Organic Cationic Chemicals as Stabilizing Agents For Iowa Loess

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This paper presents the results of preliminary evaluation studies of a number of organic cationic chemicals as stabilizing agents for Iowa loess. The loess ranges texturally from silty loam to silty clay. An unconfined compression test is used for rating the chemicals. In this test, the principal criteria of stability are compressive strength, moisture absorption, and swelling after 24 hours immersion in water. The effects of the chemicals on air-dry strength and shrinkage during air drying are also considered in evaluating the benefits to stability.

Though of a preliminary nature, the investigation demonstrates the superiority of several of the nineteen cationic chemicals used. Chemicals designated by the trade names Arquad 2HT, Arquad 2S, Armeen Residue, Armac T, Crude Amme, and Armeen Residue Arquad, are considered particularly worthy of further study. These chemicals in amounts ranging from 0.08 to less than 1.0 percent by dry weight of the soil substantially improve the stability of loess. Used in such amounts, the cost of the chemicals per square yard of base course six inches thick ranges from about 0.14 to 1.13, which is economically feasible for highway construction.

It is believed that conventional highway construction equipment and procedures could be used for the processing of soil with organic cationic chemicals. The most practical method of applying the chemicals to the soil is as solutions or dispersions in water; a solution or dispersion would be added to the soil in the amount necessary for compaction to near standard Proctor density. In the concentrations used, the viscosity of the solution or dispersion is low enough to permit spraying. Field experimentation will be necessary to evaluate more fully the effectiveness of the chemicals.

● SOIL stabilization research in progress in the Iowa Engineering Experiment Station since 1950 is directed towards finding economical ways of stabilizing the more common soil materials of Iowa for road construction purposes. To date, studies have been primarily concerned with loess, which is the surficial deposit over approximately twothirds of the state. Organic cationic chemicals are among the many compounds that have been screened as possible stabilizing agents for Iowa loess.

The term organic cationic chemical denotes a chemical, organic in nature, which dissociates in water to produce organic cations which may have exceedingly complex structures. Compared with the inorganic cations such as calcium, magnesium, hydrogen, or sodium, the organic cations are large, hence the frequently used expression large organic cations. A characteristic of organic cations is that one or more organic radicals of hydrophobic (water-hating) nature are part of the cation. When incorporated in the soil in amounts less than the cation exchange capacity, the organic cations are adsorbed rather completely to the clay surfaces of the soil in cation exchange reactions, replacing smaller, inorganic cations that are present. The adsorbed organic cations may be visualized as being oriented in such a way that the hydrophobic part of the cation is arrayed on the clay surface or directed outward from the surface. Considerable areas are thus formed on the involved clay surfaces that are not wetted by water. The more completely the clay surfaces are covered by adsorbed organic cations, the more hydrophobic the clay becomes.

Iowa Engineering Experiment Station studies of large organic cations as soil stabilizing agents date back to 1947. Results with fine-grained soils and with soil-aggregate mixtures have been reported (7, 8, 9, 17, 24) and indicate that large organic cations have considerable promise as soil stabilizing agents. Small amounts of organic cations, sufficient only to saturate partially the cation exchange capacities of the soils, were very effective in decreasing the soil's water absorption, swelling, plasticity, and shrinkage properties. Reduction of air-dry strength was the only undesirable property change noted, but this was more than compensated for by the increase in immersed strength.

Among the organic cationic chemicals investigated in the earlier Iowa studies, a long-chain fatty amine acetate known by the trade name Armac T was considered most promising. The quantitative effect of Armac T on soil properties related to all-weather stability was found to be a function of at least three variables: amount of clay, types of clay minerals, and kinds of exchangeable inorganic cations. An amount of Armac T equivalent to 10 to 25 percent of the soil's cation exchange capacity was judged to give near optimum results when both dry and immersed strength were used as criteria for stability. Treatment of two soil-aggregate mixtures with an amount of Armac T equal to 3 percent of the dry soil weight reduced the plasticity indices from 12.7 and 11.8 to less than 3 and changed the engineering classification of both soils from A-2-6(0) to A-1-b; the four-day soaked CBR values were increased from 60 percent and 57 percent to 91 percent and 73 percent.

Subsequent to the Iowa work, Waldemarson (23) used Armac T as the control chemical in a comparison of several organic compounds as stabilizing agents for a Pennsylvania clay loam. Armac T gave the best strength preserving qualities of the chemicals tested; with a 2 percent admixture based on the soil weight, over 60 percent of the natural compacted soil strength was retained after a four-day soaking period.

The number of organic cationic chemicals now in existence or that can be produced is large, and it seemed probable that Armac T, while promising, did not represent the best possible organic cationic chemical for soil stabilization purposes. Therefore in the loess stabilization research a number of additional chemicals were selected, primarily on the basis of recommendations by the manufacturer, for a preliminary-type evaluation. The most promising chemicals of previous studies, including Armac T,

	Properties	Soil A	Soil B
Physical	Liquid limit, %	30.8	51.9
properties	Plastic limit, %	24.6	18.5
	Plasticity index, %	6.2	33.4
	Shrinkage limit, %	22. 3	19.1
	Specific gravity, 25°C/4°C	2.71	2.72
	Standard Proctor density test:		
	Max. dry density, lb. /ft. ³	108.4	104.3
	Opt. moist. content, %	18.0	19.1
Chemical	Organic matter, %	0. 17	0.37
properties	Carbonates. %CaC0 ₃	10.2	0.5
	Oxidation	oxidized	oxidized
	nH	8.7	6.7
	Cation exchange capacity, $m_{e}e_{e}/100g_{e}$	13.4	24.4
	Exchangeable cations, m. e. $/100g$.		
	Na	1.5	1.3
	K	1.6	1.3
	Ca	10.3	21.8
Textural	Sand. %	1.4	0.4
a composition a	Silt %	78.8	60.2
composition	Clay: Finer than 5μ . %	19.8	39.4
	Finer than 2μ , %	16.0	33.0
Engineering cl	assification (AASHO)	A-4(8)	A-7-6(18)
^a Sand - 2.0 to	0.074 mm., silt - 0.074 to 0.005 mm., cl	ay - finer than 0.	005 mm.

