Soil-Organic Cationic Chemical-Lignin Stabilization

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This paper presents the results of a series of tests performed on four Iowa soils stabilized with two organic cationic chemicals in combination with lignin.

The chemicals used were a quaternary ammonium chloride and a fatty amine acetate known commercially as Arquad 2HT and Armac T, respectively. The lignins used were either commercially available or in pilot plant production and contained varying sugar contents. The four soils ranged from 10 to 74 percent in clay content.

Preliminary studies of the chemicals and lignins on one of the soils, a friable calcareous loess from southwestern Iowa, indicated the most desirable mixing chronology to be the addition of chemical solution first, followed by the addition of the lignin solution. The most beneficial lignin for each organic cationic chemical was also chosen and was used throughout the remainder of testing.

In general, the use of the soil-organic cationic chemical-lignin combination indicates increased compressive strength of the soil with reduction of moisture absorption and expansion during complete immersion in water, these benefits being somewhat greater with the combination of soil, chemical and lignin than with the soil and chemical only.

STUDIES OF the use of organic cationic chemicals for soil stabilization at the Iowa Engineering Experiment Station date back to 1947. Results with several Iowa soils and soil mixtures have been reported (4,5,6,9,10,11) and indicate that this method of soil stabilization has considerable promise. Two of the many organic cationic chemicals evaluated appear to be the most promising. Known commercially as Arquad 2HT and Armac T, they are, respectively, a quaternary ammonium chloride and a fatty amine acetate.

It has been shown that organic cationic chemicals act primarily as waterproofing agents, which tend to maintain the bearing capacity of soil even under adverse moisture conditions. Investigations by Nicholls (12) have shown that the addition of lignin to a soil-organic cationic chemical mixture may further improve waterproofing and strength characteristics. Lignin is a waste product of the paper industry and is available in large quantities. The cost is primarily dependent on transportation distance. The addition of lignin to soil-organic cation mixtures would not appreciably raise the cost.

The purpose of this investigation was to further evaluate the use of

lignin in soil-organic cation mixtures. The criteria used in evaluating the effectiveness of stabilization were the air-dry and immersed unconfined compressive strengths, and the moisture absorption and expansion after immersion.

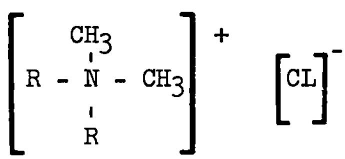
MATERIALS

Soils

The soil samples used represent important soil types found in Iowa and also provide a wide range of clay content for study of this variable. The dominant clay mineral in the samples is montmorillonite. Descriptions and properties of the soils are given in Tables 1 and 2. Due to a lack of clay size particles in the dune sand, the plastic loess was blended and mixed with the sand to produce a soil material having a clay content of about 10 percent. This dune sand-plastic loess mixture (hereafter referred to as the "sand-loess mixture") was prepared in order to provide a means for the cation exchange process to occur.

Chemicals

Arquad 2HT (2,3) is a di-hydrogenated tallow di-menthyl ammonium chloride with the following general structural formula:



in which "R" represents a fatty acid derivative. Arquad 2HT will retain its surface activity at either high or low pH and is not precipitated by calcium or magnesium in water solutions. It is easily dispersible in water up to about 8 percent by weight and is normally supplied by the manufacturer as 75 percent active in isopropanol. It has an average molecular weight of about 585. This chemical is also commercially available under the trade name Aliquat H226 (8). Concentrations referred to in this paper were calculated from the active chemical content of the supplied material.

Armac T (1,2) is a water dispersible primary amine acetate derived from tallow fatty acids. The general structural formula is $(RNH_3)^+Ac^-$. Armac T has an average molecular weight of about 310 and is marketed as 100 percent active material.

Preparation of Chemicals. Dispersions of Arquad 2HT and Armac T in water were prepared by dispersing each chemical in distilled water previously heated to 60 C. After thorough mixing the dispersions were allowed to cool to room temperature. The concentrations of the dispersions were adjusted to provide the desired mix water for compaction and the desired amounts of chemical in the soil. Treatments varied from 0.1 to 0.9 percent chemical by dry soil weight.

Lignins

The lignins used are labeled A through E and were obtained from several Wisconsin producers. Physical and chemical properties are given in Table 3.

