

## Stabilization of Loess With A Promising Quaternary Ammonium Chloride

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Previously reported investigations of the Iowa Engineering Experiment Station have indicated the promising effects of a number of organic cationic chemicals as stabilizing agents for Iowa loess. From the standpoint of economic feasibility and improvement of immersed compressive strength, moisture absorption and swelling, a quaternary ammonium chloride, known commercially as Arquad 2HT, was considered especially promising.

This paper presents a further evaluation of Arquad 2HT with calcareous silty loess and leached clayey loess. The chemical was added to the soils as a water dispersion; for most tests a 3 percent concentration in water was used with the silty loess and a 5 percent concentration in water for the clayey loess (0.47 and 0.85 percent of the dry soil weight).

Tests were made to determine the effect of Arquad 2HT on such soil properties as plasticity, moisture-density relationship, moisture-curing time relationship, unconfined compressive strength, bearing capacity, and resistance to weathering. Results indicate that:

1. Immersed compressive strength depends to a great extent on the type of curing preceding immersion. Air drying proved to be the best curing method.
2. Plasticity decreased.
3. Maximum dry density decreased slightly in both soils, whereas the optimum moisture content of the silty loess increased and that of the clayey loess decreased.
4. The soaked California Bearing Ratio increased and swelling decreased.
5. A considerable resistance to physical weathering was indicated.

On the basis of studies made and those reported by other investigators, a theoretical interpretation of the mechanism of soil stabilization with organic cationic compounds is presented. Differential thermal analyses of the silty loess treated with Arquad 2HT, both under normal and nitrogen atmospheres, are evaluated.

● LOESS OF WISCONSIN geological age is a major surficial material in Iowa and adjacent states, and a means of treating the loess to make it suitable for use as a road base course is of great importance because of a scarcity of natural aggregates in loess areas. In recent years many methods of soil stabilization have been investigated by the Iowa Engineering Experiment Station with this objective in mind. One of the methods, organic cationic stabilization, which tends

to give the loess greater stability through a chemical or molecular change in the surface characteristics of its clay, has shown a definite promise. Previous research has shown that treatment with large organic cations increases a soils hydrophobic (water-hating) characteristics and wetted strength (6, 7, 8, 10, 11, 12, 14).

A preliminary evaluation study by the Iowa Engineering Experiment Station of a number of organic cationic chemi-

cals as stabilizing agents for loess has been reported (12). This study demonstrated the superiority of several of the nineteen cationic chemicals used. Those designated by the trade names Arquad 2HT, Arquad 2S, Armeen Residue, Armac T and Crude Amine improved properties of the loess such as immersed compressive strength, moisture absorption and swelling; they were considered worthy of further study. One of them, Arquad 2HT, was particularly promising due to its commercial availability, economical cost in quantities used, and ease of preparation and mixing with soil.

This report deals primarily with the further evaluation of Arquad 2HT with loess. The principal objectives of the investigation were (1) to determine the best curing conditions for the soil-Arquad 2HT mixture; (2) to determine the effect of Arquad 2HT on such soil properties as plasticity, moisture-density relationship, and moisture-curing time relationship; (3) to evaluate the durability and bearing strength of the stabilized

soils; and (4) to present a theoretical interpretation of the mechanism of soil stabilization with organic cationic compounds on the basis of studies made and those reported by other investigators.

## MATERIALS

### Soils

Two samples of C-horizon Wisconsin loess were used in this study to represent the average extremes in property variations of the loess in western Iowa. Silty soil 20-2I and clayey soil 44A-1 were chosen because of their similarity to the friable calcareous loess and plastic leached loess (soils A and B) used by Hoover and Davidson (12). Properties of the two samples are given in Table 1. Soil 44A-1 has almost twice the cation exchange capacity of soil 20-2I. The predominant inorganic cations associated with the clay of both soils are calcium ( $\text{Ca}^{++}$ ). Montmorillonite is the predominant clay mineral of each soil.

TABLE 1  
PROPERTIES OF WHOLE WISCONSIN LOESS SAMPLES

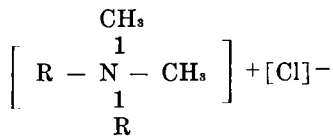
|                                      | Properties                           | Soil<br>20-2I     | Soil<br>44A-1     |
|--------------------------------------|--------------------------------------|-------------------|-------------------|
| Physical<br>properties               | Liquid limit, %                      | 34.2              | 53.1              |
|                                      | Plastic limit, %                     | 26.2              | 25.7              |
|                                      | Plasticity index, %                  | 8.0               | 27.4              |
|                                      | Shrinkage limit, %                   | 24.6              | 19.9              |
|                                      | Specific gravity, 25°C/4°C           | 2.71              | 2.72              |
|                                      | Standard Proctor density test:       |                   |                   |
|                                      | Max dry density, lbs/ft <sup>3</sup> | 104.3             | 102.7             |
| Opt moist. content, %                | 18.7                                 | 21.2              |                   |
| Chemical<br>properties               | Organic matter, %                    | 0.17              | 0.37              |
|                                      | Carbonates, % $\text{CaCO}_3$        | 10.2              | 0                 |
|                                      | Oxidation                            | oxidized          | oxidized          |
|                                      | pH                                   | 7.8               | 6.2               |
|                                      | Cation exchange capacity, me./100g   | 13.4 <sup>b</sup> | 24.4 <sup>b</sup> |
|                                      | Exchangeable cations, me./100g       |                   |                   |
|                                      | Na                                   | 1.5 <sup>b</sup>  | 1.3 <sup>b</sup>  |
|                                      | K                                    | 1.6 <sup>b</sup>  | 1.3 <sup>b</sup>  |
| Ca                                   | 10.3 <sup>b</sup>                    | 21.8 <sup>b</sup> |                   |
| Textural<br>composition <sup>a</sup> | Sand, %                              | 0.6               | 0.2               |
|                                      | Silt, %                              | 80.6              | 58.0              |
|                                      | Clay: Finer than 5 $\mu$ , %         | 18.8              | 41.8              |
|                                      | Finer than 2 $\mu$ , %               | 14.7              | 31.0              |
| Engineering classification (AASHO)   | A-4(8)                               | A-7-6(18)         |                   |

<sup>a</sup> Sand —2.0 to 0.074 mm, silt —0.074 to 0.005 mm, clay —finer than 0.005 mm.

<sup>b</sup> These values are for soils A and B used by Hoover and Davidson (12) but are considered representative of soils 20-2I and 44A-1, respectively.

### Chemicals

Amines and quaternary ammonium salts constitute the greatest source of organic cationic chemicals. The latter group are often described as being the organic counterpart of an ammonium salt. If the hydrogen atoms of ammonium chloride ( $\text{NH}_4\text{Cl}$ ) are replaced by organic groups a quaternary ammonium salt exists. Arquad 2HT is a di-hydrogenated tallow di-methylammonium chloride and falls into a group of fatty dialkyl dimethylammonium chlorides of the following general formula (3):



It is easily dispersible in water for dispersions up to about 8 percent by weight<sup>1</sup> and is supplied as 75 percent active in isopropanol with an average molecular weight of about 585. Arquad 2HT has been referred to by some investigators as DDAC and is also commercially available under the trade name Aliquat H226 (4). Concentrations of Arquad 2HT in distilled water as used in this study are on a weight basis of the material as supplied by the manufacturer and not on the active Arquad 2HT contained in the supplied material. Other organic cationic chemicals referred to in this report have been described in a previous investigation (11).

### Preparation of Soil-Chemical Specimens

Unless otherwise described, soil-chemical specimens were prepared in the following manner: The amount of chemical required for a desired water concentration was heated until liquified, and then diluted to 1000 ml with distilled water previously heated to 60 deg C. After thorough mixing and cooling, an amount of the subsequently formed dispersion, equal to the optimum moisture content of

the raw soil, was added gradually to air dried soil while being mixed in a Kitchen Aid (model K4-B) mixer at moderate speed, supplemented when necessary by hand mixing. Immediately after mixing, 2-in. diameter by 2-in. high specimens were molded to near standard Proctor density, and their heights and weights measured in a manner previously described (5, 12).

