon loess (soil sample 212-5) test specimens after 7-day air-drying.

absorption period. Though the strength of the lignin-treated specimens was greatly reduced by capillary absorption, all remained intact and could be tested. Best strength results were obtained when the amount of additive was in the range of 6 to 9 percent; in most cases, about 6 percent was the optimum amount. Inspection of the moisture absorption data in figures 21, 22, and 23 shows that moisture absorption was also near the minimum amount when the lignin admixture was about 6 percent. The correlation of specimen density with strength and moisture absorption is not as good as the correlation between strength and moisture absorption but there is an indication that some of the waterproofing and strength preservation is related to the beneficial effect of lignin on soil density.

The data presented is not sufficient for rating the lignins as waterproofers, but it is of interest to note that the difference in results was least with the soil having the highest clay content, the B-horizon loess; all the lignins gave this soil about the same degree of waterproofness. With the C-horizon loess, sulphite lignin grade A, and clarian extract gave the best results. Only sulphite lignin grade D and bindarene flour were evaluated with the A-horizon loess, and there was only a slight difference in test results.

### *Effect of Lignin on L.L., P.L., P.I., and pH*

The test data presented in Table 4 definitely indicate that 6 percent admixture of sulphite lignin grade A and sulphite lignin grade D increased the plasticity indices of the C-horizon and B-horizon loess. It is apparent that the increase is mainly due to the lowering of the plastic limit, although there is some indication that the liquid limit increased slightly with air curing. The increase in plasticity

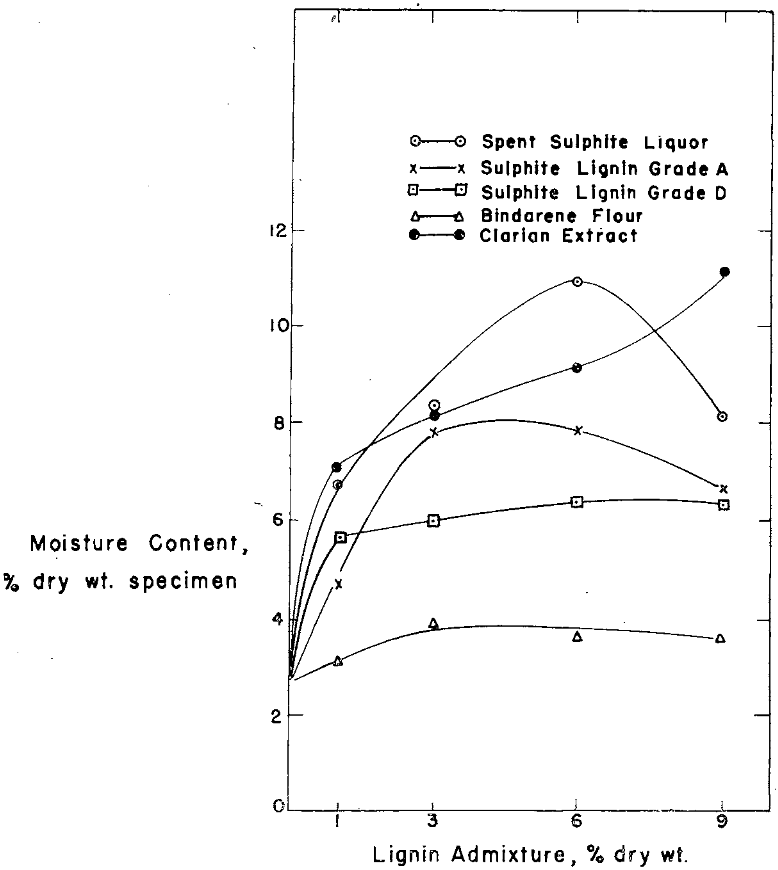

Figure 16. Effect of amount and kind of lignin admixture on moisture retention of B-horizon loess (soil sample 207-2) test specimens after 7-day air-drying.

is probably related to the fact that lignin is a dispersing agent and when incorporated in soil, increases the effective surface area, especially that of the clay-size fraction. In general, the air-curing data is too erratic for definite conclusions concerning the effect of length of curing time on the consistency limits.