Preparation of Lignins. Solutions of the lignins were prepared by dissolving each lignin in distilled water at room temperature. The con-

TABLE 1

FIELD DESCRIPTION OF SOILS USED

Soil	Dune Sand Lab. No. S-6-2	Plastic Loess Lab. No. 119-5	Friable Loess Lab. No. 20-2VI	Glacial Till Lab. No. 416-4	Gumbotil Lab. No. 528-8
Location	NE$\frac{1}{4}$, SE$\frac{1}{4}$, S-16 T86N-R10W, Benton County E. Iowa	NE$\frac{1}{4}$, NW$\frac{1}{4}$, S-31 T78N-R10W, Iowa County E. Cent. Iowa	S-15, T78N-R43W, Harrison County, SW Iowa[a]	SW$\frac{1}{4}$, SW$\frac{1}{4}$, S-6 T76N-R25W, Warren County Cent. Iowa	NW$\frac{1}{4}$, NW$\frac{1}{4}$, S-7 T75N-R10W, Keokuk County SE Iowa
Geological description	Wisconsin age aeolian sand, fine-grained, oxidized, leached	Wisconsin plastic loess, oxidized, leached	Wisconsin friable loess, oxidized, calcareous	Kansan glacial till, unoxidized, calcareous	Kansan glacial till, oxidized calcareous
Soil series	Carrington	Fayette	Hamburg	Shelby	Mahaska
Horizon	C	C	C	C	Buried B
Sampling depth, ft	2 - 4	$6\frac{1}{2}$ - 9	39 - 40	7 - $16\frac{1}{2}$	$7\frac{1}{2}$ - $8\frac{1}{2}$

[a]Sampled from bluff behind third ward school, Missouri Valley, Iowa.

TABLE 2

PROPERTIES OF SOILS SELECTED FOR STUDY

Properties	Dune Sand	Plastic Loess	Sand-Loess Mixture	Friable Loess	Glacial Till	Gumbotil
Physical properties:						
Liquid limit, %	19.0	38.4	N.D.[a]	32.9	38.2	87.1
Plastic limit, %	N.P.	17.2	N.P.	21.1	15.1	34.5
Plasticity index, %	-	21.2	N.D.	11.8	23.1	52.6
Shrinkage limit, %	12.64	17.4	N.D.	28.3	N.D.	N.D.
Specific gravity	2.64	2.70	N.D.	2.68	2.65	N.D.
Standard Proctor density:						
Max. dry density, pcf	109.9	110.3	129.0	105.0	112.1	95.2
Opt. moist. content, %	12.3	16.6	9.6	18.1	14.1	28.0
Chemical properties:						
Organic matter, %	0.04	0.3	0.3	0.2	0.75	0.2
Carbonates, % $CaCO_3$	0.02	1.8	0.35	10.2	3.5	0.8
pH	6.5	5.7	6.3	7.8	7.4	6.5
Cation exchange capacity, me/100g	1.76	15.28	4.66	13.4	14.8	45.3
Particle size analysis[b], %						
Sand (2mm to 74μ)	95.8	1.0	70.0	0.2	32.0	17.5
Silt (74μ to 5μ)	1.2	65.0	20.0	82.6	2.8	8.5
Clay: (<5μ)	3.0	34.0	10.0	17.0	40.0	74.0
(<2μ)	2.9	27.0	4.0	12.3	34.0	71.0
Engineering classification AASHO	A-3(0)	A-6(13)	A-2-4(0)	A-4(8)	A-6(11)	A-7-5(20)
Textural classification (BPR)	Sand	Silty clay	Sandy loam	Silty clay loam	Clay	Clay

[a]Not determined.

[b]Based on that fraction of the soil material passing the No. 10 U. S. Standard sieve. Normal samples of the glacial till and gumbotil contain small quantities of gravel.

TABLE 3

PROPERTY ANALYSIS OF THE LIGNINS[a]

Property	Lignin A	Lignin B	Lignin C	Lignin D	Lignin E
Total solids, %	50.0	52.4	51.0	94.0	94.0
Total sugar, %	1.2	8.84	19.9	1.2	5.70
Total sulfur, %	N.D.[b]	N.D.[b]	6.02	N.D.[b]	5.73
Ash, %	17.3	8.7	15.4	17.3	9.15
Calcium and/or magnesium oxide, %	7.50	N.D.[b]	5.68	7.50	N.D.[b]
Methoxyl	8.8	N.D.[b]	N.D.[b]	8.8	9.54
Specific gravity	1.24	1.281	1.2764	N.D.[b]	N.D.[b]
pH	4.55	4.0	4.7	4.65	N.D.[b]
Physical state	Water solution	Water solution	Water solution	Powder	Powder
Chemical name	Calcium lignosulfonate	Calcium lignosulfonate	Calcium lignosulfonate	Calcium lignosulfonate	Ammonium lignosulfonate
Trade name	Toranil A	----[c]	Norlig	Toranil B	----[c]
Manufacturer	Lake States Yeast Corporation Rhinelander, Wisconsin	Consolidated Water Power and Paper Company Wisconsin Rapids, Wisconsin	Marathon Corporation Rothschild, Wisconsin	Lake States Yeast Corporation Rhinelander, Wisconsin	----[d]

[a]Mostly contributed by the manufacturer.
[b]Not determined.
[c]Not a commercial product.
[d]Supplied by Sulphite Pulp Manufacturers Research League.

centrations in the soils varying from 0.25 to 2.0 percent by dry soil weight. The concentrations of lignin are based on the total amount of solids.

