EFFECT OF SOIL SURFACE AREA AND EXTRACTABLE SILICA, ALUMINA, AND IRON ON LIME STABILIZATION CHARACTERISTICS OF ILLINOIS SOILS

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> Several years ago, a group of about 40 Illinois soils was collected by M. R. Thompson of the University of Illinois. Thompson determined the lime reactivity of the soils and also subjected the samples to a number of routine analytical procedures in an attempt to determine if lime reactivity is closely related to any particular engineering, mineralogical, or physicochemical property of the soil. The writers sought to extend Thompson's analytical work to include determination of several other soil properties not included in the original work. Specific properties investigated included soil surface area, amount of finely divided iron present in the soil, and amount of silica and alumina entering an aqueous solution maintained at the pH level of a saturated solution of lime in water. Work was done by using Thompson's original soil samples, and results obtained by the writers were compared with his determination of lime reactivity of the soils. It was found that lime reactivity does not appear to be very closely correlated with soil surface area and even less so with the availability of dissolved silica or alumina or both. However, it was found that lime reactivity does appear to be significantly inhibited by the presence of finely divided iron intimately distributed through the soil mass, a condition that is typical of many better drained soils in Illinois.

• CONSIDERABLE INTEREST has arisen in recent years in the improvement of the engineering properties of natural soil materials through lime treatment. Several years ago, a representative group of Illinois soils was collected by M. R. Thompson of the University of Illinois. Thompson subjected these soils to lime treatment in the laboratory and determined the effect of treatment on unconfined compressive strength. The soils were all essentially surface materials and included samples of A-, B-, and C-horizons of some 14 pedological series. Some deeper occurring parent materials (loesses and glacial tills) were also included in the study.

Soils were treated with varying percentages of lime (3, 5, and 7 percent), compacted at optimum water content by a procedure similar to the standard AASHO specification, and cured under moist conditions for 28 and 56 days. Specimens were then tested in unconfined compression, and the results were compared with strengths of untreated but similarly processed specimens of the same materials.

An attempt was made by Thompson to relate the effect of lime treatment on soil strength with a number of soil properties that were routinely determined for all the soils that he used. Determined engineering properties included grain-size distribution and Atterberg limits. Mineralogical properties included determination of predominant clay minerals present and determination of the CaCO₃ equivalent for calcareous

materials. Physicochemical properties included pH, percentage of organic carbon present, cation exchange capacity, total exchangeable bases, and specific amounts of Ca, Mg, Na, and K. It was found that organic carbon content exerted a significant adverse effect on lime reactivity. A significant positive correlation appeared to exist between lime reactivity and soil pH, percentage of base saturation of the soil, and presence of mixed-layer minerals in the clay fraction.

The authors became interested during the summer of 1969 in possible physicochemical explanations for lime reactivity. Thompson provided samples of 24 of the soils

used in his study (Table 1).

It has been concluded by several investigators, including Thompson and Eades (3, 8), that the strength increase accompanying lime treatment of many soils results from a pozzolanic reaction. This would imply that the lime releases free silica or alumina or both from the minerals present in the soil and then reacts with the released material to form crystalline calcium silicates and aluminates that cement individual soil particles together. With this in mind, the authors concluded that lime reactivity of a soil should be strongly correlated with the amount of silica and alumina found to be present in a slurry made from a particular soil in which the pH is maintained at 12.3 for a given time. This is the pH presented by a saturated lime-water solution. It was also thought that high soil surface area should enhance lime reactivity because it would appear that

TABLE 1
LIME REACTIVITY AND CHEMICAL PROPERTIES OF 24 ILLINOIS SOILS

Soil Type	Horizon	Drainage Classifi- cation	Lime Re- activity (psi)	Percent Increase in STR With Lime	Surface Area (m²/gm)	Extractable Fe ₂ O ₃ (percent)	Extractable Al ₂ O ₃ (percent)	Extractable SiO ₂ (percent)
Bryce silty clay	В	Poor	131	162	178	1.47	0.21	0.57
Cisne silt loam	В	Poor	97	104	159	1.35	0.54	0.42
Cowden silt loam	В	Poor	43	74	191	1.79	0.30	0.37
Cowden silt loam	C	Poor	90	134	117	1.56	0.17	0.73
Cowden silt loam Drummer silty	CMC	Poor	90	134	104	1.64	0.17	0,32
clay loam	В	Poor	118	173	171	1.04	0.19	0.23
Elliott silt loam	В	Moderately well to imperfect	12	12	159	2.60	0.28	0.33
Fayette silt loam	В	Well	44	63	144	2.14	0.27	0.41
Fayette silt loam	C	Well	145	363	74	1.24	0.21	0.43
Hosmer silt loam	B_2	Well to moderately well	60	95	136	1.83	0.36	0.36
Hosmer silt loam	В'2	Well to moderately well	37	42	174	1.91	0.27	0,45
Huey silt loam	В	Imperfect	131	128	134	1.43	0.17	0.33
Huey silt loam	C	Imperfect	133	149	124	1.74	0.14	0.45
Miami silt loam	В	Well	21	26	76	1.64	0.30	0.34
Miami silt loam Sable silty clay	С	Well	98	134	82	1.58	0.08	0,23
loam	В	Poor	127	130	187	0.92	0.17	0.21
Tama silt loam	В	Well to moderately well	24	32	191	1.37	0,26	0.35
Wisconsinan clay till Wisconsinan loam			89	114	170	1.31	0.18	0.50
till			79	75	48	1.22	0.11	0.41
Illinoian till Peorian loess.			135	265	47	1.43	0.07	0.39
calcareous Peorian loess,			76	345	54	1.02	0.09	0.46
leached			24	72	72	1.52	0.17	0.31
Buried Illinoian profile Accretion gley	В	Poor	93	216	75	0.73	0.08	0.40
profile	G	Very Poor	230	328	100	0.24	0.35	0.86