The effect of curing time on pH is also shown in Table 4. The pH of the raw soil was not significantly affected; that of the lignin-treated soils showed a slight increase at 1-day curing time, but thereafter, remained more or less constant.

### *Effect of Lignin Admixtures on C.B.R.*

The California Bearing Ratios of the three soils were decreased by the 6 percent admixtures of sulphite lignin grade D, and spent sulphite liquor. As shown in Table 5, this was generally true for both

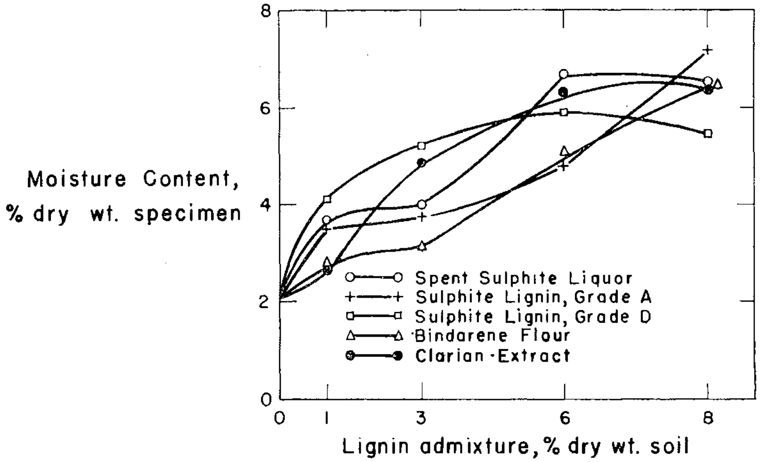

Figure 17. Effect of amount and kind of lignin admixture on moisture rentention of A-horizon loess (soil sample 207-1) test specimens after 7-day air-drying.

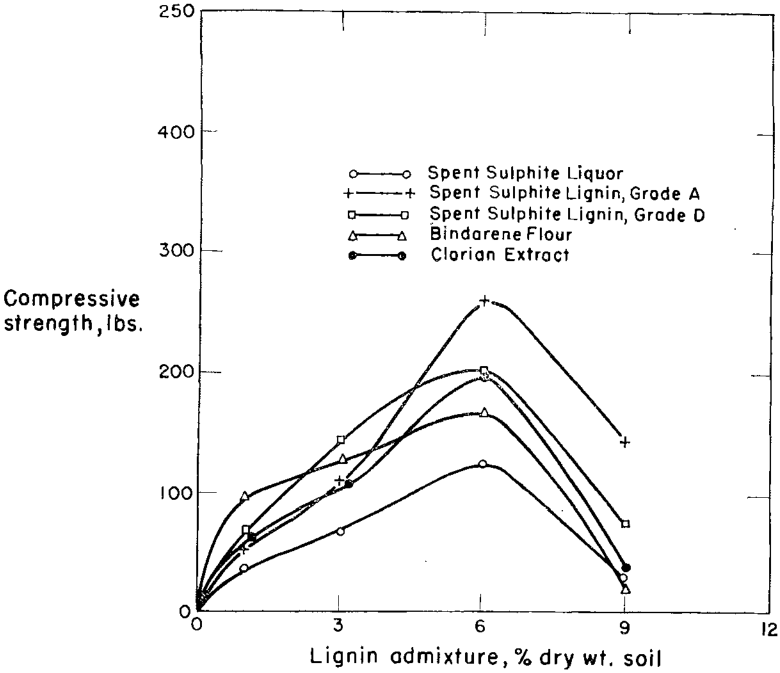

Figure 18. Variation of 7-day air-dried plus 3-day capillary moisture absorption unconfined compressive strength of C-horizon loess (soil sample 212-5) test specimens with amount and kind of lignin admixtures.