METHODS OF INVESTIGATION

The investigation was divided into three phases: (1) A preliminary study with the friable loess was made to determine the best order of mixing the chemicals and lignins with the soil. One amount of each organic chemical and varying amounts of lignin A were used. (2) Using the best mixing order, varying amounts of each of the five lignins were evaluated with mixtures of friable loess and varying amounts of the cationic chemicals. (3) The lignin producing the best results in (2) was used in the third phase, an evaluation of chemical-lignin treatments with all the soils.

Mixing, molding, curing and testing techniques are briefly described in the following paragraphs.

Mixing and Molding. Mixing was done with a Model C-100 Hobart mixer at moderate speed. Six 2-in. diameter by 2-in. high cylindrical specimens were molded to near standard Proctor density for each combination of soil, chemical and lignin evaluated. The drop hammer molding procedure has been previously described (9,11,14). The height and weight of each specimen was recorded following molding. Representative samples of the mixture were obtained from the mixing bowl for moisture content determinations after molding the second and fourth specimens.

Curing. Previous studies (11) have shown air-drying to be the most

beneficial method of curing soils treated with organic cationic chemicals. All specimens in this study were air-cured at room temperature for seven days, weighed, and their height measured. Three specimens for each soil, chemical and lignin combination were then tested for unconfined compressive strength and three were immersed in distilled water for 24 hr. Following immersion the specimens were weighed, measured, and then tested for strength.

Unconfined Compression Testing. Specimens were tested for unconfined compressive strength at a deformation rate of 0.05 in. per minute per inch height of specimen. The maximum load, in psi, causing failure of the specimen was recorded as the compressive strength. Moisture determinations were made after testing on two whole specimens of each air-dry and immersed group.

MIXING ORDER STUDY

A preliminary study was made to determine the best order of mixing chemical, lignin and soil. Various percentages of lignin A and 0.2 percent of each chemical (dry weight of soil basis) were mixed with the friable loess. Three orders of mixing were tried:

A. The organic cationic chemical dispersion was mixed with the soil for 1 min after which the mixture was scraped from the sides and bottom of the bowl. The lignin solution was then added and mixing was continued for 1 min.

B. This procedure was the same as in (A) above, with the exception that the lignin solution was introduced first and the chemical second.

C. The chemical dispersions and the lignin solutions were mixed together and then incorporated into the soil. The two time intervals of mixing were the same as in (A).

The best strength results were obtained from mixing order (A) as shown in Figure 1. Two percent lignin A and 0.2 percent of each chemical produced the highest air-dry and immersed strengths. Mixing order B gave slightly lower air-dry and immersed strengths. Mixing order C was the least desirable procedure as shown by the reduced immersed strengths. A flocculent precipitate was formed when the chemicals and lignin A were combined (mixing order C). The precipitate may indicate a polymerization of lignin and cationic chemical. Moisture absorption, expansion and density variations generally followed the pattern of the immersed strength results, being most desirable with mixing order A and least desirable with mixing order C. Mixing order A was therefore chosen for use throughout the remainder of the study.

COMPARISON OF LIGNINS

In order to determine the lignin producing maximum benefits with each of the organic cationic chemicals, varying percentages of the five lignins were evaluated with the friable loess and various amounts of each chemical.

As shown in Figure 2, the air-dry strength of the friable loess generally increased with increased lignin content for each concentration of Arquad 2HT. The immersed strengths show the same general trend except in a few cases the effect of the Arquad 2HT was slightly better than the combined effects of chemical and lignin. This is most noticeable with lignin A and 0.3 percent Arquad 2HT. The best over-all compressive strengths