### OPTIMUM CHEMICAL CONTENT

A preliminary study was made for the purpose of determining an optimum Arquad 2HT content for the soils used. The optimum for each soil was taken as the amount of chemical giving maximum unconfined compressive strength after 7 days air drying and 24 hours immersion in water. Seven days air drying was found to be a reasonable length of time for the soil-Arquad mixture to dry to an equilibrium moisture content.

Figure 1 shows that the air dry compressive strength of both soils decreased with higher concentrations of the chemical (the data presented in this report are averages of tests made on at least 2 specimens). However, the immersed compressive strength of silty soil 20-2I increased to an optimum at 3 percent water concentration of chemical then tapered off, while the immersed strength of clayey soil 44A-1 increased to a leveling off point at about 5 percent water concentration of the chemical. These concentrations represent 0.47 and 0.85 percent of the dry soil weight and each is equivalent to slightly less than 5 percent of the cation exchange capacity of soils 20-2I and 44A-1, respectively. For ease of presenting the data, all chemical quantities will be expressed as a concentration in water.

### CURING STUDIES

Since organic cationic chemicals introduced into a soil appear to maintain the soil's stability by establishing a hydrophobic condition within the soil, it was considered probable that the strength of the soil-Arquad specimens would depend

<sup>1</sup> From private communication with the manufacturer.

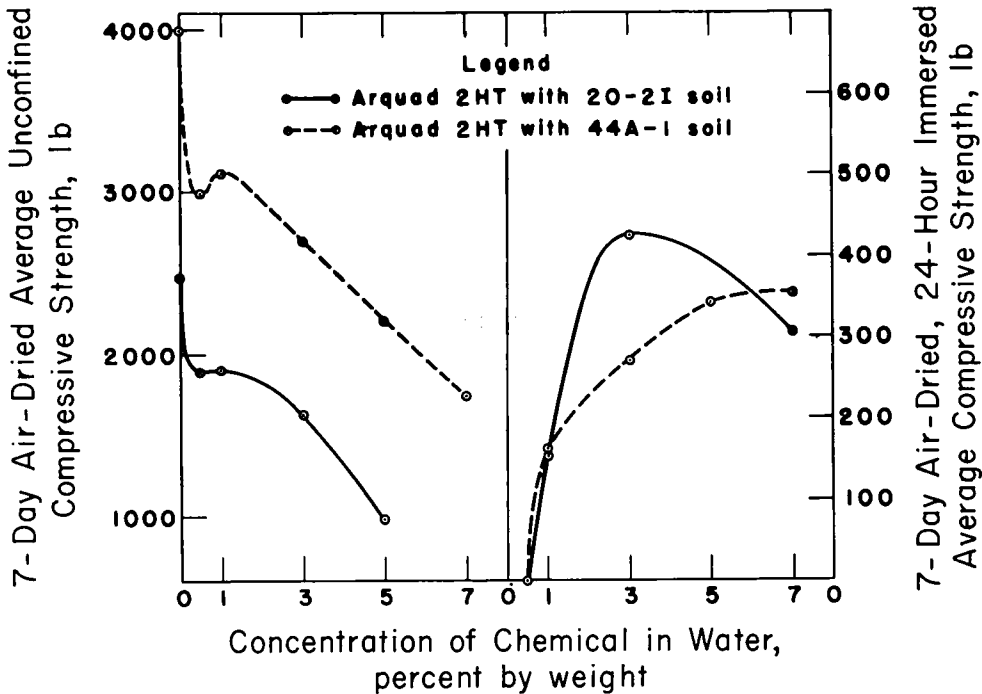


Figure 1. Effect of amount of chemical on average unconfined compressive strength of soils 20-2I and 44A-1.

to a great extent on the type of curing used prior to testing. Though only a small portion of the fourteen curing conditions investigated are likely to occur in the field, it was anticipated that information could be obtained to assist in understanding the mechanism of soil stabilization with large organic cations. To study various combinations of time, humidity, and temperature which might affect curing, specimens treated with the previously determined optimum amounts of Arquad 2HT were subjected to the following curing conditions and then tested for unconfined compressive strength after 24 hours water immersion:

1. No curing.
2. Curing at room temperature.
  - a. In open air of approximately 30 percent relative humidity.
  - b. In a dessicator with approximately 65 percent relative humidity.
3. Moist curing in a moisture cabinet at  $70 \pm 3$  deg F and approximately 90

percent relative humidity.

4. Curing at elevated temperatures.
  - a. In 110 deg F oven, wrapped.<sup>2</sup>
  - b. In 160 deg F oven, wrapped.
  - c. In 160 deg F oven, unwrapped.
5. Combination heat-moist curing.
  - a. One and two days in 90 deg F oven, then moist cured in a moisture cabinet at  $70 \pm 3$  deg F and approximately 90 percent relative humidity, unwrapped.
  - b. Same as above except in 110 deg F oven.
  - c. Same as above except in 140 deg F oven.
  - d. Same as above except in 160 deg F oven.

The length of curing for each condition varied from 0 to 14 days prior to immersion, except for the combination heat-moist curing. For this condition the specimens were first cured at the specified

<sup>2</sup> Specimens were wrapped in waxed paper and then covered with aluminum foil to prevent evaporation of moisture and contact with carbon dioxide in the air.

temperature, then were placed in a moisture cabinet for periods of 0 to 14 days followed by immersion for 24 hours.

### Results

The curing study results (Tables 2 and 3) indicate:

1. Seven days air curing at 30 percent relative humidity was the best curing

method. Therefore, determination of the optimum chemical content after 7 day air drying was justifiable.

2. The effect of length of curing was different with each method used, but was more pronounced with air curing. Even with no curing, some immersed strength was gained since untreated soil specimens disintegrated within a few minutes after immersion in water.

3. The change in relative humidities

TABLE 2  
EFFECT OF CURING METHOD AND CURING TIME ON IMMERSSED STRENGTH OF 20-21 SPECIMENS TREATED WITH 3 PERCENT WATER CONCENTRATIONS OF ARQUAD 2HT

| Curing Method                  | Immersed Compressive Strength, lb |     |     |     |     |                |                |
|--------------------------------|-----------------------------------|-----|-----|-----|-----|----------------|----------------|
|                                | Curing Time, days                 |     |     |     |     |                |                |
|                                | 0                                 | 1   | 3   | 5   | 7   | 10             | 14             |
| Air dry, 30% R.H. <sup>a</sup> | 20                                | 142 | 255 | 303 | 390 | 327            | 335            |
| Air dry, 65% R.H.              | 20                                | 45  | 45  | 55  | 67  | 70             | 70             |
| Moist, 90% R.H.                | 20                                | 40  | 52  | 65  | 65  | 65             | 83             |
| 110° F, wrapped                | 20                                | 62  | 75  | 77  | 70  | 83             | 100            |
| 160° F, wrapped                | 20                                | 75  | 83  | 83  | 90  | 113            | 113            |
| 160° F, unwrapped              | 20                                | 225 | 18  | 0   | 0   | 0              | 0              |
| 1 day, 90°F; moist, 90% R.H.   | 20                                | 40  | 60  | 68  | 125 | — <sup>b</sup> | — <sup>b</sup> |
| 2 days, 90°F; moist, 90% R.H.  | 20                                | 34  | 58  | 87  | 120 | —              | —              |
| 1 day, 110°F; moist, 90% R.H.  | 20                                | 260 | 255 | 247 | 247 | 250            | 242            |
| 2 days, 110°F; moist, 90% R.H. | 20                                | 93  | 213 | 213 | 223 | 243            | 252            |
| 1 day, 140°F; moist, 90% R.H.  | 20                                | 228 | 243 | 210 | 197 | —              | —              |
| 2 days, 140°F; moist, 90% R.H. | 20                                | 20  | 182 | 183 | 173 | —              | —              |
| 1 day, 160°F; moist, 90% R.H.  | 20                                | 220 | 205 | 235 | 250 | —              | —              |
| 2 days, 160°F; moist, 90% R.H. | 20                                | 45  | 200 | 195 | 193 | —              | —              |

<sup>a</sup> R.H. = relative humidity.

<sup>b</sup> Not determined.