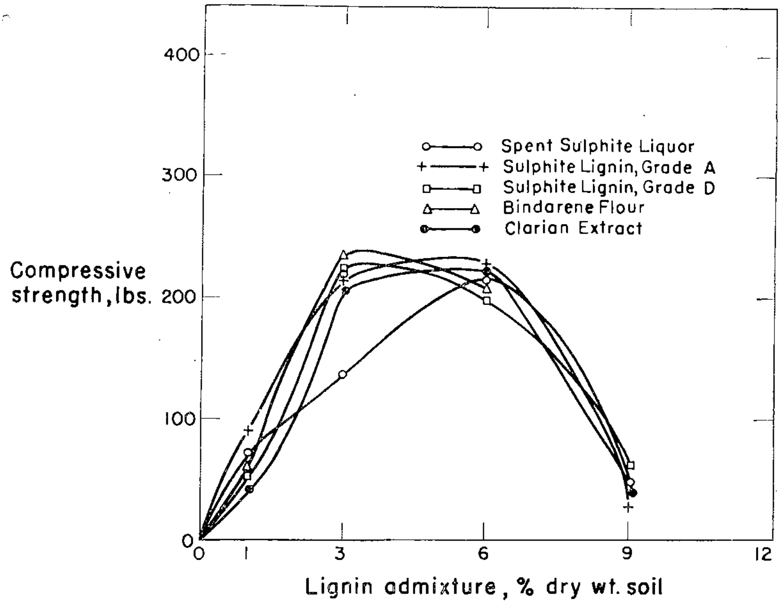

Figure 19. Variation of 7-day air-dried plus 3-day capillary moisture absorption unconfined compressive strength of B-horizon loess (soil sample 207-2) test specimens with amount and kind of lignin admixtures.

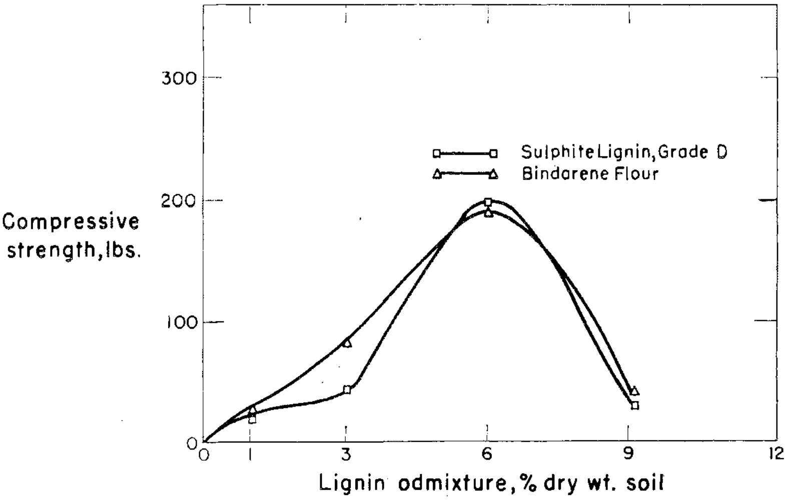

Figure 20. Variation of 7-day air-dried plus 3-day capillary moisture absorption unconfined compressive strength of A-horizon loess (soil sample 207-1) test specimens with amount and kind of lignin admixtures.

Table 4
Effect of Lignin Admixtures on Consistency Limits of C-Horizon and B-Horizon Loess[a]

| Types of Mixture | Curing Period, days | Liquid Limit, % | Plastic Limit, % | Plasticity Index | pH |
|---|---|---|---|---|---|
| Raw soil 212-5 (C-hor.) | 0 | 34.7 | 20.3 | 14.4 | 7.5 |
| | 2 | 33.4 | 20.3 | 13.1 | 7.4 |
| | 7 | 33.8 | 20.4 | 13.4 | 7.4 |
| Soil stabilized with 6% sulphite lignin grade A | 0 | 33.4 | 14.4 | 19.0 | 6.90 |
| | 1 | 32.6 | 14.8 | 17.8 | 7.4 |
| | 2 | 36.7 | 15.2 | 21.5 | 7.48 |
| | 4 | 35.4 | 14.9 | 20.5 | |
| | 7 | 36.7 | 16.5 | 20.2 | |
| Soil stabilized with 6% sulphite lignin grade D | 0 | 33.5 | 14.9 | 18.6 | 6.85 |
| | 1 | 32.5 | 14.7 | 17.8 | 7.50 |
| | 2 | 37.0 | 19.5 | 18.5 | 7.46 |
| | 4 | 36.5 | 17.9 | 18.6 | 7.62 |
| | 7 | N.D. | N.D. | N.D. | 7.60 |
| Raw soil 207-2 (B-hor.) | 0 | 47.2 | 22.3 | 24.9 | 6.95 |
| | 2 | 46.3 | 20.4 | 25.9 | 6.94 |
| | 7 | 46.2 | 19.8 | 26.5 | 6.93 |
| Soil stabilized with 6% sulphite lignin grade A | 0 | 46.0 | 16.1 | 29.9 | 4.71 |
| | 1 | 56.8 | 18.4 | 38.4 | 5.01 |
| | 2 | 53.8 | 19.0 | 34.8 | 5.08 |
| | 4 | 50.0 | 19.9 | 30.1 | 5.10 |
| | 7 | 44.6 | 20.1 | 24.5 | 5.18 |
| Soil stabilized with 6% sulphite lignin grade D | 0 | 47.9 | 18.6 | 29.3 | 4.56 |
| | 1 | 56.9 | 18.4 | 38.5 | 5.05 |
| | 2 | 52.0 | 19.0 | 33.0 | 4.93 |
| | 4 | 49.6 | 21.2 | 28.4 | 5.08 |
| | 7 | 47.0 | 19.2 | 28.8 | 5.10 |