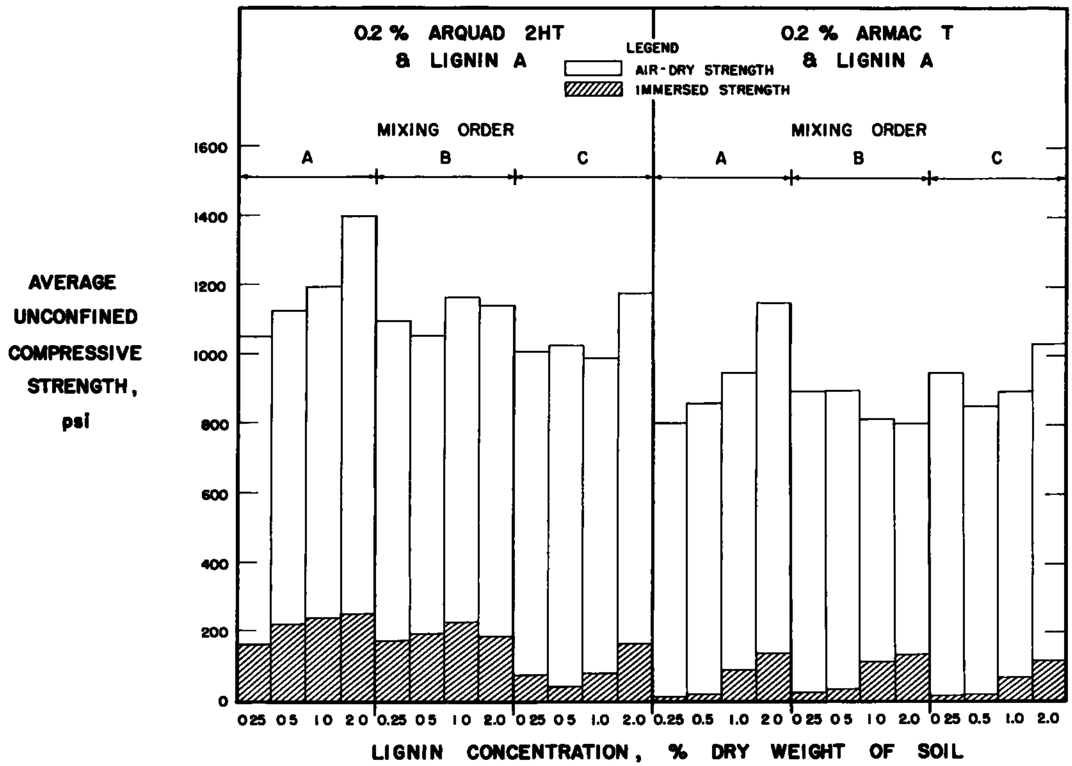


Figure 1. Effect of mixing order on the average unconfined compressive strength of the friable loess using 0.2 percent cationic chemical and varying amounts of lignin A.

were obtained at 0.2 percent Arquad 2HT for lignins A and B; for lignins C and D the compressive strengths remain nearly constant for Arquad percentages of 0.2 and above; very little benefit is shown for Arquad percentages above 0.2 with lignin E. The percent moisture absorption of the friable loess-Arquad 2HT-lignin combinations was generally lowest with lignins A and B; the percent expansion after immersion was a minimum with lignin B. The dry densities were consistently greater with lignin B which may have contributed to the good results with this lignin. Lignin B was chosen for use with Arquad 2HT for the remainder of the investigation.

Figure 3 shows that the air-dry strengths of the loess-Armac T-lignin mixtures generally increased with increasing amount of lignin, but the air-dry strengths generally are not as high as those obtained with the Arquad 2HT. In general 2.0 percent of each of the lignins gave the highest immersed strengths for each amount of Armac T. The variation of immersed strengths with additive content was much more erratic than with the Arquad 2HT. The highest immersed strength for each lignin was not obtained with the same amount of Armac T. Lignins C and E produced best immersed strength results at 0.1 percent Armac T, while lignins A, B and D were best at 0.5, 0.3 and 0.2 percent Armac T, respectively. Minimum expansion and moisture absorption with each lignin appeared at or near the respective Armac T concentrations noted above. Lignin C generally gave the lowest expansion and