TABLE 3  
EFFECT OF CURING METHOD AND CURING TIME ON IMMERSSED STRENGTH OF 44A-1 SPECIMENS TREATED WITH 3 PERCENT WATER CONCENTRATION OF ARQUAD 2HT

| Curing Method                  | Immersed Compressive Strength, lb |     |     |     |     |                |                |
|--------------------------------|-----------------------------------|-----|-----|-----|-----|----------------|----------------|
|                                | Curing Time, days                 |     |     |     |     |                |                |
|                                | 0                                 | 1   | 3   | 5   | 7   | 10             | 14             |
| Air dry, 30% R.H. <sup>a</sup> | 40                                | 117 | 147 | 268 | 358 | 372            | 407            |
| Air dry, 65% R.H.              | 40                                | 75  | 97  | 85  | 95  | 70             | 50             |
| Moist, 90% R.H.                | 40                                | 65  | 90  | 95  | 85  | 78             | 80             |
| 110° F, wrapped                | 40                                | 100 | 115 | 97  | 105 | 100            | 130            |
| 160° F, wrapped                | 40                                | 112 | 117 | 95  | 105 | 123            | 150            |
| 160° F, unwrapped              | 40                                | 202 | 122 | 130 | 80  | 60             | 82             |
| 1 day, 90°F; moist, 90% R.H.   | 40                                | 80  | 100 | 90  | 108 | — <sup>b</sup> | — <sup>b</sup> |
| 2 days, 90°F; moist, 90% R.H.  | 40                                | 72  | 92  | 68  | 75  | —              | —              |
| 1 day, 110°F; moist, 90% R.H.  | 40                                | 222 | 245 | 153 | 117 | 315            | 200            |
| 2 days, 110°F; moist, 90% R.H. | 40                                | 278 | 388 | 228 | 338 | 358            | 295            |
| 1 day, 140°F; moist, 90% R.H.  | 40                                | 340 | 345 | 335 | 343 | —              | —              |
| 2 days, 140°F; moist, 90% R.H. | 40                                | 130 | 343 | 272 | 287 | —              | —              |
| 1 day, 160°F; moist, 90% R.H.  | 40                                | 320 | 277 | 225 | 215 | —              | —              |
| 2 days, 160°F; moist, 90% R.H. | 40                                | 83  | 265 | 293 | 262 | —              | —              |

<sup>a</sup> R.H. = relative humidity.

<sup>b</sup> Not determined.

from 65 to 90 percent caused little variation in the immersed strengths.

4. Wrapped specimens cured at elevated temperatures of 110 and 160 deg F had immersed strengths slightly greater than those of specimens moist cured at  $70 \pm 3$  deg F. Moisture was preserved in wrapped specimens, and the relative humidity inside the wrapping was at least as high as that in the moisture cabinet used for normal moist curing.

5. Prolonged oven drying of unwrapped specimens at 160 deg F was extremely detrimental to the silty soil specimens and caused considerable reduction in strength of the clayey soil specimens.

6. The combination of one and two days oven drying at 90 deg F followed by normal moist curing resulted in low immersed strengths for both soils. The strength values were about the same as those obtained by normal moist curing only.

7. Drying at temperatures of 110, 140 and 160 deg F gave similar test results, except that the 2 day drying at 140 and 160 deg F caused a reduction in the immersed strengths of specimens of both soils. This strength decrease indicates a critical moisture content-immersed strength relationship. Drying of the stabilized soils to below this critical moisture content results in a reduction of immersed strength; rehydration of the stabilized soil by normal moist curing is

highly beneficial after 1 to 3 days in a moisture cabinet.

8. The loss of strength below the critical moisture content may be caused by the increase in air voids that accompanies any reduction of moisture. Below the critical moisture value, the air-void content may increase to such an extent that during immersion, water not only enters the soil rapidly but also causes compression of the air trapped within and a consequent loss of specimen strength (Figure 2).

The curing studies were performed with only one Arquad 2HT treatment of each soil, the optimum treatment determined from the immersed strength of 7 day air dried specimens (Figure 1) in the preliminary study. To determine the effect of curing method on the optimum chemical content, unwrapped specimens containing varying amounts of Arquad 2HT were cured one day at 140 deg F followed by normal moist curing for 3 days. Strength results after 24 hours immersion are compared with those from seven day air curing (Figure 3). For both methods of curing the optimum chemical concentration in water is about the same; 3 percent for soil 20-2I and 5 percent for soil 44A-1. There is an indication that 7 percent concentration might be slightly better than 5 percent for soil 44A-1, however, the viscosity of a 7 percent concentration is such that

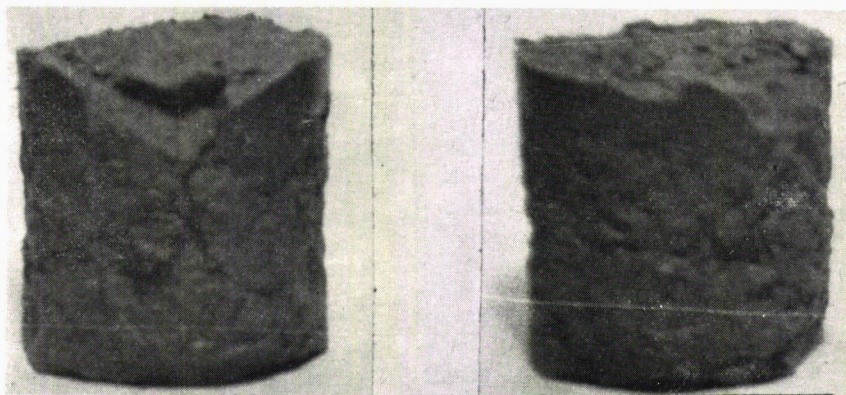


Figure 2. Heat cured specimens following 24-hr immersion in water.

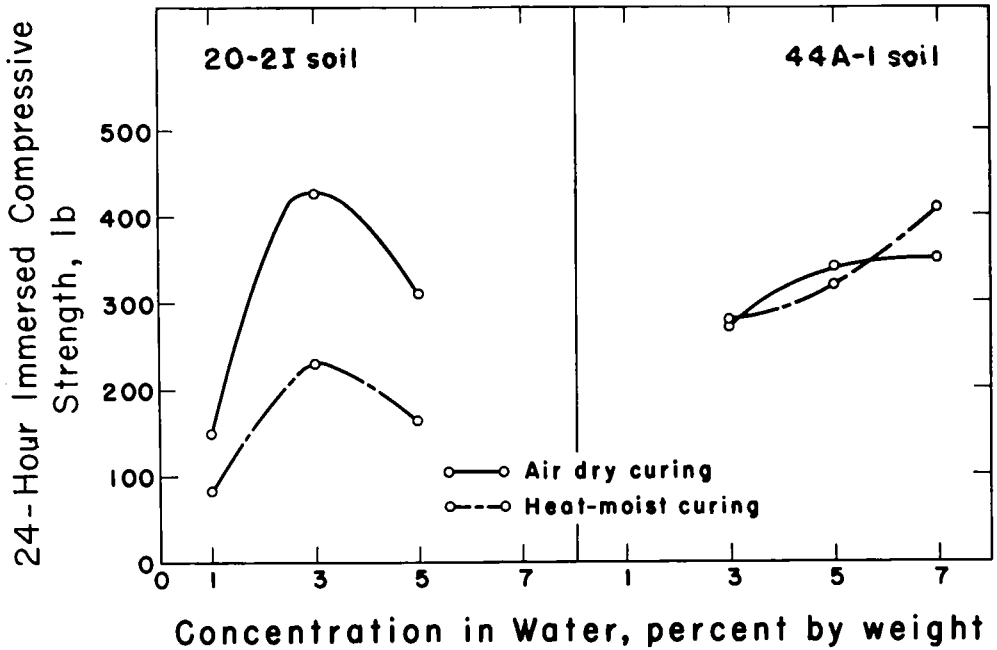


Figure 3. Comparison of 7-day air dry curing with heat-moist curing for various concentrations of chemical.

the ease of mixing the chemical with the soils is greatly reduced.

Arquad 2S, another quaternary ammonium salt, when tested under conditions exactly the same as described in the preceding paragraph gave comparable immersed strength results, indicating that treatments of Arquad 2S and Arquad 2HT have similar effects on both soils.

#### CONSISTENCY LIMITS

To determine the effect of Arquad 2HT on the consistency limits of the two soils, soil-Arquad specimens were molded, air cured for seven days, then broken and ground by mortar and pestle to pass the No. 40 sieve. The consistency limits were then determined according to standard procedures (ASTM Designations: D423-54T and D424-54T).

