Effect of Heat on Physico-Chemical Properties of Soils

M. C. LI, Associate Professor of Civil Engineering, New York University

This paper is based on a laboratory investigation of 36 soil samples prepared from 9 various clay minerals and their modifications as a result of breakdown of chemical constituents and changes in physico-chemical properties by heat treatment at three different temperatures. The multiple correlation drawn between the independent variables of plasticity index, particle-size less than 2 μ, cation exchange capacity, and sodium and the dependent variable, free-swelling, gives a coefficient of 0.974. If surface area is used instead of clay fractions less than 2 μ, the coefficient of multiple correlation is 0.976. Simple correlations among clay fractions less than 2 μ, surface area, and composite index are also analyzed.

• THE BEHAVIOR and action of soil are affected not only by the inherent physical, chemical, mineralogical, hydraulic, microbiological, and optical properties but also are governed by many environmental factors, such as water, pressure, weather, and geological formation. The interrelationships in this polydisperse and multicharacter soil system are so intricate that many of them are only partially understood and yet to be found by basic researches.

SOIL SAMPLES

To insure uniformity, homogeneity, and purity of clay fractions in soil samples under strict laboratory control, ten clay minerals were collected from various parts of the United States and Canada. A pilot clay mineral (No. 0) was heated to 400 and 800 C for 12 and 36 hr to determine the effect of prolonged heating on physico-chemical properties. There were only insignificant changes. Thirty-six soil samples were prepared by heat treatment of the nine clay minerals (Nos. 1 to 9) at three different temperatures (300, 600, and 900 C) for 24 hr in a laboratory furnace with electronic controller. Table 1 gives the clay minerals, their chief constituents and sources of supply.

EXPERIMENTAL PROCEDURES

Physical tests of soils were performed in accordance with ASTM and AASHO standard methods and procedures (1, 2). Surface areas were determined by the glycerol retention method with pretreatment developed by the Bureau of Public Roads (3, 4). Specimens, 1 in. in diameter and 2 in. in height, were molded by Iowa State University drop-hammer impact compaction machine modified and constructed by New York University. The specimens were cured in a constant humidity room for 1 wk and tested on unconfined compression apparatus for cohesive strength.

Cation exchange capacities and exchangeable cations (calcium, sodium, magnesium, and potassium) were tested on prepared leachates (5) by means of a flame attachment on a Beckman spectrophotometer (6). A Beckman Zeromatic pH meter (5) was used to determine pH values, exchangeable hydrogen, and total exchangeable metallic bases.

A simple free-swelling test procedure in which the soil is allowed to expand freely

Paper sponsored by Committee on Physico-Chemical Phenomena in Soils.

TABLE 1

CONSTITUENTS AND SOURCES OF CLAY MINERALS

No.	Type	Source	Chemical Constituents (%)									S.R.[a]	O.M.[b]
			SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	SO_3		
0	Kaolinite	Ga.	---	---	---	---	---	---	---	---	---	---	0
1	Na-Ca bentonite	Manitoba, Can.	57.9	19.9	6.1	---	1.8	4.2	---	---	3.2	4.13	15.2
2	Ca-bentonite	Miss.	58.8	18.4	5.9	0.9	3.3	3.3	0.5	0.6	0.1	4.50	4.9
3	Ca-bentonite	Miss.	56.8	20.3	8.4	0.9	1.4	3.1	0.3	0.7	0.2	3.74	15.2
4	Attapulgite	Ga.	---	---	---	---	---	---	---	---	---	---	3.8
5	Na-bentonite	Wyo.	61.9	21.1	3.9	0.2	1.2	2.4	2.6	0.4	0.6	4.45	15.3
6	Kaolinite	Fla.	46.5	37.9	0.8	0.2	0.1	0.2	0.3	0.2	---	2.05	19.5
7	Illite	Ill.	61.5	17.8	5.0	1.1	0.6	1.5	4.3		1.2	4.97	10.3
8	Kaolinite	Ill.	63.6	19.9	2.4	1.5	0.1	0.2	2.4		---	5.03	5.5
9	Pyrophyllite	---	---	---	---	---	---	---	---	---	---	----	3.2

[a]Silica-sesquioxide ratio.
[b]Organic matter as determined by decrease in liquid limit of oven-dried samples.