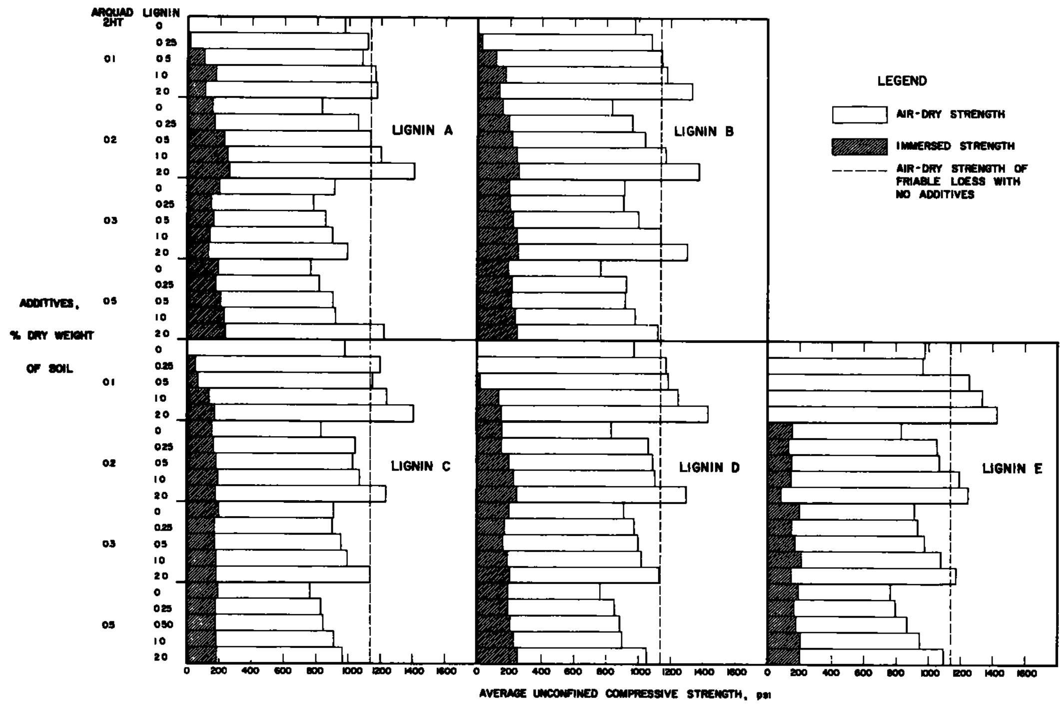


Figure 2. Effect of amount of Arquad 2HT and each lignin on the average unconfined compressive strength of the friable loess.

moisture absorption, and lignin E gave the highest. The dry densities of the loess-Armac T-lignin mixtures did not correlate well with the strengths, though lignin C generally produced the highest densities. Lignin C was chosen for use with Armac T in the remainder of the investigation.

Three known lignin variables which could have affected the results of this phase of the investigation were commercial form, total sugar content, and ionic composition (Table 3). Lignins A, B and C were supplied in water solutions while D and E were powders. The test results do not indicate that the commercial form of the lignins prior to preparation of solutions and incorporation into the soil contribute to the variation in results.

With Arquad 2HT the total sugar content of the lignin does not appear to affect strength. With Armac T the sugar content may have affected strength results:

1. Lignins A and D (1.2 percent sugar) gave the lowest immersed strengths.
2. Lignin C (19.9 percent sugar) gave high immersed strengths at low Armac T contents.
3. Lignin B (8.8 percent sugar) gave slightly higher immersed strengths than lignin C but only with higher Armac T contents.
4. Lignin E (5.7 percent sugar) gave the highest immersed strengths but also gave the greatest amount of expansion and absorption.

Lignins A, B, C and D are calcium lignosulfonates and lignin E is an ammonium lignosulfonate. The lignin E gave the poorest results which may possibly be due to the ammonium ion.

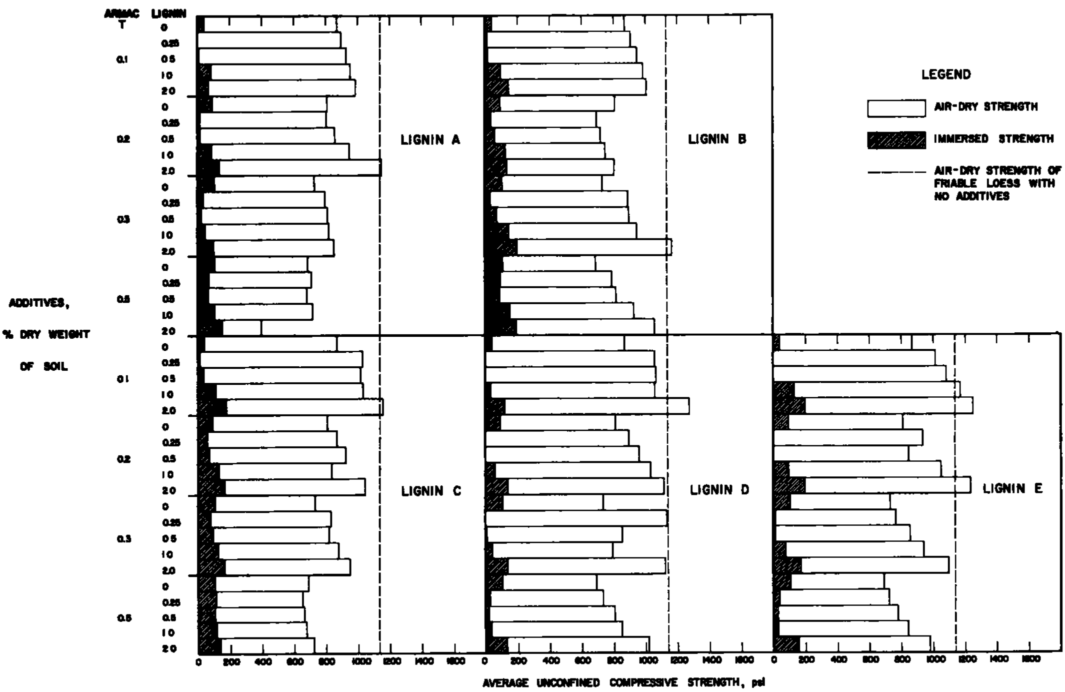


Figure 3. Effect of amount of Armac T and each lignin on the average unconfined compressive strength of the friable loess.

EFFECTIVENESS AND CLAY CONTENT

The remainder of the investigation was an evaluation of combinations of the cationic chemicals and the lignins selected for use with each chemical. The combinations were used with four soils in which the 5μ clay content ranged from 10 to 74 percent (Table 2).