TABLE 1

PROPERTIES OF WHOLE WISCONSIN LOESS SAMPLES

Group	Chemical	Physical state of chemical for mixing with soil	Availability from manufacturer	1954-55 price dollars per lb. in car load quantities
	Armac T	Solution in water	Commercial	0.30
	Arquad 2HT	Dispersion in water	Commercial	0.36
	Arquad 2S	Dispersion in water	Commercial	0.43
А	Armeen Residue Arquad Rosin Amine D Citrate in	Dispersion in water	Experimental	0. 23(est.)
	Pine Oil and Emulsifier N (3-aminopropyl) Rosin	Emulsion in water	Experimental	-
	Amine D Diacetate	Solution in water	Experimental	-
	Vinsol NVX	Solution in water	Commercial	0.0615
	Duomeen T	Solution in water	Commercial	0.37
	Ethomeen T $/12$	Solution in water	Pilot Plant	0.49 ^a
В	Ethomeen T/15	Solution in water	Commercial	0.47
	Ethoduomeen T/13	Solution in water	Commercial	0.45
	Crude Amine	Solution in water	Commercial	0.18
	Armine Residue	Solution in water	Commercial	0.09
	Rosin Amine D Acetate Monoethanol Rosin	Solution in water	Commercial	0.18
	Amine D Acetate	Solution in water	Commercial	0, 25
С	Polyrad 0200 Acetate	Solution in water	Experimental	-
-	Polyrad 0500 Acetate	Solution in water	Experimental	-
	Polyrad 1100 Acetate	Solution in water	Experimental	-
	Polyrad 2000	Solution in water	Commercial	0.36
D	Rosin Amine D Citrate	Powder	Experimental	-
	Pulverized Vinsol	Powder	Commercial	0.04
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CHEMICALS EVALUATED, GROUPED ACCORDING TO PROCEDURE FOR PREPARING THEM FOR ADDITION TO SOIL

^a In quantities of 1 to 9 drums.

were included for comparison. The principal objectives of the investigation were: (1) to rate the chemicals according to their promise as soil stabilizing agents, and (2) to determine if one or more of them would warrant further and more detailed study.

DESCRIPTION OF MATERIALS

Soils

Loess of Wisconsin geological age is one of the most abundant and widely distributed soil materials in Iowa. The Iowa Engineering Experiment Station has been engaged in engineering and geological property studies of Iowa loess since 1950, and several reports on this work have been presented (10, 11, 12, 13, 16, 20, 22). The loess ranges texturally from silty loam to silty clay.

Two samples of C-horizon loess were used in the evaluation of the organic cationic chemicals; Soil A was chosen to represent the friable, calcareous loess, Soil B to represent the plastic, leached loess. Properties of the two samples are given in Table 1; X-ray analysis of the minus 2 micron clay material of the two soils indicate; (1) for Soil A, montmorillonite is abundant, with about one-third as much illite, possibly a trace of kaolinite, and about 10 percent quartz, and (2) for Soil B, montmorillonite is predominant, with very little illite, no kaolinite, and about 10 percent quartz. Attention is directed to the difference in cation exchange capacity of the two soils; Soil B has almost twice the exchange capacity of Soil A. The predominant cations associated with the clay in both soils are calcium (Ca++).

Chemicals

The amines and quaternary ammonium salts constitute the largest groups of organic cationic chemicals, and most of the chemicals studied are of these groups. In Table 2, the chemicals are listed by their trade names as a matter of convenience, since more descriptive designations are space consuming, and for the average civil engineer difficult to understand. Available information on the chemistry of the chemicals is given in Appendix A. Vinsol NVX and Pulverized Vinsol are not considered to be cationic; they were included for comparison with the cationic materials and because they have been investigated as soil stabilizers by other investigators (21). The major criteria for the selection of the chemicals were present or future availability and cost, and probable effectiveness as soil stabilizing agents. The chemicals designated as available in pilot plant or experimental quantities in Table 2 could, according to the manufacturers, be produced in commercial quantities if a demand existed.

<u>Preparation of Chemicals</u>. It was determined by experimentation and correspondence with the manufacturers that the most practical way of adding the chemicals to soil was either as a solution or dispersion in distilled water, or as a powder, depending on the chemical nature of the material used. The chemicals are grouped in Table 2 according to the procedure followed in preparing them, in the physical state indicated, for addition to the soil. Details of the procedure for each group are given in Appendix B. The concentrations of active chemical in water solution or dispersion prepared for the first two chemicals tested were $\frac{1}{2}$, 1, 3, 5, 7, and 9 percent by weight. The completion of these tests indicated that optimum results might be expected from $\frac{1}{2}$ to 3 percent concentrations; thereafter, concentrations of $\frac{1}{2}$, 1, 3, and 7 percent were prepared of the other chemicals. Since the specific gravities of the chemicals were very close to that of water, solutions were prepared on a weight basis without adjustments for variations in specific gravity.

METHOD OF PRELIMINARY EVALUATION

An unconfined compression test of 2 inch diameter by 2 inch high cylindrical specimens molded to near standard Proctor density was used for rating the chemical treatments. In this test the principal criteria of stability of specimens are compressive strength, moisture absorption, and swelling after 24 hours complete immersion in water. The effect of the chemical treatment on air-dry strength and shrinkage during air drying may also be considered in evaluating the benefits to stability.

This method of evaluation was selected because the nature of the investigation called for a simple and quick, yet sufficiently severe, method of comparing a large number of chemical treatments. Other tests will be used for further evaluation of the chemicals considered most promising as soil stabilizing agents.