TABLE 2

SUMMARY OF TEST RESULTS[a]

No.[b]	Atterberg Limits (%)					Size and Surface Area			Cation Exchange (m.eq./100 gm)			Free Swelling (%)	Cohesion (psi)
	w_{100}	w_L	w_P	I_P	w_S	$< 2\mu$ (%)	C.I.	S.A. (%)	CEC	Ca	Na		
1 A	127.2	134.8	55.5	79.3	11.8	92.8	0.796	36.0	123.5	27.7	31.3	41.2	176
B	130.6	85.5	49.1	36.4	29.7	78.0	0.746	39.8	62.2	30.0	30.9	54.6	161
C	71.2	44.5	47.6	0	43.9	23.0	0.400	7.5	19.6	26.4	13.9	5.1	10
D	61.2	40.9	42.9	0	47.4	18.2	0.303	0.6	1.5	21.6	0.7	4.4	0
2 A	124.2	98.9	53.5	45.4	12.3	84.6	0.633	32.6	82.7	79.8	0.5	45.3	243
B	132.2	78.5	51.9	26.6	15.5	54.0	0.792	32.9	79.0	76.9	0.5	43.4	174
C	78.0	51.7	47.5	4.2	50.8	34.2	0.627	12.1	7.7	47.7	0.3	14.9	81
D	62.5	43.2	---	NP	41.1	25.4	0.600	7.6	5.8	14.3	0	7.1	0
3 A	122.5	155.0	45.0	110.0	4.2	100.0	0.781	31.3	59.0	55.0	0.6	50.2	330
B	118.5	117.0	45.0	72.0	0	100.0	0.879	29.8	47.4	24.6	0.4	62.5	118
C	60.4	45.5	42.4	3.1	40.2	23.0	0.482	7.4	15.5	6.9	0.1	7.2	3
D	---	---	---	NP	44.0	18.0	0.314	0.6	2.1	0.8	0	5.3	0
4 A	239.0	255.0	128.4	126.6	57.8	99.8	0.699	16.4	37.2	23.2	0.5	36.3	248
B	231.0	166.6	139.0	27.6	87.4	58.0	0.424	16.6	31.1	23.2	0.4	31.3	98
C	209.5	134.5	136.0	0	114.6	39.4	0.338	10.2	39.7	6.3	0.4	12.6	6
D	153.0	87.9	---	NP	91.8	34.0	0.331	0.8	8.4	6.1	0.1	12.1	2
5 A	273.0	623.0	45.1	577.9	0	100.0	0.781	35.1	75.8	22.2	63.4	239.0	281
B	310.5	592.0	44.7	547.3	0	100.0	0.482	32.5	73.6	23.6	66.4	271.0	109
C	194.0	146.2	47.1	99.1	10.9	62.0	0.608	26.4	60.3	23.6	57.0	156.4	49
D	---	---	---	NP	30.6	17.2	0.438	0	3.3	14.3	0.7	7.4	1
6 A	83.5	90.0	45.0	45.0	43.4	96.2	0.677	2.7	12.6	1.0	2.2	24.0	138
B	81.7	64.6	41.7	22.9	25.8	89.0	0.645	2.0	2.6	0.9	0.5	20.6	115
C	88.7	59.5	56.4	3.1	55.9	22.8	0.380	1.4	0	0.6	0.1	5.3	5
D	90.1	63.2	---	NP	59.0	21.2	0.364	1.2	0	0.5	0	4.5	0
7 A	56.1	58.1	22.2	35.9	13.8	65.8	0.745	7.9	38.4	13.7	1.8	21.2	83
B	56.0	47.2	27.1	20.1	11.8	53.0	0.563	4.7	16.1	14.9	1.9	25.5	285
C	52.7	34.3	29.2	5.1	26.1	23.2	0.613	6.9	20.5	14.7	2.8	12.9	49
D	52.2	28.1	30.1	0	24.8	15.0	0.546	0	2.4	6.1	0.4	4.2	1
8 A	32.6	27.1	16.6	10.5	9.7	38.5	0.620	3.4	9.9	4.4	0.9	13.3	149
B	33.6	21.4	15.4	6.0	11.3	34.0	0.770	3.4	6.8	4.0	1.0	5.7	97
C	35.5	23.8	19.7	4.1	24.0	18.6	0.714	2.2	6.4	4.3	1.2	7.3	22
D	37.6	24.7	24.5	0.2	26.2	13.5	0.632	0.4	1.9	2.8	0.3	6.9	0
9 A	64.0	47.6	34.3	13.3	41.1	26.0	0.405	0.3	2.3	0.5	0.1	0	8
B	55.3	41.7	36.8	4.9	36.0	25.0	0.379	0	0	0.4	0.1	0.5	2
C	57.5	46.4	42.6	3.8	37.1	24.7	0.370	0	0	0.4	0.1	2.4	2
D	76.9	51.0	47.6	3.4	48.9	22.8	0.360	0	3.8	0.4	0.4	3.9	1

[a]Terms and symbols used are as follows: w_{100} = water content for 100% saturation; w_L = liquid limit; w_P = plastic limit; I_P = plasticity index; w_S = shrinkage limit; $<2\mu$ = clay fraction less than 2μ; CEC = cation exchange capacity; C.I. = particle-size composite index = $\log (Q_3/Q_1)^{1/2}$ in which Q_3 and Q_1 are the third and first quartile deviations or the particle sizes having 75% and 25% passing on the grain-size distribution curve, respectively; and S.A. = surface area (to obtain surface area in sq m/gm, multiply by 17.65).
[b]A = raw soil; B, C, D = soils heated for 24 hr at 300, 600, and 900 C, respectively.