The plasticity of both soils was decreased by the addition of Arquad 2HT. The amount of reduction increased as the amount of chemical in the soil increased and reached an almost constant value at 3 and 5 percent concentration for soil 20-

2I and 44A-1 respectively; further increases in chemical resulted in very little change in the plasticity index of either soil.

Table 4 shows the results of the consistency limits for raw soils and for specimens containing optimum concentrations of Arquad 2HT after subjection to two curing conditions. The PI of clayey soil 44A-1 was reduced to a greater degree than that of silty soil 20-2I although the shrinkage properties of both soils were not changed to any appreciable extent by the Arquad treatments. Also, the curing method affected the liquid limit and P I of both treated soils, being lower with heat-moist curing than with air drying.

#### MOISTURE-DENSITY RELATIONSHIP

Standard Proctor moisture-density relationships (ASTM Designation: D698-42T) of raw and Arquad 2HT treated soils are compared in Figure 4 and Table 4. There was a slight decrease of the maximum dry density of both soils when

TABLE 4  
EFFECT OF CURING METHOD ON CONSISTENCY AND DENSITY PROPERTIES OF RAW AND ARQUAD-TREATED SOILS

| Soil No.                                   | 20-2I                  |                        |                         | 44A-1                  |                        |                         |
|--|------------------------|------------------------|-------------------------|------------------------|------------------------|-------------------------|
|  | 0                      | 3                      | 3                       | 0                      | 5                      | 5                       |
| Water Concentration Arquad 2HT, %          |                        |                        |                         |                        |                        |                         |
| Curing Condition                           | Air Dried <sup>a</sup> | Air Dried <sup>a</sup> | Heat-Moist <sup>b</sup> | Air Dried <sup>a</sup> | Air Dried <sup>a</sup> | Heat-Moist <sup>b</sup> |
| Liquid limit, %                            | 34.2                   | 31.5                   | 30.1                    | 53.1                   | 44.1                   | 42.6                    |
| Plastic limit, %                           | 26.2                   | 24.8                   | 24.9                    | 25.7                   | 28.1                   | 27.9                    |
| Plasticity index, %                        | 8.0                    | 6.7                    | 5.2                     | 27.4                   | 16.0                   | 14.7                    |
| Shrinkage limit, %                         | 24.6                   | 24.2                   | 24.2                    | 19.9                   | 19.9                   | 17.1                    |
| Shrinkage ratio                            | 1.64                   | 1.67                   | 1.67                    | 1.81                   | 1.81                   | 1.83                    |
| Max std Proctor density, pcf               | 104.3                  | 101.0                  | —                       | 102.7                  | 100.2                  | —                       |
| Optimum moisture at std Proctor density, % | 18.7                   | 19.5                   | —                       | 21.2                   | 19.5                   | —                       |

<sup>a</sup> Seven days air drying at room temperature.

<sup>b</sup> One day at 140° F, 6 days moist curing at 90% relative humidity.

treated with the chemical; however, the optimum moisture content of the silty soil was slightly increased, whereas that of the clayey soil was slightly decreased. From these results, it is permissible to

use the optimum moisture content of the raw soil as a basis for estimating the amount of chemical-water dispersion to be added to the soil in preparing test specimens.

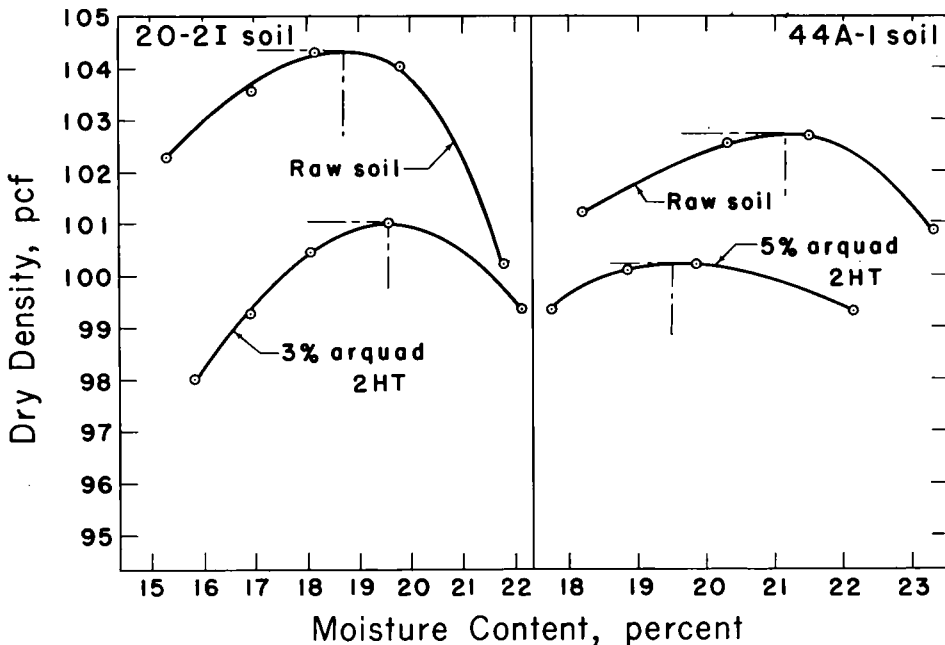


Figure 4. Comparison of moisture-density relationship of raw and stabilized soils.



## MOISTURE-CURING TIME RELATIONSHIP

To compare the effect of curing on the moisture content of raw and optimum Arquad 2HT treated soil specimens, a check was made on moisture variation during air drying and during heat-moist curing (one day at 140 deg F followed by 6 days normal moist curing). Moisture determinations, expressed as a percentage of the moisture content at the time of molding, were made at 12 hours and at 1, 3, 5, and 7 days from the time of molding (heat-moist cured specimens were also checked at 4 and 8 hours). The same specimens were then immersed in water and moisture determinations, expressed in the same manner, were made at 4 and 8 hours, and at 1, 3, 5, and 7 days after immersion (Figure 5).

Only slight differences could be detected in rate of moisture loss between raw and stabilized soils. The moisture content of heated specimens was reduced to 7 or 8 percent of their optimum moisture content in 24 hours. After placement in the moisture cabinet for 6 days, they had regained moisture up to 22 and 34 percent of the optimum moisture contents of the silty and clayey soils, respectively. After immersion, the moisture contents of both soils increased rapidly for the first day but then began to level off. Because of partial satisfaction of their affinity for moisture during the moist curing period, specimens cured in

this manner absorbed less moisture during 7 days immersion than did specimens previously air dried for 7 days, indicating that elevated 140 deg F temperature during curing does not destroy the waterproofing characteristics of Arquad 2HT.

## BEARING AND DURABILITY STUDIES

To further evaluate Arquad 2HT with the two soils on a basis which might be indicative of its field performance, bearing and durability tests were made.

*California Bearing Ratio*

CBR tests were performed on uncured, unsoaked samples at standard Proctor density, and on uncured, 4 day soaked samples, using U. S. Corps of Engineers procedures (2). Samples containing optimum amounts of Arquad 2HT were compared with raw soil samples.

Table 5 summarizes the CBR determinations. A definite improvement was observed in the soaked strengths of the Arquad-treated soils and swelling was cut in half. The treated soils gained strength during the soaking period, an indication of a benefit from moist curing that did not appear in the unconfined compression tests of the curing study.

*Iowa Bearing Value*

In some respects the Iowa bearing

TABLE 5  
EFFECT OF ARQUAD 2HT ON CALIFORNIA BEARING RATIO OF SOILS 20-2I AND 44A-1

|                               | 20-2I          |                |               |      | 44A-1 |     |               |      |
|-------------------------------|----------------|----------------|---------------|------|-------|-----|---------------|------|
|                               | Raw            |                | 3% Arquad 2HT |      | Raw   |     | 5% Arquad 2HT |      |
|                               | U <sup>a</sup> | S <sup>b</sup> | U             | S    | U     | S   | U             | S    |
| CBR at 0.1 in. penetration, % | 17.7           | 2.5            | 18.5          | 25.6 | 4.5   | 2.0 | 5.0           | 10.0 |
| CBR at 0.2 in. penetration, % | 28.2           | 5.2            | 30.0          | 38.4 | 6.0   | 2.8 | 7.2           | 13.3 |
| Swell, % of original height   | —              | 0.5            | —             | 0.2  | —     | 0.8 | —             | 0.4  |

<sup>a</sup> Unsoaked condition.