[a] Two inch diameter by 2 inch high specimens were molded, air-dried for the period indicated, then crushed to pass the No. 10 sieve prior to performing tests.

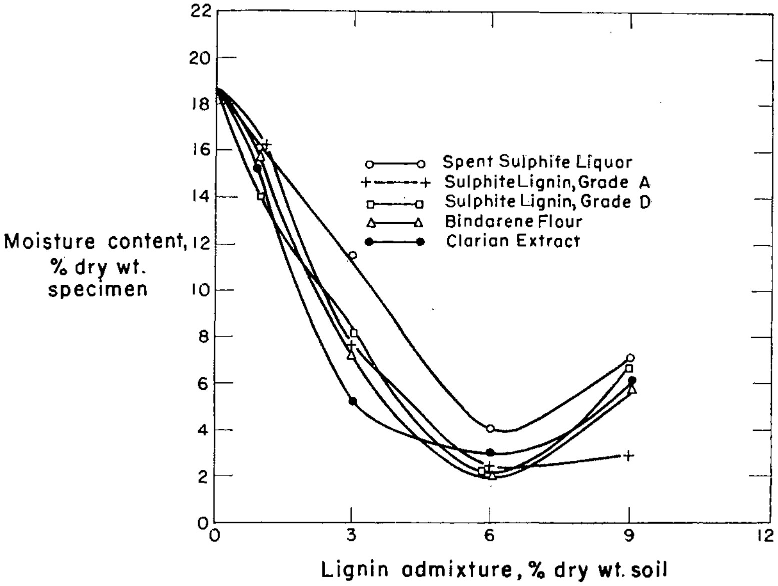

Figure 21. Effect of amount and kind of lignin admixture on moisture content of 7-day air-dried C-horizon loess (soil sample 212-5) test specimens subjected to 3 days capillary moisture absorption.

the "as molded" and "soaked 4 days" test condition. Swelling and moisture absorption were increased by the lignin admixtures. The soils highest in clay and organic matter, the B- and A-horizon soils, were most adversely affected by the admixtures (Table 5). The adverse effect of lignin on the bearing capacity of the soils, as measured by the C.B.R. test procedure used, may be due to:

1. The dispersing effect of lignin on soil,

2. The leaching out of the lignin during four days complete immersion, and

3. The lack of proper curing of the lignin treated soils prior to the C.B.R. test.

### *Effect of Method of Curing on Bearing Strength*

The Iowa Bearing Value test study of the effect of moist and air curing on the bearing strength and related properties of lignin-treated soils was an extension of the C.B.R. study, since in the C.B.R. study the method of curing was not a variable. The reason for using the IBV test method instead of the C.B.R. has been discussed. Test results of the study are presented in Tables 6 and 7 and in Figure 24.