Unconfined Compressive Strength

Unconfined compressive strength results obtained with mixtures of the four soils and varying amounts of Arquad 2HT-lignin B and Armac T-lignin C are shown in Figures 4 and 5. The maximum cationic chemical content shown in these figures is 0.7 percent; with the glacial till and gumbotil, treatments of 0.9 percent showed no difference in results and were not included in these figures.

Air-Dry Strength. Air-dry strengths of all the soil-organic cationic chemical mixtures (no lignin) decreased as the amount of chemical increased. This is in agreement with previous investigations (9,11) and is attributed to reduced bond energy due to increased amounts of the organic material on the particle surfaces.

The dry strengths of the sand-loess mixture and the friable loess generally were increased by increasing amounts of lignin when the amount of chemical was constant. This strength increase was as much as 75 percent with 2.0 percent lignin and Arquad 2HT in the sand-loess mix. Strength is decreased at equal lignin contents as the cationic chemical content in-

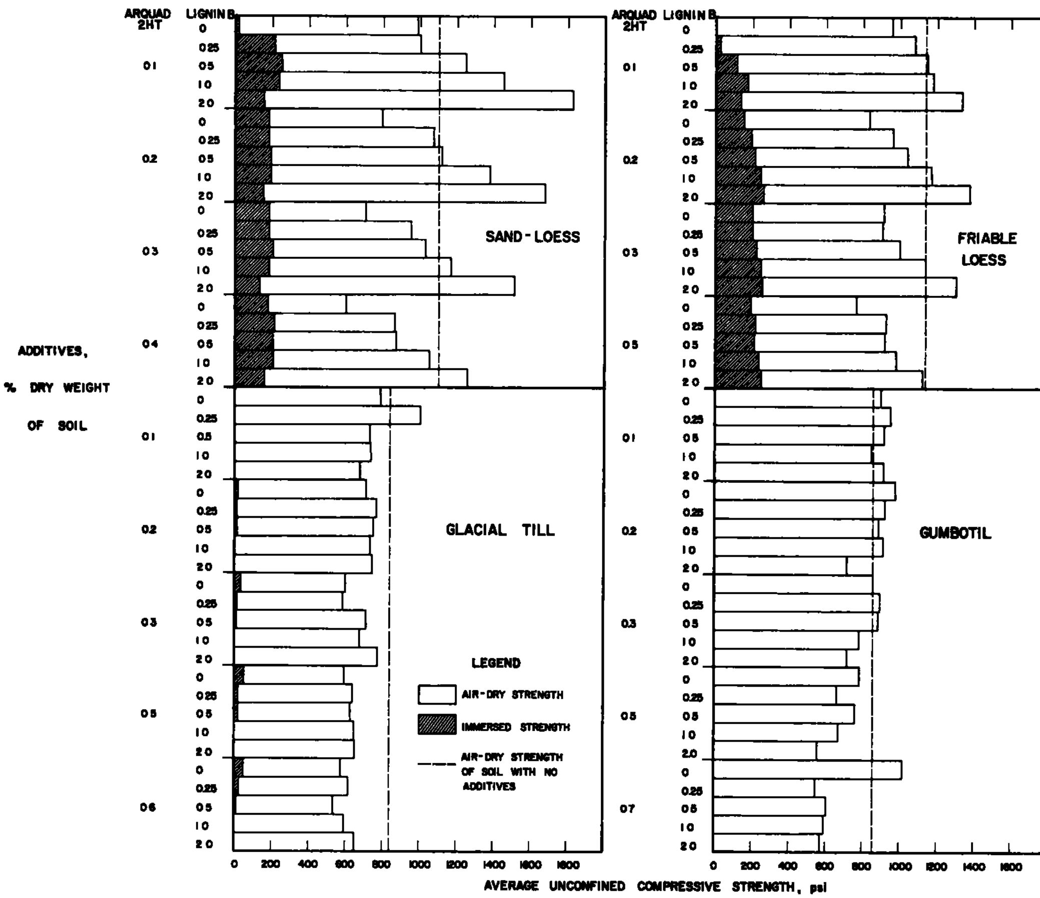


Figure 4. Effect of amount of Arquad 2HT and lignin B on the average unconfined compressive strength of four soils of different clay content.

creases. An exception is the maximum firable loess dry strength with 0.2 percent Arquad 2HT and 2.0 percent lignin B.

The additives produced erratic air-dry strength results with glacial till and gumbotil particularly in the latter soil. Most of these erratic data may be attributed to the extreme difficulty in mixing and molding these highly plastic materials. The gumbotil exhibited considerable balling during mixing and molded samples were not uniform in quality or density. This also was true to a lesser degree with the glacial till. The addition of lignin does not materially increase the air-dry strength.