Mixing

Mixing was done in a Model K4-B Kitchen Aid mixer. Twelve hundred grams of airdry soil passing the No. 10 sieve, enough for preparing six specimens, was placed in the mixing bowl, and, for a given concentration of active chemical in water solution or dispersion, an amount necessary for compaction to standard Proctor density was added; the exact amount added depended on the optimum and hygroscopic moisture contents of the soil, and on allowances for evaporation,¹ but was the same amount for the concentrations of each chemical used. Mixing was done at No. 2 speed for about two minutes.

The two chemicals used in powder form were added to the air-dry soil in amounts equivalent to the additives of active chemical in water solution or dispersion, and then dry mixed with the soil; next, enough distilled water was added to bring the mixture to optimum standard Proctor moisture content plus 0.5 percent for evaporation; mixing was then continued as described in the preceding paragraph.

The amount of chemical mixed with the soil may be expressed in three ways: (1) percent concentration of chemical in the water added for compaction to standard Proctor

¹The amount added for evaporation during mixing and molding was 0.5 percent.

density, (2) percent of oven-dry weight of soil, and (3) percent saturation of the cation exchange capacity of the whole soil. The relationships between these different ways of expressing the amount of chemical additive are given in Tables 3, 4, 5, and 6. It will be noted in the latter two tables that none of the treatments were equivalent to 50 percent saturation of the cation exchange capacity of either soil, and the percent saturation

TABLE 3

RELATIONSHIP BETWEEN CHEMICAL CONCENTRATION IN WATER AND PERCENT DRY WEIGHT OF SOIL FOR SOILS A AND B AND GROUP I CHEMICALS

Ob any is a 1	Soil A						Soil B					
admixture	Amo	unt of	chemi	cala a	dded t	o soil	for w	ater c	oncent	ration	s ^b be	low.
	¹ /2%	1%	3%	5%	7%	9%	¹∕₂%	1%	3%	5%	7%	9%
Armac T	0.08	0.16	0.49	0.82	1.14	1.47	0.08	0.15	0.45	0.76	1.06	1.36
Duomeen T	0.08	0.16	0.49	0.82	1.14	1.47	0.08	0.15	0.45	0.76	1.06	1.36
Ethomeen $T/12$	0.08	0.16	0.49	_ d	1.14	-	0.08	0.15	0.45	_	1.06	
Ethomeen T/15	0.08	0.15	0.46		1.08	-	0.08	0.15	0.45	-	1.06	-
Ethoduomeen T/13	0.08	0.15	0.46	-	1.08	-	0.08	0.15	0.45	-	1.06	-
Arquad 2HT	0.08	0.16	0.49	-	1.14	-	0.08	0.15	0.45		1.06	-
Arquad 2S	0.08	0.16	0.49	0.82	-	••	0.08	0.15	0.45	0.76	_	-
Crude Amine	0.08	0.15	0.46	-	1.08	-	0.08	0.15	0.45	4	1.06	-
Armeen Residue Armeen Residue	0.08	0. 15	0.47	-	1.08	-	0.08	0.15	0.45	-	1.06	-
Arquad	0.08	0.16	<u>0</u> . 48	0.95	c _	-	0.08	0.15	0.45	0.909	з.	-

^a Percent dry weight of soil.

b Percent by weight of total solution.

c 6% chemical concentration in water.

d Dashes indicate that specimens were not molded.

TABLE 4

RELATIONSHIP BETWEEN CHEMICAL CONCENTRATION IN WATER AND PERCENT DRY WEIGHT OF SOIL FOR SOILS A AND B AND GROUP II CHEMICALS

		:	Soil A			Soil	В			
Chemical admixture	Amount of chemical ^a added to soil for water concentrations ^b shown below.									
	¹ /2%	1%	3%	7%	¹ /2%	1%	3%	7%		
RADA	0.08	0.16	0.49	1.14	0.08	0.15	0.45	1.06		
Monoethanol RADA	0. 08	0.16	0.49	1, 14	0.08	0.15	0.45	1.06		
P 0200 A	0.08	0.16	0.46	1.08	0.09	0.17	0.51	1.19		
P 0500 A	0.08	0.16	0.46	1.08	0.09	0.17	0.51	1.19		
P 1100 A	0.08	0.16	0.46	1.08	0.09	0.17	0.51	1, 19		
P 2000	0.08	0.16	0.46	1.08	0.09	0.17	0.51	1.19		
RAD Citrate .	0.07	0.15	0.45	1.04	0. 08	0.17	0.50	1 16		
RAD Cit. Solution in				-		•• ••		1.10		
Pine Oil and Emulsifier	0.07	0.15	0.45	1.04	0, 08	0.17	0.50	1 16		
N (3-aminopropyl) RAD						••••	0.00	1. 10		
Diacetate	0.07	0.15	0.45	1.04	0.08	0, 15	0.45	1 05		
Vinsol NVX	0.07	0.15	0.45	1.04	0.08	0.17	0.50	1 16		
Pulverized Vinsol	0.07	0.15	0.45	1.04	0.08	0.17	0.50	1.16		

^a Percent dry weight of soil.

b Percent by weight of total solution.

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TABLE 5

RELATIONSHIP BETWEEN CHEMICAL CONCENTRATION IN WATER AND PERCENT SATURATION OF CATION EXCHANGE CAPACITY FOR SOILS A AND B AND GROUP I CHEMICALS