TABLE 3

CORRELATION EQUATIONS[a]

Equation No.	Independent Variables	Dependent Variable	Estimating Equation	Standard Error of Estimate	Coefficient: Determination	Coefficient: Correlation
			(a) Simple Correlation: S = Free Swelling			
1	I_P	S	$S = 0.4436\ I_P + 11.3219$	21.32	0.8823	0.9393
2	w_S	S	$S = 69.6024 - 1.0092\ w_S$	56.04	0.1869	0.4323
3 a	$< 2\ \mu$	S	$S = 1.1689\ (< 2\ \mu) - 21.6976$	49.57	0.3638	0.6031
3 b	S.A.	S	$S = 3.0182$ S.A. $+ 0.5524$	45.35	0.4362	0.6606
3 c	C.I.	S	$S = 100.7489$ C.I. $- 21.4823$	57.98	0.0784	0.2800
4	CEC	S	$S = 1.1845$ CEC $+ 3.5958$	49.40	0.3680	0.6067
5	Ca	S	$S = 0.8115$ Ca $+ 21.0915$	59.90	0.0710	0.2664
6	Na	S	$S = 3.0594$ Na $+ 11.2133$	25.71	0.8288	0.9104
			(b) Multiple Correlation: S = Free Swelling			
7	I_P, $< 2\ \mu$, CEC, & Na	S	$S = 0.2521\ I_P + 0.1694\ (< 2\ \mu) + 0.0040$ CEC $+ 1.4535$ Na $+ 1.8894$	14.67	0.9492	0.9743
8	I_P, S.A., CEC, & Na	S	$S = 0.2638\ I_P + 0.4798$ S.A. $+ 0.0006$ CEC $+ 1.3245$ Na $+ 5.1013$	13.09	0.9530	0.9762
			(c) Simple Correlation			
9 a	$< 2\ \mu$	S.A.	S.A. $= 0.3187\ (< 2\ \mu) - 4.0343$	8.97	0.5646	0.7514
9 b	S.A.	$< 2\ \mu$	$< 2\ \mu = 1.7717$ S.A. $+ 28.3253$	20.56	0.5646	0.7514
10 a	$< 2\ \mu$	C.I.	C.I. $= 0.00338\ (< 2\ \mu) + 0.3978$	0.135	0.3935	0.6273
10 b	C.I.	$< 2\ \mu$	$< 2\ \mu = 116.4463$ C.I. $- 16.8229$	24.27	0.3935	0.6273
11 a	C.I.	S.A.	S.A. $= 46.9578$ C.I. $- 14.9329$	10.91	0.3557	0.5964
11 b	S.A.	C.I.	C.I. $= 0.00758$ S.A. $+ 0.4753$	0.135	0.3557	0.5964
12 a	w_L	I_P	$I_P = 0.9463\ w_L - 44.1404$	28.76	0.9517	0.9755
12 b	I_P	w_L	$w_L = 1.0057\ I_P + 49.5460$	29.65	0.9517	0.9755
13 a	w_L	w_{100}	$w_{100} = 0.4534\ w_L + 59.0300$	38.34	0.7301	0.8545
13 b	w_{100}	w_L	$w_L = 1.6104\ w_{100} - 66.2519$	70.10	0.7301	0.8545
14	C	S	$S = 0.2590\ C + 13.2330$	56.54	0.1722	0.4150

[a]For notations (definitions) see Table 2 footnotes.

in a vertical direction without any load and restraint was developed to measure the relative values of volumetric changes of clay minerals tested under identical conditions. After being placed in an air-tight cabinet with saturated humidity for at least two weeks, a 5-gm sample is molded in a 1.12-in. transparent lucite cylinder with a porous stone base under a static load of 50 psi for 1 min. The load is applied and released at a constant rate of 33-psi/min. A porous stone is placed on the top of the compacted specimen and 25 ml of distilled water at room temperature is introduced from the top. Increases in thickness are measured to 0.001 in. until no further swelling occurs. Free swelling is defined as the increase in thickness divided by the original thickness and multiplied by 100. Initial and final water contents are determined. The mass specific gravity, void ratio, porosity, degree of saturation before and after the test, and water content to reach full saturation are computed.