<sup>b</sup> Soaked for 4 days.

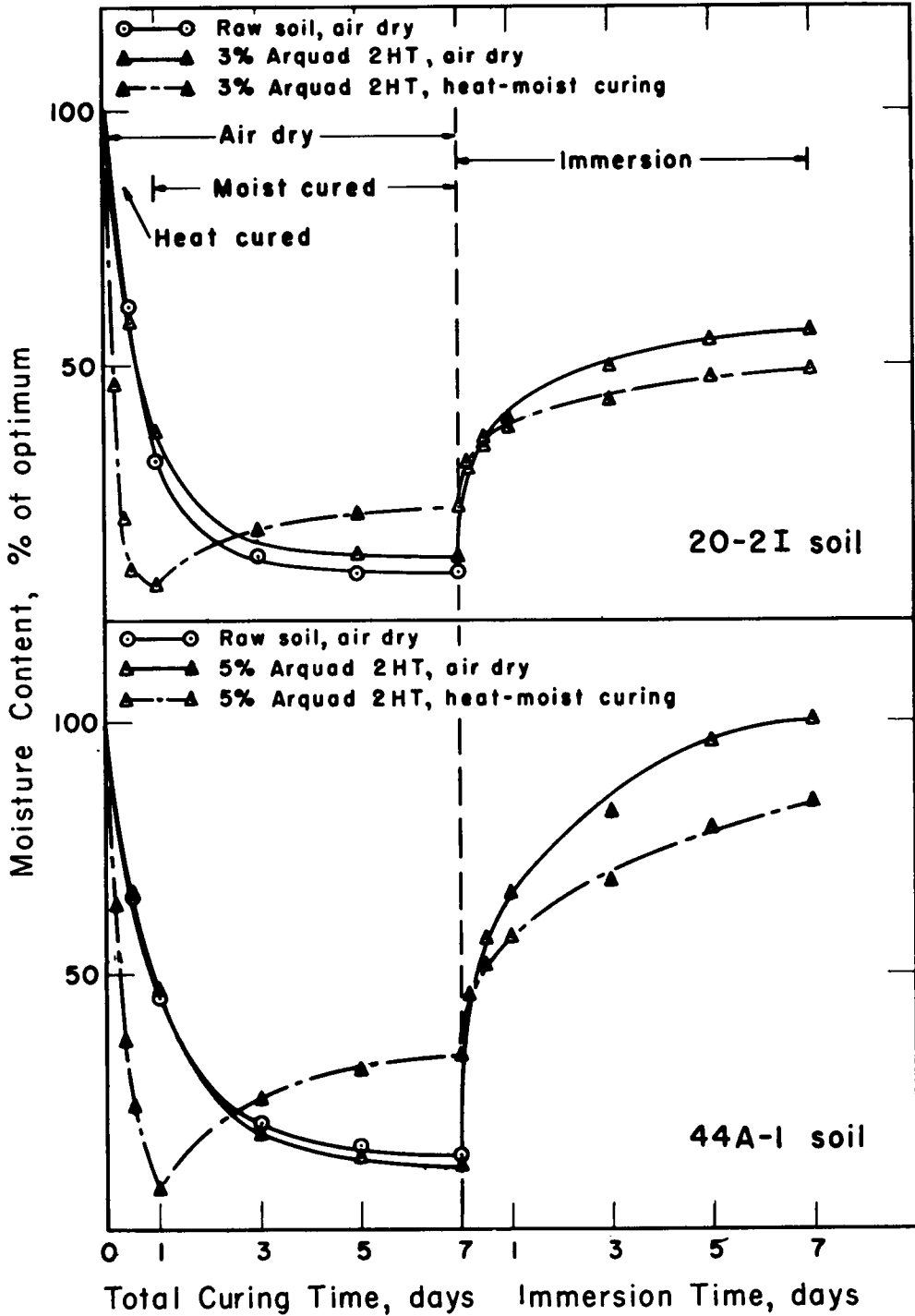


Figure 5. Moisture-curing time curves for raw and stabilized soils.

TABLE 6  
EFFECT OF ARQUAD 2HT ON IOWA BEARING VALUE OF SOILS 20-2I AND 44A-1

| Length of air curing, days | Specimen Condition | Soil 20-2I                 |                        |              |          | Soil 44A-1                 |                        |              |          |
|----------------------------|--------------------|----------------------------|------------------------|--------------|----------|----------------------------|------------------------|--------------|----------|
|                            |                    | Concentration Arquad 2HT % | Iowa Bearing Value, lb |              | Swell, % | Concentration Arquad 2HT % | Iowa Bearing Value, lb |              | Swell, % |
|                            |                    |                            | 0.1 in. pen.           | 0.2 in. pen. |          |                            | 0.1 in. pen.           | 0.2 in. pen. |          |
| 0                          | U <sup>a</sup>     | 0                          | 133                    | —            | 0        | 33                         | 46                     | 1.8          |          |
| 0                          | S <sup>b</sup>     | 0                          | 62                     | 0.8          | 0        | 19                         | 22                     | —            |          |
| 0                          | U                  | 3                          | 155                    | —            | 5        | 72                         | 96                     | —            |          |
| 0                          | S                  | 3                          | 100                    | 0.4          | 5        | 51                         | 56                     | —            |          |
| 3                          | U                  | 3                          | 312                    | —            | 5        | 185                        | 244                    | —            |          |
| 3                          | S                  | 3                          | 208                    | 0            | 5        | 81                         | 129                    | —            |          |
| 7                          | U                  | 3                          | 424                    | —            | 5        | 310                        | 574                    | —            |          |
| 7                          | S                  | 3                          | 227                    | 0            | 5        | 135                        | 216                    | 0.9          |          |

<sup>a</sup> Unsoaked, in the desired curing condition.

<sup>b</sup> Soaked for 48 hours following the desired length of curing.

value test is a miniature CBR test. It is under development at the Iowa Engineering Experiment Station and is being correlated with CBR.

*Preparation and testing of IBV specimens.* Cylindrical specimens (2-in. diameter by 2 in. high) molded to near standard Proctor density in 2-in. diameter by 5 in. high brass molds are used. The specimens are retained in the molds during curing and testing. A 5/8-in. diameter rod is penetrated into the specimen at 0.05 in. per minute. The load readings, in pounds, at penetrations of 0.1 and 0.2 in. were taken as relative measures of the bearing strength of the soil.

For each soil, specimens were molded, air cured, and tested in the cured, unsoaked, and cured, 48 hours immersed conditions. Air curing was for 0, 3, and 7 days following molding. During immersion, 500 g surcharges were kept on the specimens and measurements of swell were made. Specimens of raw soil were compared with specimens containing optimum amounts of Arquad 2HT.

*Results.* Test results are shown in Table 6. Air curing of 3 and 7 days prior to 48 hours immersion was extremely beneficial. Both the unsoaked and the immersed strengths of treated specimens increased approximately 130 percent at 3 days air curing, and 400 percent at 7 days air curing, again indicating the essential role of curing in the effectiveness of soil-organic cationic chemical stabilization. The beneficial effect of the chemical treatment is also seen in the reduction of swelling; 3 days air drying, in the case of the silty soil, caused the swell to decrease to the point where it was immeasurable.

#### Durability Tests

*Method of test.* The procedure used for evaluating the resistance of Arquad 2HT treated soils to cycles of freezing and thawing and wetting and drying was as follows:

1. Two-inch diameter by 2-inch high specimens were molded to near standard Proctor density using water concentra-

tions of Arquad 2HT previously determined as being optimum for each soil.

2. Specimens were air cured at room temperature for 7 days. After curing, the procedure for freeze-thaw evaluation was:

- a. Immerse specimens in distilled water for 2 hours.
- b. Remove specimens from water bath and place on soaked felt pads to maintain capillary absorption.
- c. With specimens on pads, place in deep freeze at  $-10$  deg F for 23 hours.
- d. Thaw in open air at room temperature for 2 hours.
- e. Place thawed specimens in a moisture cabinet at approximately 70 deg F and 95 percent relative humidity for 23 hours. This constituted the end of the first cycle. Each succeeding cycle was a repetition of steps c through e.

Three specimens of each soil were tested for unconfined compressive strength following 1, 3, 5, 7, 10, and 12 cycles of freezing and thawing.