Note: Drainage classification and lime reactivity taken from Thompson (8, Tables 3 and 8). Surface area and extractable Fe 2O3, Al 2O3, and SiO2 were determined by authors using Thompson's samples.

more silica and alumina would be released in a given treatment under such conditions. It was also thought that a correlation between the presence of iron oxide and lime reactivity might exist because several investigators (4, 10) have proposed that in well-drained soils iron oxide takes the form of tiny positively charged molecular aggregations that adhere to the surface of clay minerals rendering them less vulnerable to attack by materials present in the surrounding soil solution.

METHODS AND PROCEDURES

Routine tests performed on the 24 samples included the following:

- 1. Soils were treated in a sodium hydroxide solution in which the pH was adjusted to 12.3. Two grams of soil were placed in 20 ml of solution. The resulting slurries were placed in 4-oz polyethylene bottles and shaken continuously for 3 days. Following this, the slurries were centrifuged, and the resulting clear solutions were subjected to analysis for silica and alumina. The method proposed by Ingamells was used in silica analysis (6). Alumina content was determined by the aluminon lake procedure discussed in several publications (5, 6). The writers experienced some difficulty with this procedure, and some comment regarding the analysis should probably be made at this point. Best results were obtained by adding 3 ml of 1N HCl to a 1-ml aliquot of solution. This acidulated solution was then heated for 1 hour at 80 C to ensure that all aluminum present would be in the form of Al+++ ions without associated hydroxyl ions. The solution was then built to 50 ml by the addition of a buffer solution presenting a pH of 4.2 prepared from acetic acid and sodium acetate. Five milliliters of aluminon solution, also buffered at a pH of 4.2, were then added. The solution was then shaken, and the aluminum content was determined colorimetrically by using a Coleman Junior spectrophotometer 20 min after the addition of the aluminon solution. This time period did not vary by more than 1 min and yielded good results for a number of standard solutions of various concentrations. It was found that the solution tends to darken and become turbid if allowed to stand for a longer period of time before the colorimetric determination.
- 2. Surface area of the soils was determined. Ethylene glycol monoethyl ether was used instead of the more traditional ethylene glycol. It has been demonstrated that the ether provides good results with a considerably shorter time requirement for equilibration (2).
- 3. Iron was extracted from the samples by using sodium citrate and sodium dithionate in a process described by Aguilera and Jackson (1). Iron dissolved from the soil was determined colorimetrically by the thiocyanate method. This iron-removal procedure results in the solution of the finely divided iron oxy-hydroxides that may be present as coatings on clay particles. However, concretionary iron and iron ions that may have proxied for other ions in the mineral structure of the clays are not attacked and dissolved.

RESULTS

Somewhat to the surprise of the authors, it was found that only a fair correlation exists between surface areas of the soils studied and the response of the soils to lime stabilization treatment. Perhaps even more surprising was the fact that this correlation is an inverse one: Soils presenting a high surface area exhibited poorer response to treatment. A possible explanation may be drawn from Eades' work (3) in which it was found that montmorillonitic soils appear to require the addition of a certain percentage of lime before strengthening effects are noted and that response occurs with only slight additions of lime to soils containing other clay minerals. However, in Thompson's study (8) many of the soils presenting high surface areas, notably those of the Cowden, Bryce, Tama, and Sable series, responded best to a 5 percent lime treatment. Strengths obtained by using either a 3 or 7 percent treatment were lower.

At this point it may be well to note that, in most Illinois soils, the clay fraction is predominantly made up of 3-layer minerals: illite in the case of glacial tills and montmorillonite in the case of loessial soils. Strictly local conditions may alter the pattern

just described. A locally acid environment appears to result in the release of aluminum from the 3-layer, clay-mineral structure and the subsequent formation of chlorite or mixed-layer clays (4).

A considerable variation in content of extractable silica and alumina was found to exist in the group of soils analyzed. However, as Figure 1 shows, correlation of these properties with strength increase resulting from lime treatment does not appear to be highly significant. The inverse effect of alumina was particularly surprising. Eades (3) has suggested that bonds resulting from the formation of calcium aluminate cementing agents develop in a relatively short time but that such bonds are ultimately much weaker than those developed by the more slowly formed calcium silicate cements. A comparison of Thompson's 28- and 56-day strengths of lime-treated soils in which extractable alumina is notably more abundant than extractable silica, or vice versa, does not appear conclusive with regard to this concept.