				Soil A					S	01l B		
Chemical		P	ercent chem	satura ical cor	tion of ncentr	catio ation i	n exc in wa	hang ter ^a	e cap show	acity : n belo	for w	
aumixture	¹ /2%	1%	3%	5%	7%	9%	¹ /2%	1%	3%	5%	7%	9%
Armac T	1.9	3.8	11.3	18.9	26.4	34.0	1.0	1.9	5.7	9.6	13.4	17.3
Duomeen T	2.4	4.7	14.1	23.5	32.9	42.2	1. 2	2.4	7.2	12.0	16.7	21.6
Ethomeen T/12	1.5	3.0	9.0	_b	20.9	-	0.8	1.5	4.6	-	10.6	-
Ethomeen T/15	1.1	2. 1	6.4	-	14.9	-	0.6	1.2	3.4	-	8.0	-
Ethoduomeen T/13	1.0	2.1	6.2	-	14.4	-	0.6	1.1	3.3	-	7.8	-
Arouad 2HT	0.8	1.6	4.7	-	10.9	-	0.4	0.8	2.4	-	5.6	-
Arouad 2S	1.0	2.0	6.1	14.3	-	-	0.5	1.0	3.1	5.2	-	-
Crude Amine	1.7	3.3	10.0	_	23.4	-	0.9	1.8	5.4	-	12.6	-
Armeen Residue	1.2	2.3	7.0	-	16.2	-	0.6	1 . 2	3.7	-	8.7	-
Armeen Residue Arquad	0.7	1.5	4.4	8. 8c	-	-	0.4	0.8	2.3	4.6	c _	-

^a Percent by weight of total solution.

b Dashes indicate that specimens were not molded.

c 6% chemical concentration in water.

TABLE 6

RELATIONSHIP BETWEEN CHEMICAL CONCENTRATION IN WATER AND PERCENT SATURATION OF CATION EXCHANGE CAPACITY FOR SOILS A AND B AND GROUP II CHEMICALS

		3	Soil A			Soil	В	
Chemical	Perc	ent satu	concent	f cation ex ration in v	kchange water ^a	capacit shown b	y for cl elow.	nemical
	¹ /2%	1%	3%	7%	¹ /2%	1%	3%	7%
RADA	1.1	2.3	6.8	15.8	0.6	1.2	3.5	8.1
Monoethanol RADA	1.1	2. 1	6.3	14.6	0.5	1.2	3.2	7.4
P 0200 A	1.3	2.6	7.9	18.4	0.8	1.6	4.8	11.1
P 0500 A	1.0	2, 1	6.2	14.4	0.6	1.2	3.7	8.7
P 1100 A	0.6	1.3	3.8	9.8	0.4	0.8	2.3	5.4
P 2000	0.4	0.9	2.7	6.2	0.3	0.5	1.6	3.8
RAD Citrate RAD Cit. Solution in	1. 2	2.4	7. 2	16.7	0.7	1.4	4.4	10.2
Pine Oil and Emulsifier N (3-aminopropyl) RAD	1. 2	<u>.</u> 2.4	7. 2	16.7	0.7	1.4	4.4	10 . 2
Diacetate	1.0	2.0	6.1	14. 2	0.6	1.1	3.4	7.8
a Percent by weight of tot	al solu	tion.						

with most treatments was much less than 50 percent. An example of the calculation of the percent saturation of cation exchange capacity has been given previously (7, 8).

Molding

Six 2-inch diameter by 2-inch high specimens were molded for each combination of soil and chemical evaluated. The drop-hammer type molding apparatus has been pre-

viously described (6). Approximately 200 grams of soil-chemical mixture was placed in the cylindrical mold in one layer and compacted to near standard Proctor density by ten blows of the 5 pound hammer dropping from a height of 12 inches. Immediately after molding, each specimen was weighed and the height measured. After the third of the six specimens was molded, a representative sample of the mixture was taken from the mixing bowl for a moisture content determination; it was found to be unnecessary to correct the moisture determination for the amount of chemical present in the sample.

Curing

The effectiveness of organic cationic chemicals as soil stabilizers was found to be related to the curing procedure used (19). The highest degree of stability as determined by the immersion-compression test was obtained by air drying the compacted 2-inch diameter by 2-inch high specimens to constant weight prior to testing; seven days at room temperature proved sufficient to accomplish this. Specimens tested without drying back to a low moisture content had a lower degree of stability, while the use of oven drying at elevated temperatures was not beneficial.

It was decided to use the most favorable curing procedure in the preliminary evaluation of the chemicals. Accordingly, all molded specimens were air dried for seven days, then weighed, and the decrease in height measured.

Testing

Specimens were tested for compressive strength by the unconfined compression test. The maximum load in pounds causing failure of the specimen was taken as the compressive strength. The test load was applied at a rate of deformation of 0.05 inch per minute per inch of height of the specimen. Three specimens of each set were tested in the air-dry condition, and the other three were tested after 24 hours complete immersion in distilled water; the heights of the immersed specimens were measured prior to testing. Moisture determinations were made after testing on the second specimen of each group; the whole soaked specimen, and a sample obtained from the central portion of the air-dry specimen were used as moisture samples.

RESULTS OF PRELIMINARY EVALUATION

The relative effectiveness of the different chemicals and amounts of chemicals as stabilizing agents for the two loess materials was compared by using the previously described method of evaluation. Since the test method used has not been correlated with the field performance of stabilized soils, the test results give only an indication of relative stability. Criteris for judging relative stability of the test specimens are the change in height and the amount of moisture absorbed during 24 hours immersion, and the air-dry and immersed compressive strengths. Low volume change and moisture absorption and improvement in immersed strength are the principal indications of benefits to stability by chemical treatment.

In considering the numerical value of the test results, it should be realized that complete immersion of air-dry test specimens in an unconfined condition is a very severe treatment, particularly when the



Figure 1. Effect of amount of chemical admixture on average unconfined compressive strength of Soil A with Group I chemicals.



Figure 2. Effect of amount of chemical admixture on average unconfined compressive strength of Soil B with Group I chemicals.

caused a reduction of air-dry strength, but some caused less than others. In general, the trend of the data shows a rather sharp reduction in air-dry strength from 0 to 0.17 percent admixture, based on the oven-dry weight of the soil, and then a gradual leveling off with the larger amounts of additive. The air-dry strengths of Soil A specimens varied from a maximum of 2,850 lb. for the chemically untreated soil to a minimum of about 300 lb. for the largest amount of additive; with Soil B specimens, this range was

3,220 lb. to about 550 lb. The irregularities to be noted in some of the curves were checked by testing additional samples, but no appreciable change from the original data was found. It is of interest to note that the two non-cationic resinous materials, Pulverized Vinsol, and Vinsol NVX, had the least affect on air-dry strength.