After curing, the procedure for wet-dry evaluation was:

- a. Immerse specimens in distilled water for 24 hours. This ended the first cycle.
- b. Air dry specimens at room temperature for 24 hours.
- c. Immerse specimens in distilled

water for 24 hours. This ended the second cycle. Each succeeding cycle was a repetition of steps b and c.

Three specimens of each soil were tested for unconfined compressive strength following 1, 3, 5, 7, 10, and 12 cycles of wetting and drying.

*Results.* In addition to strength determinations, a visual examination of the specimens was made after each cycle. Following the sixth cycle of freezing and thawing, surface foliations appeared and steadily increased with each succeeding cycle, while vertical cracks began to appear after the sixth cycle of wetting and drying, though all specimens maintained their original shapes.

Freezing and thawing was much more detrimental to the Arquad-treated specimens than wetting and drying (Table 7). Each cycle of either test caused a decrease in the unconfined compressive strengths, with the exception of wet-dry cycles with the silty soil; its strength steadily increased up to 10 cycles but began decreasing slightly at 12 cycles.

#### DIFFERENTIAL THERMAL ANALYSIS STUDY

##### *Preparation of Samples*

Samples of soil 20-2I were prepared at optimum moisture content with a high percentage water concentration of Arquad 2HT. An equal amount of the chemical dispersion was also added to a sam-

TABLE 7  
EFFECT OF FREEZE-THAW AND WET-DRY CYCLES ON UNCONFINED COMPRESSIVE STRENGTH OF ARQUAD 2HT-TREATED SOIL SPECIMENS

| No. of Cycles | Unconfined Compressive Strength, lb |                                |                                |                                |
|---------------|-------------------------------------|--------------------------------|--------------------------------|--------------------------------|
|               | Freeze-thaw                         |                                | Wet-Dry                        |                                |
|               | Soil 20-2I, 3% Conc. Arquad 2HT     | Soil 44A-1 5% Conc. Arquad 2HT | Soil 20-2I 3% Conc. Arquad 2HT | Soil 44A-1 5% Conc. Arquad 2HT |
| 1             | 200                                 | 510                            | 190                            | 300                            |
| 3             | 200                                 | 305                            | 178                            | 169                            |
| 5             | 128                                 | 182                            | 207                            | 195                            |
| 7             | 90                                  | 52                             | 220                            | 150                            |
| 10            | 60                                  | 63                             | 235                            | 135                            |
| 12            | 60                                  | 60                             | 230                            | 130                            |

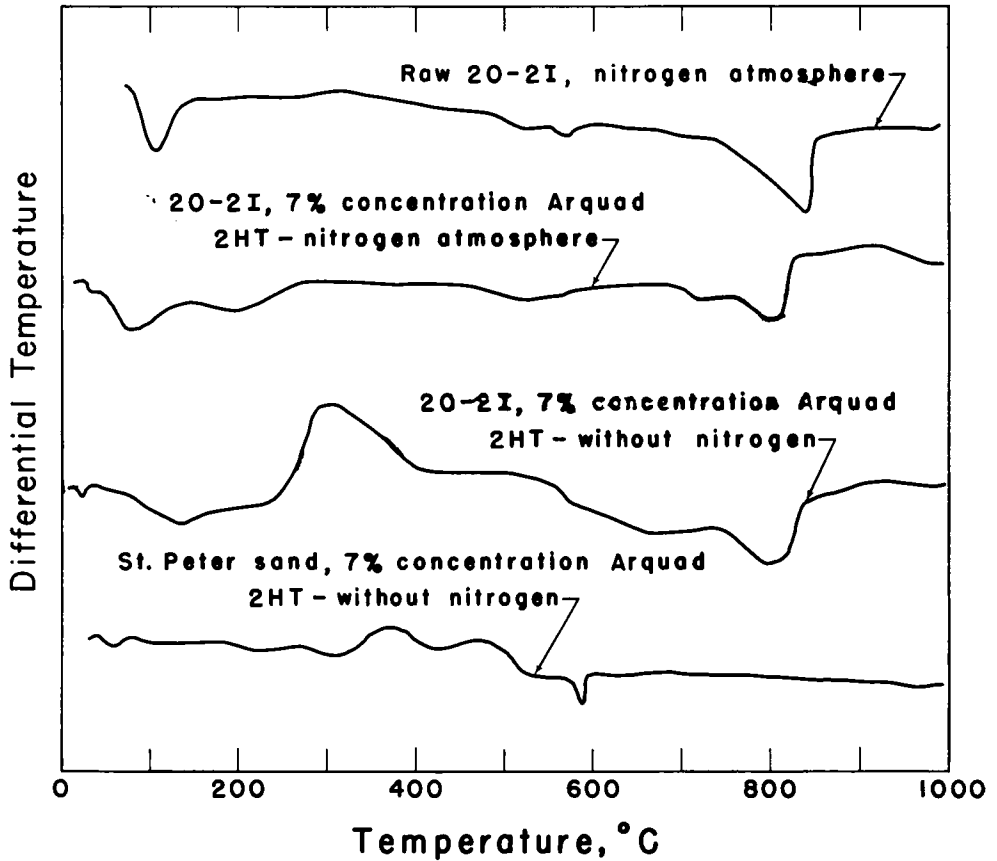


Figure 6. Differential thermal curves of samples containing a quaternary ammonium salt.

ple of St. Peter sand. After mixing, all samples were allowed to come to equilibrium in an atmosphere controlled at 50 percent relative humidity for two weeks prior to testing.

### Results

Differential thermal analysis has been shown to be a useful tool for study of clay-organic chemical complexes in soils (13). A sample is heated at a linear rate and the thermal reactions in the sample are recorded. In Figure 6 the initial downward peak at about 100 deg C indicates the endothermic reaction of adsorbed water being driven from the clay. The next two curves show a reduction in size of this peak due to treatment by Ar-

quad 2HT. In samples run in an oxidizing atmosphere (third and fourth curves), the organic material oxidizes in a drawn-out exothermic reaction between 250 and 550 deg C. The reaction with sand shows a definite double peak.

A significant observation from thermal analysis study of Arquad 2HT is the lack of any delayed exotherm related to breakdown of the clay at around 600 deg C. Treatment of soils or clays with some organic chemicals results in a delayed exotherm believed caused by carbon adsorption on the clay (1). When the clay structure breaks down, the carbon is released and burned. These reactions have been observed with the loess samples studied. If the interpretation is correct, the breakdown of Arquad does not leave

any such residual carbon attached to the clay.

A second factor in thermal breakdown of Arquad-treated soil is the effect on the breakdown of calcium carbonate in soil. Thermal analysis of the natural soil gives a large, sharp endothermic peak starting at about 700 deg C and continuing to about 850 deg C. In thermal analysis of Arquad-treated soils the carbonate reaction assumes a lower temperature, more rounded endotherm. At first it was believed that the Arquad might be acidic and react with the carbonates on mixing with soil. However, pH checks by titration and a measurement of CO<sub>2</sub> gas release from treated soil failed to verify this. Perhaps one of the high-temperature breakdown products from Arquad may be absorbed on and catalyze the thermal decomposition of the soil carbonates.

In summary, differential thermal analysis offers additional evidence for waterproofing of soil organic cationic chemicals. Organic cations are preferentially and irreversibly adsorbed and are substituted for water on clay, but the bond is not strong enough to retard later burning of the organic chemicals. Other observed high-temperature thermal reactions appear to be related to thermal disintegration of the organic chemicals and may have no direct bearing on soil stabilization.

#### THEORETICAL INTERPRETATIONS

The resistance of soils to external forces is due to friction between the solid particles and the cohesion furnished by films of moisture covering these particles. When such a film thickens it performs as a lubricant, reducing the friction between adjacent particles and consequently diminishing the resistance value.

The clay-size material of both soils used in this work was predominantly montmorillonitic. Petrographic studies by Handy (9) show that the clay occurs mainly as coatings on larger soil grains; in the more clayey soil, clay also occurs as separate aggregates. Montmorillonitic minerals are among the most active com-

monly found in soil clays. Particles of montmorillonite are made up of flake-like crystal units composed of two silica sheets with an alumina sheet tenaciously held together by mutually shared oxygen atoms. The 3-layer structural units themselves, however, are so loosely held together that the crystal lattice is often bellows-like and expands and absorbs water readily. The strength of this bond and the amount of expansion and hydration depend to a large extent on the strength of the kind of cation present. The natural cation in the two soils used is calcium, which gives an aggregated, moderately hydrating and expanding clay. Complete substitution of organic cations for calcium would reduce the expansion and hydration to zero. Part of this effect is due to the water-repellency of the cationic Arquad 2HT once its charge is neutralized on the negative clay surface.