It was found that a rather significant inverse correlation appears to exist between the extractable iron content of a soil and its response to lime stabilization treatment as represented by the percentage increase in unconfined compressive strength.

The primary mineral sources of iron in Illinois soils are oxides (principally magnetite and ilmenite) and silicates (including amphiboles and pyroxenes). Fragments of these minerals have been incorporated into the several glacial tills and loesses that cover most of the state. Also present is some iron that is substituted for aluminum in

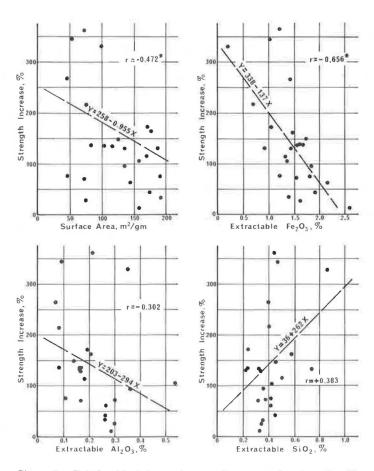


Figure 1. Relationship between increase in compressive strength with lime treatment and several physicochemical properties of 24 Illinois soils.

the crystalline structure of clay minerals. The primary iron-bearing minerals are subjected to chemical weathering processes in which soil microorganisms play an important role. Chemical change and redistribution of the iron within the soil mass occur. The nature of the redistribution is largely dependent on soil drainage conditions. Poorly organized oxy-hydroxides and goethite are the principal secondary minerals formed.

In poorly drained soils, the pH and oxidation potential of the environment have been such that much of the iron has been reduced to the Fe⁺⁺ valence state. In this state the iron may be dissolved by the soil water and, hence, is quite mobile. Whereas some of the iron is no doubt leached from the soil, most of it moves a short distance within the soil mass to a nearby location where the microenvironment is favorable to oxidation and, hence, precipitation with formation of concretions. Eventually, most of the iron present in the soil becomes concentrated in these locations, and the bulk of the soil mass will contain very little iron.

In better drained soils, iron is moved through microscopically small distances by chemical and microbial weathering agents. However, the pH and oxidation potential of the environment do not favor the reduction of iron, and the iron present does not experience a period during which macroscopic relocation occurs. Thus, secondary iron minerals in various states of hydration occur in microaggregations, perhaps containing only a few molecules each. These aggregations usually present a net positive surface charge and, thus, are attracted to clay mineral particles that usually bear a negative surface charge. In the well-drained soils, weathering tends to result in an intimate scattering of iron throughout the bulk of the soil mass. It may be seen that the clay fraction of a well-drained soil may lose such properties as cation exchange capacity as clay particles become permanently plated by insoluble and chemically stable iron aggregations.

The contrast in iron distribution between poorly drained and well-drained soils is reflected in soil color. The subsoil of a typical well-drained profile is usually of a uniform yellow-brown color reflecting the intimate scattering of iron throughout the soil mass. Imperfectly drained soils may present a variegated gray and yellow mottled appearance whereas, in even more poorly drained soils, the soil mass is of a more or less uniform gray color and contains small hard concretions of dark reddish-brown limonitic material as well as limonitic linings along root or worm holes. The gray color is indicative of reduced presence of iron throughout the bulk of the soil mass.

It has been observed that the total iron content in most Illinois soils as may be found by fusing or dissolving a sample is relatively constant from place to place and usually totals about 4 to 5 percent Fe_2O_3 . It would appear from the foregoing discussion, however, that the form in which the iron exists in the soil is of more concern than the total amount. The iron extraction method of Aguilera and Jackson (1) is well suited to this end in that iron contained in the small aggregations of a well-drained soil is dissolved whereas the concretionary iron of poorly drained soils is not attacked to any great extent. Thus the "extractable iron" found by this method is primarily iron that exists in the form of coatings on individual clay mineral particles. Determination of the magnetic susceptibility of a soil also provides a measure of the amount of iron present in the form of small aggregations and clay particle coatings because this iron is generally found in the form of a magnetic oxide. Limonitic or concretionary iron is predominantly goethite, which is far less magnetic (7).

From the fairly significant inverse correlation between extractable iron content of the soils analyzed and increase in strength of the soils with lime treatment, it would appear that successful lime stabilization is favored by the absence of finely divided iron oxy-hydroxides intimately associated with the clay fraction of the soil. Thus, it would be expected that poorly drained soils will exhibit greater lime reactivity than well-drained soils. This conclusion must, of course, be tempered by the realization that the poorly drained soils frequently contain higher percentages of organic carbon that has been shown to inhibit cementing action.

The rather poor correlations between lime reactivity and soil surface area and the availability of soluble silica and alumina in the soil would indicate that these properties are apparently not as important in the chemistry of lime stabilization treatment as the authors once thought they were.

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