The effect of the chemicals on soil density, in the amounts added to the soils, was found to be slight; and the moisture contents of the air-dry test specimens were found to decrease with increasing amounts of a chemical. On the basis of these findings and what is known about the nature of the chemicals, the reduction of air-dry compressive strength by the chemical treatments is attributed to the partial destruction of the natural cohesion in the soil furnished by thin continuous films of water on clay surfaces. Such films cannot be continuous on clay surfaces partially shielded by adsorbed large organic cations of a hydrophobic nature. The two noncationic resinous materials would appear to have a similar but lesser effect on cohesion.

stabilizer is not of the bonding or cementation type as was true in this investigation. Chemically untreated soil specimens completely disintegrate after only a few minutes immersion.

The data have been divided into two groups, Groups I and II, on the basis of the source of the chemicals (Appendix A). For easier understanding, the amount of chemical admixture is usually presented as percent of oven-dry weight of soil, but conversions to the other ways may be made by means of Tables 3 to 6, inclusive. Test results are the average of data obtained from at least three test specimens.

Unconfined Compressive Strength

Unconfined compressive strength results obtained with mixtures of Soils A and B and varying amounts of the different chemicals are shown in Figures 1, 2, 3. and 4.

Air-Dry Strength. All chemicals



Figure 3. Effect of amount of chemical admixture on average unconfined compressive strength of Soil A with Group II chemicals.



Figure 4. Effect of amount of chemical admixture on average unconfined compressive strength of Soil B with Group II chemicals.

Immersed Strength. The immersed strength data, Figures 1, 2, 3, and 4, demonstrate the superiority of some of the chemicals as waterproofing agents. Taken as a whole, Group I chemicals showed up better than Group II chemicals; untreated specimens, as was expected, failed within minutes after immersion.

The trend of the data on specimens that did not fail shows an increase in immersed strength from zero to a maximum and then either no further gain or a decrease in strength with increasing amounts of chemical. The only data showing a significant variance from this trend are those of Armeen Residue Arquad — Soil B mixtures (Figure 2). Due to the small amount of this chemical available for experimental purposes, it was impossible to carry the curve further.

In general, the chemicals were less effective with the plastic loess, Soil B, than with the friable loess, Soil A. Except for one chemical, the maximum immersed strengths obtainable with Soil A were greater than those of Soil B; Vinsol NVX gave both soils approximately the same maximum immersed strength. Also, the amount of the chemicals needed to obwas greater than with Soil A.

tain maximum immersed strength with Soil B was greater than with Soil A.

Moisture Absorption and Swelling

As a general rule the trends of the moisture absorption and volume change data agreed with the immersed strength data and showed that chemical treatments giving highest immersed strength also gave lowest moisture absorption and swelling. The moisture increase and swelling that did occur during the 24 hour immersion apparently introduced stresses and strains and increased the thickness of water films on clay surfaces to the extent that unconfined compressive strength was decreased below that of the air-dry specimens.

Most Promising Cationic Chemicals

Though of a preliminary nature, the investigation demonstrated the superiority of several of the cationic chemicals evaluated. Arquad 2HT, Arquad 2S, Armeen Residue, Armac T, Crude Amine, and Armine Residue Arquad are considered particularly worthy of further study. Test results obtained with near optimum amounts of these chemicals are summarized in Table 7, for Soil A, and in Table 8, for Soil B. Amounts of the chemicals ranging from 0.08 to less than 1.0 percent by oven-dry weight of the soil are capable of substantially improving the stability of Iowa loess. Used in such small amounts, the cost of the chemicals per square yard of base course six inches thick would, as shown in Tables 7 and 8, range from about \$0.14 to \$1.13, which is economically feasible for highway construction. It is of interest to note here that Armac T, the most promising cationic chemical discovered in the earlier studies of the Iowa Engineering Experiment Station, was not found to be best in this investigation.

From a comparison of the data on Soils A and B in Tables 7 and 8, the organic cationic chemicals selected as showing the most promise would be usable and beneficial to both firable and plastic loess, but with the plastic loess, the amounts of the chemicals needed would be greater and the degree of stability obtained would be lower.

TABLE 1

SUMMARY OF RESULTS WITH CHEMICALS SHOWING THE BEST STABILIZING EFFECTS WITH SOIL A

	Raw Soil	Arquad 2HT	Arquad 28	Armeen Residue	Armac T	Crude Amine	Armeen Residue Arquad
Amount of chemical, % dry wt. of soil		0, 08	0, 16	0.38	0.24	0, 30	0.50
Amount of chemical, % saturation of C. E. C.	-	0, 8	20	6. OC	5.5 °C	6, 6C	3.7C
Avg unconfined comp. strength after 7 days air drying, lb.	2847	2830	1660	1300	1200	1010	770
Avg. unconfined comp. strength after 7 days air drying and 24 hr. immersion, lb.	-	330	310	330	215	260	170
Avg. decrease in height after 7 day air drying. in.	0, 012	0.0074	0. 0102	0. 0085 ^c	0, 0066 ^c	0.0067 c	0.0106
Avg. increase in height after 24 hr.	-	0. 0203	0. 0173	0. 0110 ^c	0. 0155 ^c	0.0110°	0. 0110 ^c
Moist, cont. at time of molding,	18.7	18.8	19.2	18. 8 ^c	16.7C	18.7C	17.5°
Moist. cont. after 7 day air drying,	1.7	31	2. 3	18 ^c	2.9°	2.5 ^C	2 2 ^c
Moist. cont. after 24 hr. immersion,	-	9.0	7.7	7.7C	9.5 °	8 Oc	8.3 C
Avg. dry density, pcf. 8	105.0	99. 9	103 4	102.0°	100, 1 ^C	101.6°	100, 5 C
Cost of chemical/yd. ² of surf. area, 6 in. thick, \$ ^b	•	0. 14	0. 34	0. 17	0. 36	0. 27	0, 57

^a Near standard Proctor density.

b This is the cost of the chemical for a compacted volume one yard square and six inches thick, with a density of 110 pcf.