The data indicate that the strength of air-cured specimens increased with increase of curing time. The effect of curing time is

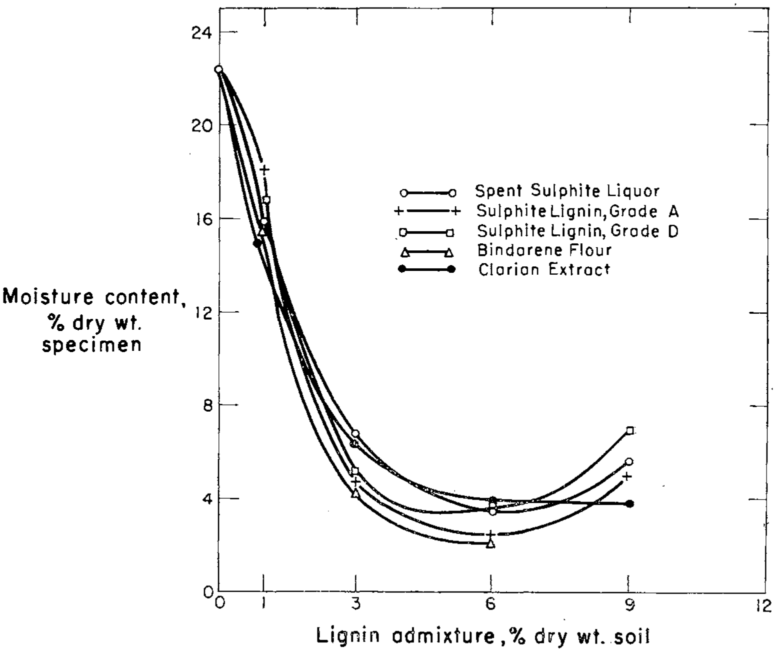

Figure 22. Effect of amount and kind of lignin admixture on moisture content of 7-day air-dried B-horizon loess (soil sample 207-2) test specimens subjected to 3 days capillary moisture absorption.

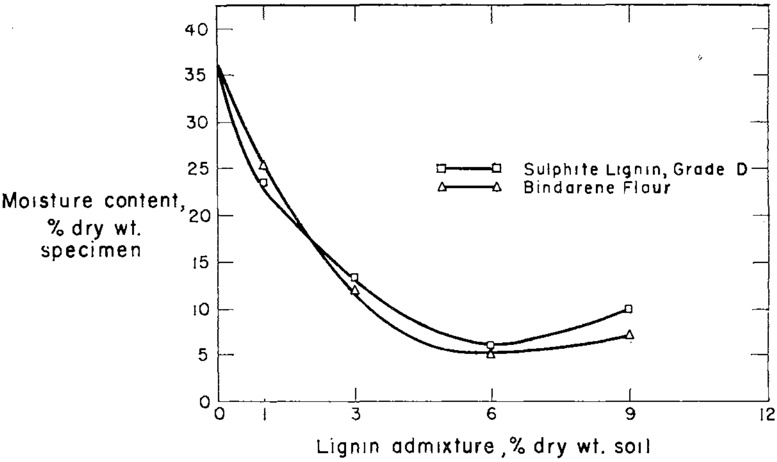

Figure 23. Effect of amount and kind of lignin admixture on moisture content of 7-day air-dried A-horizon loess (soil sample 207-1) test specimens subjected to 3 days capillary moisture absorption.

Table 5

Effect of Lignin Admixtures on C.B.R. and Related Properties of A, B, and C-Horizon Loess

| | As Molded | Soaked 4 Days | | |
|---|---|---|---|---|
| Mixture | C.B.R. 0.1 in. penetration | C.B.R. 0.1 in. penetration | Swell, % | Absorption, % |
| Raw soil 212-5 (C-hor.) | 17.8 | 7.7 | 0.36 | N.D. |
| Soil 212-5 + 6% sulphite lignin grade D | 14.2 | 7.9 | 0.34 | 3.2 |
| Soil 212-5 + 6% spent sulphite liquor | 14.1 | 4.4 | 0.52 | 5.1 |
| Raw soil 207-2 (B-hor.) | 11.4 | 9.0 | 0.52 | 1.9 |
| Soil 207-2 + 6% sulphite lignin grade D | 7.5 | 0.7 | 2.60 | 10.9 |
| Soil 207-2 + 6% spent sulphite liquor | 7.8 | 1.4 | 2.18 | 8.8 |
| Raw soil 207-1 (A-hor.) | 15.0 | 7.1 | 0.56 | 1.8 |
| Soil 207-1 + 6% spent sulphite liquor | 10.7 | 2.2 | 1.32 | 12.1 |