Figure 7 illustrates the waterproofing effects of Arquad 2HT on the two soils. Stirring of properly treated soil in water does not result in a mud, but in a mass of water-stable aggregates as in the lower photograph (Figure 7).

Based on experimental evidence, the possible mechanism of Arquad 2HT as a soil stabilizer may be summarized as follows:

#### *Cation Exchange and Ease of Mixing*

1. Mixing of water with soil is greatly facilitated if an agent is added to lower the surface tension of the water. Arquad 2HT is such an agent.

2. During or after mixing a cation exchange reaction takes place between the clay particles of the soil and Arquad 2HT. The cation exchange has the following effects:

- (a) Large organic cations tend to flocculate clay by reducing the clay surface charge. Flocculated clay has a lowered ability to take up water. It is also easier to mix.
- (b) In the amounts used, Arquad 2HT cations also partially coat the clay surfaces with a moisture repellent film. Such coated clay surfaces have little or no affinity for water.

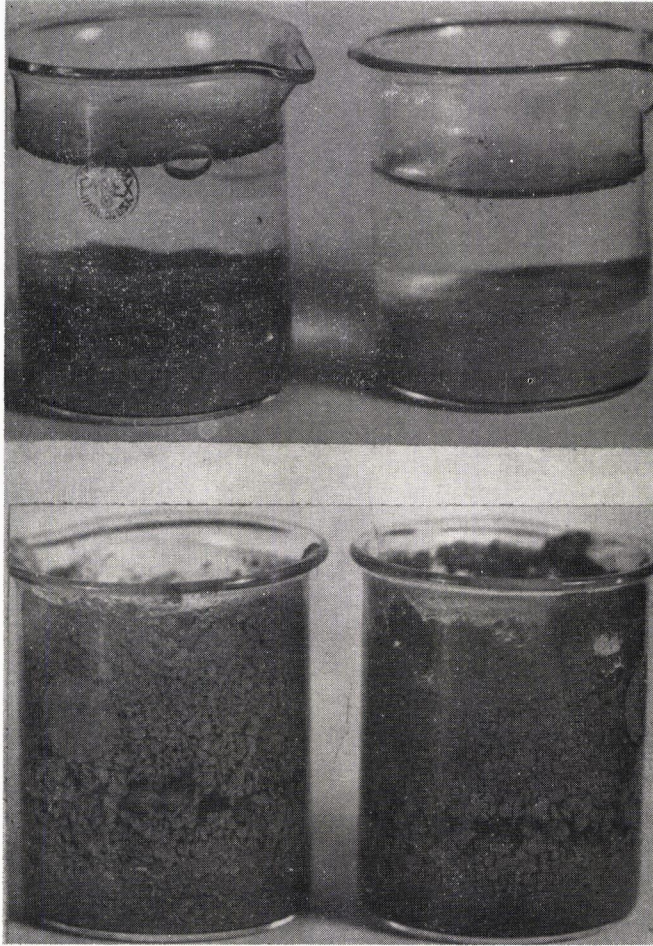


Figure 7. Aggregating effect of Arquad 2HT on silty (left) and clayey (right) loess. Above, untreated loess in water; below, optimum treated loess stirred in water.

### *Strength*

1. The strength of soil aggregates depends in part on the structure and thickness of moisture films binding the particles. Usually the thinner the film the better the binding. For example, the strength of natural soil aggregates is usually greatly increased by air or oven drying because of the decrease in thickness of water films. Immersing such a soil in water usually results in thickening of the water films, destruction of the aggregates and reduction of the soil to a mud.

2. The water-stability of aggregated soil is greatly increased by waterproofing actions of Arquad 2HT. Figures 8 and 9 show the untreated soil dispersed in water and the water-stable aggregates of treated soil.

3. However, because of the partial coating and waterproofing of clay particles by Arquad 2HT, binding by water films is decreased, and the strength of the air dry soil is decreased. Therefore, Arquad 2HT treatment of soil causes an increase in wet strength and a decrease in dry strength. It follows that in general the greater the amount of Arquad

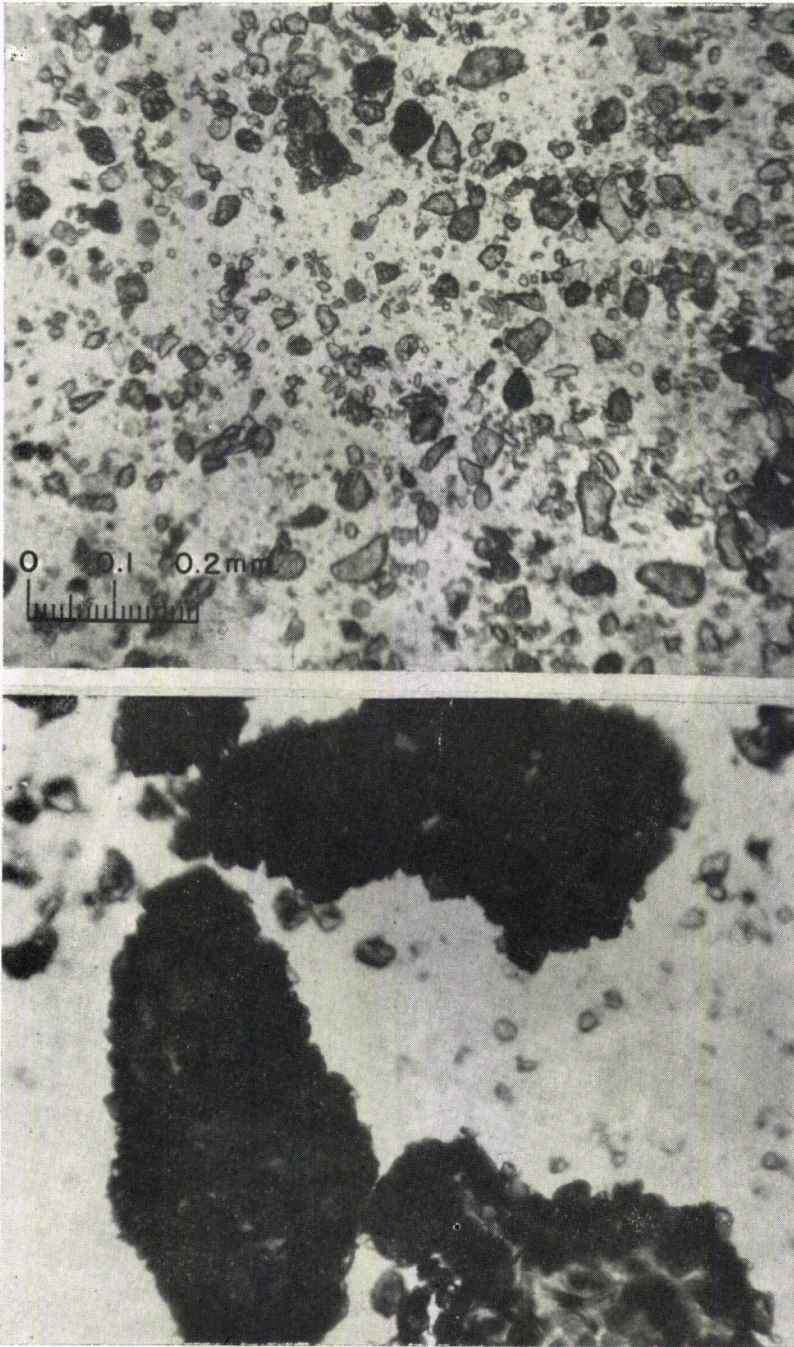


Figure 8. Photomicrograph of 20-2I soil. Above, raw soil; below, air dry stabilized soil.