^c Interpolated from tables and graphs.

TABLE 8

SUMMARY OF RESULTS WITH CHEMICALS SHOWING THE BEST STABILIZING EFFECTS WITH SOIL B

	Raw Soil	Arquad 2HT	Arquad 28	Armeen Residue	Armac T	Crude Amine	Armeen Residue Arquad
Amount of chemical, % dry wt. of soil	-	0.45	0.45	0, 60	0. 76	0,60	0. 45
Amount of chemical, % saturation of C.E.C.	-	2.4	3.1	5.4 C	9.6	8.0C	23
Avg. unconfined comp. strength after 7 days air drying, lb.	3220	1040	1330	1050	1460	1060	1520
Avg. unconfined comp strength after 7 days air drying and 24 hr. immersion, lb	-	160	120	150	90	160	150
Avg. decrease in height after 7 day air drving, in.	0. 051	0. 0120	0. 0253	0. 0135 ^c	0. 0274	0. 0130 ^c	0. 0248
Avg. increase in height after 24 hr. immersion. in.	-	0.0517	0.0503	0. 0220 c	0,0610	0. 02 0 0 c	0.0536
Moist cont. at time of molding, % oven-dry wt of treated soil	19.5	18. 2	18.4	17. 7 ^{°C}	17. 2	17, 7 C	18.6
Moist. cont after 7 day air drying, % oven-dry wt. of treated soil	37	5. 1	4.5	2.6 ^C	33	2.6 °	4, 3
Moist cont. after 24 hr. immersion, % oven-dry wt. of treated soil	-	13.9	13.6	10. 6 °	16.3	9.8°	13, 2
Avg dry density, pcf. a	103.5	97.4	97.8	92.7 C	97.4	99.2 C	100 6
Cost of chemical/yd ² of surf area, 6 in thick, \$b	-	0, 80	0, 96	0. 27	1. 13	0. 54	0.51

a Near standard Proctor density.

^bThis is the cost of the chemical for a compacted volume one yard square and six inches thick, with a density of 110 pcf.

^c Interpolated from tables and graphs.

The difference in results obtained with the two soils is mainly due to difference in amount and kind of clay minerals. Soil B has approximately twice the clay content and cation exchange capacity of Soil A, and has a higher proportion of montmorillonite type clay minerals.

Comparison with Other Kinds of Stabilizing Agents

A comparison of the 24-hour immersed unconfined compressive strengths of 2-inch diameter by 2-inch high specimens of Soil A, stabilized with four of the more promising organic cationic chemicals and with other inorganic and organic additives being studied in the soil stabilization research program of the Iowa Engineering Experiment Station, is shown in Table 9. Of the stabilizing agents compared, Portland cement, hydrated lime, and cutback asphalt have been quite widely used in highway construction with varying degrees of success; and lime-fly-ash and aniline-furfural have shown considerable promise as stabilizing agents. The immersed strength obtained with Arquad 2HT, Arquad 2S, Armeen Residue, and Crude Amine, as compared with that obtainable with the better known stabilizers, further demonstrates the potenitalities of organic cationic chemicals for the improvement of the stability of Iowa loess.

The cost per pound of the organic cationic chemicals may be much higher than that

Additive		Mothod	Compressive	Cost ^a of stabilizing
Туре	Amount, % dry wt. of soil	of curing	lb. (after 24 hr. immersion)	sq. yd. of surf. area, 6 in. thick, dollars
Portland cement, Type I	15	7 days in moist cabinet at approximately 70 degrees F.	1780	0. 70
Hydrated lime	6	Same as above	250	0. 32
Lime-fly-ash (1: 2)	18	Same as above	485	0. 42
Aniline-furfural (2: 1)	5	7 days air drying	1100	4. 71
Cutback asphalt RC-1	10	Same as above	220	1. 17
Arguad 2HT	0.08	Same as above	330	0, 19
Arquad 2S	0.16	Same as above	310	0.45
Armeen Residue	0.38	Same as above	330	0.17
Crude Amine	0.30	Same as above	260	0, 27
Raw soil with no additive		Same as above	Failed during immersion	-

AVERAGE UNCONFINED COMPRESSIVE STRENGTH OF SOIL A TREATED WITH VARIOUS ADDITIVES

^aThis is the cost of the stabilizing material for a compacted volume one yard square and six inches thick, with a density of 110 pcf.

of such stabilizing agents as Portland cement, lime, and bituminous materials. But, since smaller amounts of the cationic materials are needed, stabilization may be cheaper with their use than with some of the more common stabilizing agents. Table 2 shows the standard market prices of the additives used in this comparison at the present time.

Use in Highway Construction

It is believed that conventional highway construction equipment and procedures can be used for the processing of loess with organic cationic chemicals. The most practical method of applying the chemicals to the soil would be as solutions or dispersions in water; a solution or dispersion would be added to the soil in the amount necessary for compaction to near standard Proctor or other density. In the concentrations used, the viscosity of the solution or dispersion is low enough to permit spraying. Good mixing with the soil is important to obtain adequate exposure of clay surfaces to the large organic cations. The presence of large organic cations in the mix water tends to benefit workability of plastic soils, since the consistency properties are much improved by the cation exchange reactions.

Field experimentation with organic cationic chemicals will be necessary to develop a recommeded method of construction, but more laboratory study is needed. Many things, such as use with different kinds of soil, curing requirements, and resistance to freezing and thawing, wetting and drying, and soil organisms need further investigation. The question also naturally arises of whether a better organic cationic chemical for soil stabilization purposes can be produced. The chemical industry might be able to answer this question if more fundamental knowledge about the requirements and mechanism of soil stabilization with large organic cations were made available.