Immersed Strength. The cationic chemical-lignin treatments benefited immersed strengths of the sand-loess mixture and the friable loess (Figs. 4 and 5). The glacial till immersed strength was very slightly benefited by the cationic chemicals but the addition of lignin was detrimental. Gumbotil immersed strength derived no apparent benefits from the addition of either lignin or cationic chemical.

Immersed strengths of sand-loess and friable loess treated with 0.1 percent Arquad 2HT were increased by the addition of lignin; similar ben-

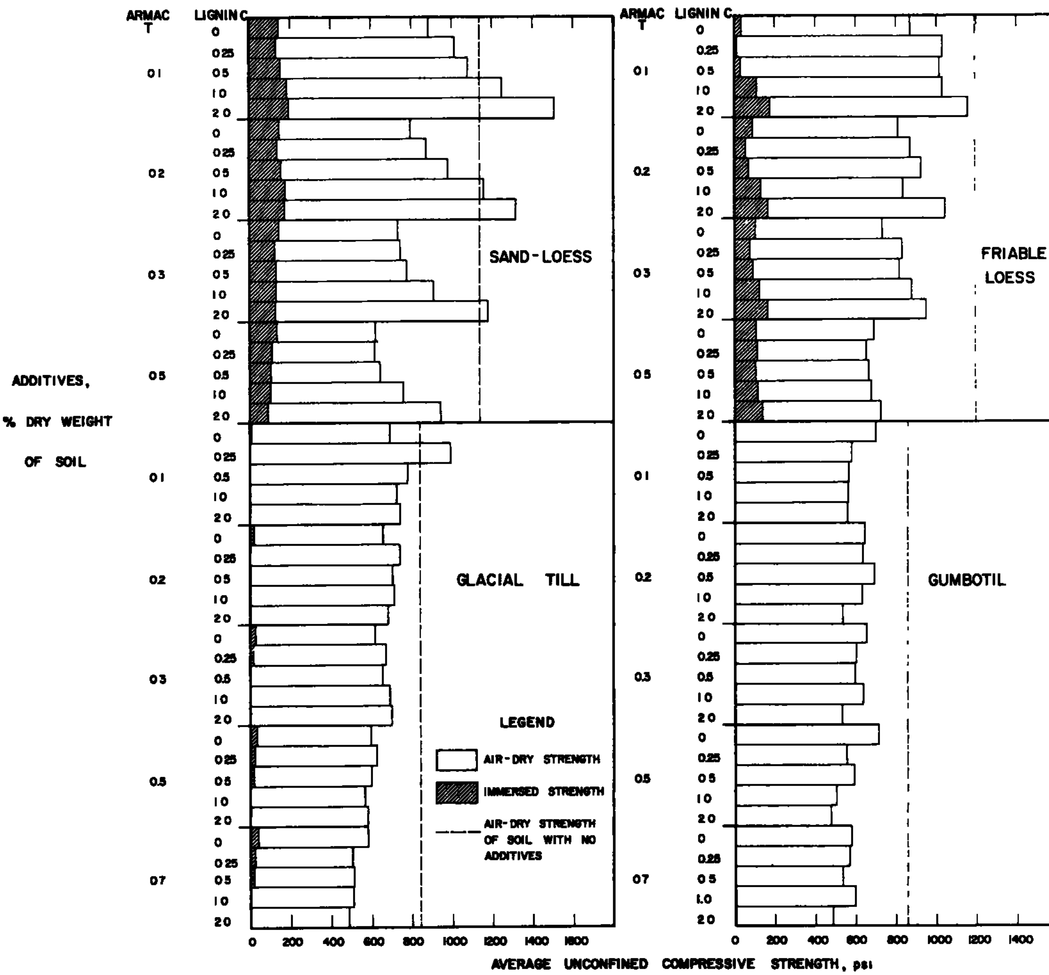


Figure 5. Effect of amount of Armac T and lignin C on the average unconfined compressive strength of four soils of different clay content.

efits were obtained at 0.2 percent Arquad with the friable loess. The addition of lignin to these soils treated with Arquad percentages of 0.2 and above for the sand-loess, and 0.3 and above for the friable loess, gave little or no immersed strength increase above that obtained with Arquad alone.

Immersed strengths of sand-loess and friable loess treated with 0.1, 0.2 and 0.1, 0.2, 0.3 percent Armac T, respectively, were reduced by the addition of 0.25 percent lignin. The addition of 0.5 percent lignin raised the strength to a value comparable to the value obtainable with Armac T alone; higher lignin percentages increased the immersed strength to values above the original value with the exception that lignin decreased the immersed strength of sand-loess treated with 0.3 and 0.5 percent Armac T. Also, the 0.5 percent Armac T treated friable loess immersed strength was benefited very little by the addition of lignin.