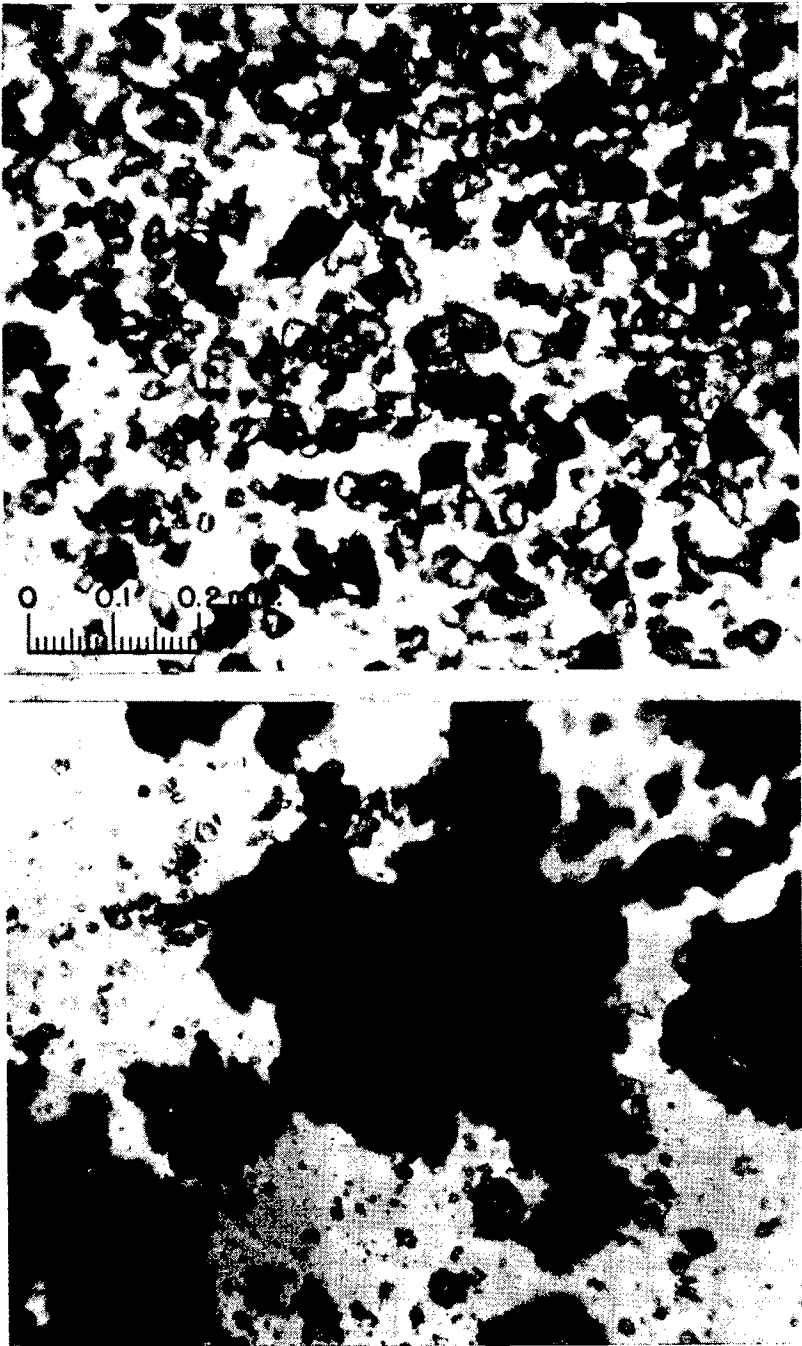


Figure 9. Photomicrograph of 44A-1 soil. Above, raw soil; below, air dry stabilized soil.

2HT used the lower the air dry strength.

4. A certain minimum amount of Arquad 2HT is necessary for effective waterproofing. This amount is often an optimum; if Arquad 2HT is used in excess of this amount the strength of the water-stable aggregates may be decreased for the reason stated.

#### CONCLUSIONS

Immersed compressive strengths of Arquad 2HT treated soils depend to a large degree on the curing treatment. These variations suggest a change in the disposition of the soil water and of the Arquad 2HT ions. The relations are as follows:

1. During air drying, treated soils lose water and gain strength (Tables 2, 3, Figure 5) at about the same rate as untreated soils.

2. The gain in strength of Arquad 2HT treated soils on drying is probably due to reduction in thickness of the moisture films. Curing in a humid atmosphere gives a lower strength gain.

3. Heat treatments show that an optimum degree of drying exists. Overdrying, which sometimes occurs for natural soils in hot climates and is referred to as dessication, causes such a decrease in water and an increase in air voids that sudden rewetting causes compression of the air, disruption of the specimen (Figure 2), and loss of strength. With Arquad 2HT treated soils this was observed after oven-drying at 140 deg F and 160 deg F. However, wrapping of oven-cured specimens to prevent drying gives strength results approximating those obtained by moist curing at room temperatures.

4. Treated soils show a satisfactory recovery from oven-drying if they are subsequently aged in a humid atmosphere before immersion and testing. These moist cured strengths are much higher than those obtained without preliminary drying; therefore, drying is instrumental in obtaining a better arrangement of water films.

The effects of Arquad 2HT treatment

on engineering properties are as follows:

1. Arquad 2HT reduces the plasticity index of soils probably by flocculating and partially waterproofing the clay. Preliminary oven drying increases this effect.

2. Arquad 2HT treatment, like lime, results in flocculation of the clays in a silty soil, giving a poorer gradation and lower compacted densities with the same compactive effort. The optimum moisture content is not greatly affected.

3. California bearing ratio tests show a substantial increase in soaked bearing strength after additions of Arquad 2HT. Iowa bearing value tests run on treated soils show further very large increases in both dry and soaked bearing strengths after air curing.

4. Data from unconfined compressive strength tests show that Arquad 2HT treatments decrease the cohesion in air-dry soil. Data from the bearing tests indicate that Arquad 2HT treatments may increase the angle of internal friction.

5. Freeze-thaw tests and wet-dry tests on air-cured Arquad 2HT treated specimens show a considerable resistance to physical weathering. The resistance of untreated soils is zero.

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## REFERENCES

1. ALLAWAY, W. H., "Differential thermal analysis of clay treated with organic cations as an aid in the study of soil colloids." *Soil Sci. Soc. Am. Proc.* 13: 183-188. 1948.
2. American Society for Testing Materials. Procedures for testing soils. July, 1950.
3. Arquads. Chemical Division, Armour and Company. Chicago. July, 1953.
4. Chemical division, General Mills. Kankakee, Illinois.
5. CHU, T. Y., DAVIDSON, D. T., GOECKER, W. L., AND MOH, Z. C., "Soil stabilization with lime-fly ash mixtures: Preliminary studies with silty and clayey soils." *Bulletin 108*: 102-112. Highway Research Board. 1955.
6. DAVIDSON, D. T., "Exploratory evaluation of some organic cations as soil stabilizing agents." *Highway Research Board Proceedings*. 1949.
7. ——— AND GLAB, JOHN E., "An organic compound as a stabilizing agent for two soil-aggregate mixtures." *Highway Research Board Proceedings*. 1949.
8. GIESEKING, J. E., "Exchange adsorption by clays of large organic cations." *Bulletin 108*. Highway Research Board. 1955.
9. HANDY, R. L., LYON, C. A., AND DAVIDSON, D. T., "Comparisons of Petrographic and engineering properties of loess in southwest, east-central, and northeast Iowa." *Proceedings of Iowa Academy of Science*, vol. 62, p. 279. December, 1955.
10. HENDERSON, DONALD H., "Practical methods of applying large organic cations as soil waterproofing agents." Unpublished M.S. thesis. Iowa State College Library. Ames, Iowa. 1949.
11. HENDRICKS, S. B., "Base exchange of the clay mineral montmorillonite for organic cations and its dependence upon adsorption due to Van der Waal's forces." *J. Phys. Chem.* 45: 65-81. 1941.
12. HOOVER, JAMES M., AND DAVIDSON, D. T., "Organic cationic chemicals as stabilizing agents for Iowa loess." *Bulletin 129*: 10-25. Highway Research Board. 1956.
13. SHEELER, J. B., HANDY, R. L., AND DAVIDSON, D. T., "Effects of a synthetic resin on differential thermal analysis of loess." Presented at the 5th National Clay Conference, Univ. of Illinois. 1956. (to be published).
14. WHITESELL, C. H., JR., "Effect of organic cations on the compaction of clay soils." Unpublished M.S. Thesis. Iowa State College Library. Ames, Iowa. 1948.