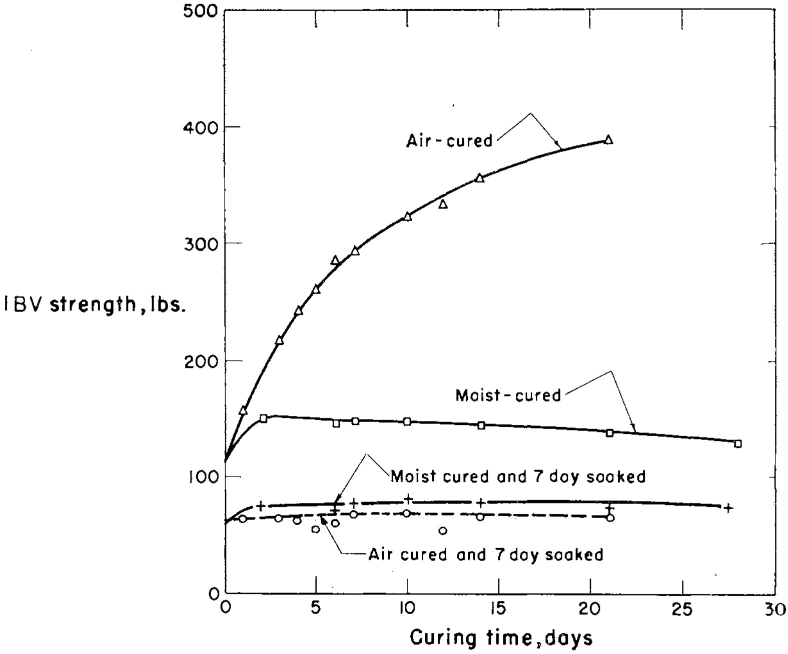

Figure 24. Comparison of air-curing and moist-curing on the bearing strength (IBV) of C-horizon loess (soil sample 212-5) treated with 6 percent spent sulphite liquor.

not very pronounced in the data on moist-cured specimens; they gained some strength during the first seven days of moist curing, and then gradually lost some of the strength gain throughout the remainder of the 28-day curing period. Without the 7-day immersion treatment, the strengths of moist-cured specimens were much lower than those of air-cured specimens.

After seven days of immersion in water, the strengths of both air-cured and moist-cured specimens were low; length of curing prior to immersion had little effect on the immersed strength. Moist-cured specimens showed less decrease of strength and had slightly higher strength after immersion than did air-cured specimens. Also, moist-cured specimens absorbed less moisture than air-cured specimens. The expansion data is too erratic for making comparisons.

## CONCLUSIONS

The following conclusions are based on the results of the previously described experiments with five kinds of lignins and three northeastern Iowa fine-grained soils:

1. Lignins used alone as admixtures do not show much promise as stabilizing agents for loess or loess-derived soils. The findings of the

Table 6

Iowa Bearing Value Test Data for Air-Cured Specimens of Soil 212-5 (C-Hor.) Treated with 6 Percent Spent Sulphite Liquor

| No. of Days Air-Cured[a] | No. of Days Immersed | Specimen Dry Density,[b] pcf | Absorption,[c] % | Expansion,[d] % | IBV Strength, lbs. at 0.2 in. penetration |
|---|---|---|---|---|---|
| 0 | 0 | 113.4 | — | — | 111 |
| 0 | 7 | 113.0 | N.D. | N.D. | 62 |
| 1 | 0 | 115.0 | — | — | 157 |
| 1 | 7 | 115.0 | 3.99 | 0.25 | 65 |
| 3 | 0 | 113.6 | — | — | 215 |
| 3 | 7 | 114.3 | 6.36 | 0.08 | 63 |
| 4 | 0 | 115.8 | — | — | 242 |
| 4 | 7 | 116.2 | 6.12 | 0.15 | 61 |
| 5 | 0 | 115.2 | — | — | 260 |
| 5 | 7 | 115.2 | 6.53 | — | 55 |
| 6 | 0 | 115.4 | — | — | 285 |
| 6 | 7 | 116.3 | 6.44 | 0.64 | 60 |
| 7 | 0 | 113.4 | — | — | 290 |
| 7 | 7 | 114.4 | 6.8 | 0.3 | 67 |
| 10 | 0 | 114.8 | — | — | 323 |
| 10 | 7 | 114.6 | 8.83 | 0.87 | 67 |
| 12 | 0 | 114.9 | — | — | 333 |
| 12 | 7 | 115.1 | 10.57 | 0.35 | 48 |
| 14 | 0 | 114.5 | — | — | 358 |
| 14 | 7 | 115.0 | 8.9 | 0.85 | 66 |
| 21 | 0 | 114.5 | — | — | 407 |
| 21 | 7 | 113.7 | 6.7 | 0.77 | 71 |