Moisture Absorption and Expansion. The amount of moisture absorption

TABLE 4

SUMMARY OF RESULTS WITH ADDITIVES SHOWING THE BEST STABILIZING EFFECTS

Determination	Sand-Loess Mixture	Friable loess	Glacial till	Gumbotil
Clay content, $< 5\mu$, %	10.0	17.0	40.0	74.0
Arquad 2HT, % dry wt. of soil	0.1	0.2	0.5	0.7
Lignin B, % dry wt. of soil	0.5	2.0	0.0	0.5
Air dry strength, psi	1,244	1,377	588	603
Immersed strength, psi	248	256	48	--[a]
Shrinkage during curing, %	0.1	0.4	0.6	4.7
Expansion during immersion, %	0.0	0.2	2.6	--[a]
Moisture absorption after immersion, % oven-dry wt.	2.4	6.1	15.8	--[a]
Dry density, pcf	128.4	106.7	113.6	92.4
Armac T, % dry wt. of soil	0.1	0.1	0.5	0.5
Lignin C, % dry wt, of soil	2.0	2.0	0.0	0.0
Air dry strength, psi	1,509	1,158	595	713
Immersed strength, psi	192	176	31	--[a]
Shrinkage during curing, %	0.1	0.5	1.1	5.9
Expansion during immersion, %	0.0	0.6	3.6	--[a]
Moisture absorption after immersion, % oven-dry wt.	2.4	7.9	12.6	--[a]
Dry density, pcf	126.2	106.3	114.0	92.4

[a]Specimens failed during immersion.

and expansion (expansion was calculated as the ratio of the difference in height after curing and after immersion to the height of the sample after curing) measured after 24 hr immersion in water was generally inversely related to the immersed strength and directly to the clay content; that is, as the immersed strength increased, moisture absorption and expansion decreased, whereas as clay content increased, moisture absorption and expansion increased. The sand-loess mixture showed the least amount of moisture absorption (2 to 3 percent) at the maximum immersed strengths for each concentration of Arquad 2HT and Armac T. The amount of swelling was negligible for the sand-loess mixture and ranged from zero to about 0.3 percent for all combinations of Arquad 2HT and lignin B. Moisture absorption and expansion of the friable loess was generally low though slightly greater than for the sand-loess mixture. The absorption and expansion of the glacial till was generally excessive and due to the failure of all immersed specimens of gumbotil, absorption and swelling data were unobtainable.

Density. In general when cationic chemical content was constant the oven-dry densities increased with increased lignin contents for the sand-loess mixture and the friable loess. The maximum density obtained with each cationic chemical content decreased slightly with increased amounts

of chemical. Densities of the glacial till and gumbotil specimens decreased with increased amounts of both cationic chemical and lignin.

Discussion of Effect of Clay Content. Table 4 presents a summary of results with additives showing the best stabilizing effects with the four soils of different montmorillonitic clay contents. The best treatments for the gumbotil were chosen on the basis of dry strength, shrinkage, and dry density.

As reported in previous investigations (9,11) the optimum amount of cationic chemicals required for maximum immersed strength increases with increasing amount of clay. The data presented in Figures 4 and 5 and the summary shown in Table 4 indicate that a range of montmorillonitic clay contents exist, within which cationic chemicals and lignin are effective. The data also support the findings of previous investigations, although amounts of cationic chemical necessary to stabilize the high clay content soils were not investigated. It appears that at some clay content between 17 and 40 percent, the cationic chemical and lignin treatment will produce an optimum immersed strength. Soils containing different clay minerals may exhibit different results with this type of treatment.

CONCLUSIONS

The following conclusions are based on phases one and two of the investigation and represent the use of the chemicals and lignins with the friable loess only:

1. The cationic chemical should be mixed with the soil prior to the addition of lignin.
2. Commercial form, solution or powder, of the lignin does not appear to affect the stability of the soil.
3. Total sugar content of the lignin does not affect the stability when used with Arquad 2HT. The stability of soil-Armac T-lignin mixtures appears to be improved by higher sugar contents.
4. The use of calcium lignosulfonates appears to be more beneficial than ammonium lignosulfonates.

The remaining conclusions are based on the third phase of the investigation and represent the use of the cationic chemicals and lignins with four Iowa soils of different montmorillonitic clay contents:

5. The use of lignin and small amounts of the organic cationic chemicals is beneficial to stabilization of Iowa soils with low to medium clay contents.
6. Lignin is detrimental when used with the organic cationic chemicals to stabilize medium to high clay content soils.
7. Soils of low clay contents may be adequately stabilized with 0.1 percent Arquad 2HT-0.5 percent lignin or 0.1 percent Armac T-2.0 percent lignin.
8. Soils of moderate clay content may be adequately stabilized with 0.2 percent Arquad 2HT-2.0 percent lignin or 0.1 percent Armac T-2.0 percent lignin; the stability benefits may be higher using the Arquad 2HT.
9. Cationic chemical-lignin stabilization can be economical with low to medium clay content soils.

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