The subject matter of this report was obtained as part of the research being done under Project 283-S of the Iowa Engineering Experiment Station, Iowa State College. The project, entitled "The Loess and Glacial Till Materials of Iowa; An Investigation of Their Physical and Chemical Properties and Techniques for Processing Them to Increase Their All-Weather Stability for Road Construction" is being carried on under contract with the Iowa Highway Commission, under sponsorship of the Iowa Highway Research Board. Funds for this work are supplied by the commission and the U.S. Bureau of Public Roads.

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Appendix A

Description of Chemicals Used

Information on each of the chemicals used in this study is summarized in the following paragraphs. Where they are available, the trade names of each are used to assist the reader.

Chemicals

<u>Armac T (1)</u> is a water soluble primary amme acetate made from fatty acids. It may be written as $[RNH_3]^+[Ac]^-$, similar to ammonium acetate except that a fatty group replaces one of the hydrogens. It has a molecular weight of 310.

Duomeen T (2, 3) is a fatty di-amine of the general formula:

The R represents an alkyl group derived from a tallow fatty acid. Being only slightly dispersible in water, the Duomeen T was treated with 30.0 parts of glacial acetic acid per 100.0 parts of 80 percent active Duomeen T by weight to form the water soluble diacetate salt. Duomeen T has a molecular weight of 320.

Ethomeens (3, 4). The Ethomeens are tertiary amines of the general formula:

 $\stackrel{(CH_2CH_2O)_X H}{RN}_{(CH_2CH_2O)_Y H}.$

The R represents a fatty alkyl group derived from various fatty sources having from 12 to 18 carbon atoms. Attached to the nitrogen are two polyoxethylene groups. Although the Ethomeens are normally mildly cationic, these properties are more pronounced in the water soluble salts found by neutralizing the Ethomeens with acids. However, these salts are not stable to strong alkali.

Ethomeen T/12 has one fatty alkyl group derived from tallow amine. In the previous general formula for the Ethomeens, the T/12 has two mols. of ethylene oxide (x + y). It has an average molecular weight of 365 and was made water soluble by neutralizing with 16.4 parts glacial acetic acid per 100 parts of T/12, by weight.

Ethomeen T/15 has one fatty alkyl group derived from tallow amine but has five mols. of ethylene oxide (x + y) in the two polyoxyethylene groups shown in the previous general formula for the Ethomeens. It has an average molecular weight of 497 with a water soluble salt being formed by neutralizing 100 parts of T/15 with 12.1 parts by weight of glacial acetic acid.

Ethoduomeen T/13 has the following general structural formula: a

$$\begin{array}{c} CH_2CH_2OH \\ / H H H \\ R-N-C-C-C-N \\ H H H \\ \end{array} CH_2CH_2OH \\ CH_2CH_2OH \end{array}$$

The d1-acetate salt of the T/13 was prepared by adding 21.6 parts of glacial acetic acid to every 100 parts by weight of the chemical. The molecular weight of Ethoduomeen T/13 is 558.

<u>Arquads</u> (3, 4). The Arquads are quaternary ammonium chlorides that retain their surface activity at either high or low pH and are not precipitated by calcium or magnesium hardness in water. They are described by the manuracturer as follows:

A quaternary ammonium salt may be thought of as the organic ounterpart of an ammonium salt. For example, if ammonium chloride (NH_4Cl) has all its hydrogen atoms replaced by organic groups, it becomes a quaternary ammonium salt. Arquads are quaternary ammonium salts of this type. They fall into two general groups: fatty alkyl trimethylammonium chlorides:

$$\begin{bmatrix} CH_3 \\ R-N-CH_3 \end{bmatrix} + \begin{bmatrix} C1 \end{bmatrix}^{-1}$$

and di-fatty alkyl, dimethylammonium chlorides:

$$\begin{bmatrix} \mathbf{C}\mathbf{H}_{3} \\ \mathbf{R} - \mathbf{N} - \mathbf{C}\mathbf{H}_{3} \end{bmatrix} + \begin{bmatrix} \mathbf{C}\mathbf{I} \end{bmatrix}^{-1}$$

Arquad 2HT is a d1-hydrogenated tallow di-methylammonium chloride which is easily dispersible in water for dispersions up to about 8 percent by weight.^a It is supplied as 75 percent active in isopropanol and has an average molecular weight of about 585.

<u>Arquad 2S</u> is a di-methyl dialkyl quaternary ammonium salt derived from soybean oil.^a It is currently supplied as 75 percent active in isopropanol and 1s dispersible in water at 1 to 2 percent concentrations forming flowable gels as high as $12\frac{1}{2}$ percent. It has an average molecular weight of about 595.

Armeen Residue is a crude material resulting from the process of amine distillation with a molecular weight of 497. It is a mixture of 30 to 40 percent primary amine, approximately 30 percent secondary amine, and a remainder of crude material including some polymerized material.^a Due to variations in the composition of the Armeen Residue from lot to lot, it was necessary to determine the amount of glacial acetic acid needed to form the water soluble acetate. By plotting a titration curve for Armeen Residue with varying amounts of the acid, it was determined that 6.3 parts glacial acetic acid per 100 parts Armeen Residue, by weight, were needed.

Armeen Residue Arquad is a quaternary made by treating Armeen Residue with methyl chloride. It is made up as a 50 percent solution in isopropanol, the active portion having a molecular weight of 804. The structural formula is a mixture of $RN(CH_3)_3 + Cl -$ and $R_2N(CH_3)_2 + Cl -$ where the R is primarily tallow.^a

<u>Crude Amine</u> is a 50-50 combination of Armeen Residue and Armeen T. It has a primary amine content of approximately 60 percent, the remainder being made up of

^a From private communication with the manufacturer.

secondary and tertiary amine.^a Due to the variation in percentages of amines with each lot manufactured, a titration curve for Crude Amine with varying amounts of glacial acetic acid were plotted to determine the amount of acid needed for the water soluble acetate formation. Thus it was found that 15.6 parts acid per 100 parts Crude Amine, by weight, were needed. It should be noted that there was incomplete acetate formation for both the Crude Amine and the Armeen Residue following treatment with glacial acetic acid; though the former is more complete due to its higher primary amine content.