[a] Curing was at room temperature which averaged about 77° F.

[b] No correction was made in the density for the amount of lignin present in the specimens.

[c] Absorption is the amount of water absorbed by the specimens, expressed as percent dry weight of specimens, after 7 days complete immersion.

[d] Expansion is the percent increase of the specimens' height over its original height after 7 days complete immersion.

Table 7

Iowa Bearing Value Test Data for Moist-Cured Specimens of Soil 212-5 (C-Hor.) Treated with 6 Percent Spent Sulphite Liquor

| No. of Days Moist-Cured[a] | No. of Days Immersed | Specimen Dry Density,[b] pcf | Absorption,[c] % | Expansion,[d] % | IBV Strength, lbs. at 0.2 in. penetration |
|---|---|---|---|---|---|
| 0 | 0 | 113.4 | — | — | 111 |
| 0 | 7 | 113.5 | 2.57 | 0.75 | 58 |
| 2 | 0 | 114.5 | — | — | 152 |
| 2 | 7 | 114.0 | 2.54 | 0.70 | 76 |
| 6 | 0 | 115.5 | — | — | 145 |
| 6 | 7 | 114.8 | 2.78 | 0.62 | 65 |
| 7 | 0 | 114.8 | — | — | 148 |
| 7 | 7 | 115.5 | 5.11 | 0.35 | 76 |
| 10 | 0 | 114.2 | — | — | 147 |
| 10 | 7 | 115.0 | 1.33 | 0.48 | 79 |
| 14 | 0 | 116.2 | — | — | 142 |
| 14 | 7 | 113.6 | 3.79 | 0.55 | 77 |
| 21 | 0 | 115.8 | — | — | 137 |
| 21 | 7 | 115.6 | 1.81 | N.D. | 72 |
| 28 | 0 | 115.0 | — | — | 133 |
| 28 | 7 | 116.0 | 0.86 | 0.35 | 75 |

[a] Relative humidity was 90 to 95% and temperature about 70° F.

[b] Density was determined at optimum moisture content. No correction was made for the amount of lignin present in the soil sample.

[c] Absorption is the amount of water absorbed by the specimens, expressed as percent dry weight of specimens, after 7 days complete immersion.

[d] Expansion is the percent increase of the specimens' height over its original height after 7 days complete immersion.

investigation do, however, indicate that lignins should be much more effective as stabilizing agents for granular soils or soil-aggregate mixtures. This should be verified.

2. Lignin admixtures to soil do improve some engineering properties related to stability; they increase compacted density and retard absorption of moisture, though the latter benefit may be temporary if leaching out of the lignin occurs. The effect of the lignins on frost action was not investigated.

3. Lignin admixtures increase the moisture retention capacity of soil. Because of this, air-dried strength of lignin-treated soil may be lower than that of the raw soil dried for the same length of time. The strength of lignin-treated soil increases rapidly with increase in length of air curing, and elevated temperature drying would probably be beneficial.

4. Moist curing of lignin-treated soil specimens results in much lower strength than air curing, but moist-cured specimens absorb less moisture and have slightly higher strength after immersion in water. The length of moist curing does not seem to have much effect on moist-cured or immersed strength.

5. Lignin admixtures increase the plasticity index of soil; the amount of increase is greatest for soils having the highest clay and organic matter contents. Lignins do not appreciably change the soil pH value.

6. Lignin in powdered form was easier to use than lignin in liquid form; otherwise, the five lignins used gave much the same results.

7. There is an indication that the optimum amount of lignin admixture (dry solids basis) is about 6 percent of the soil dry weight.

## ACKNOWLEDGMENTS

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