Rosin Amine D Derivatives (18). Rosin Amine D is a technical grade of dehydroabietylamine with the following structural formula:



It is relatively stable up to about 100 deg. C; decomposing at higher temperatures over extended periods of time. It is slightly soluble in water, being less than 0.5g per 100g of water at 100 deg. C.

Rosin Amine D Acetate (18) is the acetic acid salt of Rosin Amine D. RADA, as it shall be referred to throughout the remainder of this paper, is water soluble and in the 70 percent paste form supplied by the manufacturer has a molecular weight of 338.

Monoethanol Rosin Amine D Acetate (18) is soluble in water and has a molecular weight of approximately 407. The manufacturer discusses Monoethanol RADA as follows:

Rosin Amine D reacts with ethylene oxide to form the N-substituted monoethanol derivative:

$$RNH_2 + CH_2 \quad CH_2 \rightarrow RNHCH_2CH_2OH$$

<u>Polyrads (18)</u>, or the Polyrad chemicals, are formed by reacting increasing amounts of ethylene oxide per mole of Rosin Amine D to form consistencies varying from balsam-like liquids to wax-like solids. The Polyrad chemicals used in this study and their respective approximate molecular weights ^a are: Polyrad 0200 Acetate, 440; Polyrad 0500 Acetate, 560; Polyrad 1100 Acetate, 910; and Polyrad 2000, 1300. The number of moles of ethylene oxide per mole of Rosin Amine D is indicated by the first two numbers in the trade name. The acetate salts of the first three were used because those Polyrads containing less than 10 moles of ethylene oxide per mole of Rosin Amine D are only partially soluble in water; those with higher ethylene oxide contents are soluble.

Rosin Amine D Citrate is a salt of Rosin Amine D in which one carboxyl of citric acid has been neutralized.^a It is cationic, though insoluble in water, and must be pulverized and used as a powder as described in Appendix B. It has a molecular weight of approximately 466.

Rosin Amine D Citrate Solution is a mixture formulated to render the RAD Citrate water dispersible. The mixture comprises one part RAD Citrate, one part Synthetics A. F. 150, and one part Yarmor 302-W^a, the latter two ingredients being a pine oil and an emulsifying agent respectively.

<u>N (3-aminopropyl) Rosin Amine D Diacetate</u> was supplied by the manufacturer as a 100 percent active material, a very small yellowish-white aggregate varying in size from powder to small pea gravel and easily soluble in water. It has a molecular weight of approximately 550. a

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Vinsol Resins, though not considered as cationic were included in this study for comparison with the cationic chemicals. Vinsol Resin is manufactured from residues obtained in the distillation process for turpentine (15). Vinsol Resin is a complex organic resin, phenolic in nature. It contains carboxyl, phenolic hydroxy, and methoxy groups.^a The two Vinsol products used in this study are Vinsol NVX and Pulverized Vinsol. The Vinsol NVX is water soluble, while the Pulverized Vinsol is insoluble in water and should be used in the powder form, as explained in Appendix B. The average molecular weights of Vinsol NVX and Pulverized Vinsol are 470 and 450 respectively.^a

Appendix B

Procedures for Preparing Chemicals for Addition to Soil

The following procedures were determined as being the most desirable for preparation of the chemical solutions and dispersions prior to incorporation in the soil. Table 2 indicates the chemicals prepared under each procedure.

<u>Procedure A.</u> The amount of chemical necessary to prepare a one liter volumn of the concentration of active chemical in water desired was placed in a 400 ml. beaker. Distilled water, previously heated to 60 deg. C or slightly above, was mixed with the chemical until it was entirely dissolved or dispersed. The mixture was then washed into a 1000 ml. volumetric flask and diluted with the heated water to 1000 ml. To compensate for the change in volume due to temperature, it was calculated that an additional 13 ml. of water were needed to have a volume of 1000 ml. at room temperature.

Though there was little visible separation noticed in any of the physical states prepared under this procedure, each container was vigorously shaken for several minutes for complete dispersion of any separated particles before being used.

<u>Procedure B.</u> Each of the six chemicals for which this procedure was used are insoluble in water in the form they are obtained from the chemical manufacturer. Upon treatment with glacial acetic acid, in the amounts previously given in Appendix A, the acetate salts formed are either entirely or partly water soluble. The amount of acid needed was added slowly to the chemical with constant stirring. This was continued until the temperature caused by the heat of reaction had decreased sufficiently to permit the acetate salt to begin to thicken to its normal room temperature status. The acetate salt was then placed in an air tight container and allowed to stand overnight before being dissolved in water. The remainder of the procedure was exactly that of Procedure A.

Except for the Crude Amine and Armeen Residue, the prepared solutions were completely dissolved; as previously stated there is incomplete water soluble acetate formation with the Crude Amine and Armeen Residue upon treatment with glacial acetic acid. Therefore the insoluble material floated to the top of the container after standing for a short time. Before each use, the containers of these two chemicals were shaken vigorously to disperse the residual material.

<u>Procedure C.</u> This procedure was recommended by the manufacturer. The chemical, as supplied by the manufacturer, was placed in a large Erlenmeyer flask and was diluted with an equal quantity of distilled water at room temperature. This mixture was stirred thououghly, yielding a heterogeneous mass, allowed to stand overnight, and with some additional stirring, yielded a homogeneous solution which was then diluted to a 10 percent stock solution by the further addition of water. The amount of stock solution needed for a desired concentration was placed in a volumetric flask and diluted to 1000 ml. with distilled water at room temperature.

<u>Procedure D.</u> The Pulverized Vinsol was received from the manufacturer as a powder and required no further preparation before mixing with the soil. However, it was necessary to pulverize the RAD Citrate to a powder form. The powders thus prepared were added to the soil in the manner described in the procedure for mixing.