The test results significant in subsequent correlation analyses are summarized in Table 2.

CORRELATION ANALYSIS

Simple and multiple correlation analyses (8) of various combinations were made on the 36 soil samples by using free swelling as the dependent variable and physico-chemical properties as the independent variables after elimination of some of the test results which were either too insignificant, erratic, or confusing to show any apparent relations. Simple correlation analyses show the predominance of linear relationships between the dependent and independent variables.

The most rational combination (9, 10, 11, 12) seems to be the multiple correlation between free swelling as the dependent variable and the four independent variables

(plasticity index, clay fraction in terms of particle size less than 2 μ or surface area, cation exchange capacity, exchangeable sodium) as shown by Eqs. 7 and 8 in Table 3. Both equations give a coefficient of multiple correlation of about 0.975.

Because the relationships between free swelling and shrinkage limit (13) and between free swelling and calcium are weak, as shown by their coefficients of correlation of 0.432 and 0.266, respectively, they are not included in the equations.

Equations 9, 10 and 11 (Table 3) show the simple relationships among surface area, clay fraction less than 2 μ, and composite index. The coefficient of correlation of 0.751 between surface area and clay fraction less than 2 μ is highest. The relationship between liquid limit and plasticity index (14, 15) in Eq. 12 is excellent, as indicated by a coefficient of correlation of 0.976.

DISCUSSION

Free swelling tested under conditions in this study is a measure of relative volume changes of soils and may be used as an indication of swelling tested under other conditions (16, 17). Free swelling developed in this study can be estimated by either of two equations:

$$S\ (\%) = 0.252\ I_P + 0.169 < 2\ \mu + 0.004\ CEC + 1.454\ Na + 1.889 \qquad (15)$$

$$S\ (\%) = 0.264\ I_P + 0.480\ S.A. + 0.001\ CEC + 1.325\ Na + 5.101 \qquad (16)$$

By using surface area instead of clay fraction less than 2 μ, Eq. 16 gives a slightly higher coefficient of multiple correlation. Surface area may be a better indication of the true activity of the clay fraction than a single 2-μ size (18). A coefficient of determination of about 0.95 indicates that 95 percent of the variations in free swelling is associated with variations in the four factors included in the equations.

The effect of heat treatment on physico-chemical properties of soils can be readily seen from the test results in Table 1. With heating at 900 C for 24 hr, practically all soils become nonplastic (19). Plasticity index decreases with increase in temperature.

ACKNOWLEDGMENTS

This research was supported by a grant from the National Science Foundation under the Engineering Sciences Basic Research Program and was performed in the soils laboratories of New York University. Gratitude is expressed to Howard L. Walowitz of the New York University Computing Center for his assistance in computer work. The suppliers of the various types of clay minerals used in the study are gratefully acknowledged.

REFERENCES

1. "Procedures for Testing Soils." ASTM (1958).
2. "Standard Specifications for Highway Materials and Methods of Sampling and Testing." AASHO (1961).
3. Kinter and Diamond, "Pretreatment of Soils and Clays for Measuring External Surface Area by Glycerol Retention." Public Roads, pp. 187-190 (June 1959).
4. Kinter and Diamond, "Surface Areas of Clay Minerals as Derived from Measurements of Glycerol Retention." Proc. 5th Nat. Conf. on Clay and Clay Minerals, pp. 334-347 (1958).
5. Jackson, M. L., "Soil Chemical Analysis." Prentice-Hall (1958).
6. Willard, Merritt and Dean, "Instrumental Methods and Analysis." D. Van Nostrand Co. (1958).
7. Krumbein, W. C., "The Use of Quartile Measure in Describing and Comparing Sediments." Amer. Jour. of Sci., 37:98-111 (1936).
8. Ezekiel and Fox, "Methods of Correlation and Regression Analysis." John Wiley (1959).

9. Winterkorn, H. F., "Physico-Chemical Properties of Soils." Proc. 2nd Internat. Conf. on Soil Mechanics and Foundation Eng., 1:23-29 (1948).
10. Gill and Reaves, "Relationships of Atterberg Limits and Cation Exchange Capacity to Some Physical Properties of Soil." Proc. Soil Sci. Soc. of Amer., 21:491-494 (1957).
11. Odell, Thornburn and McKenzie, "Relationships of Atterberg Limits to Some Other Properties of Illinois Soils." Proc. Soil Sci. Soc. of Amer., 24(4):297-300 (1960).
12. Brooks, Bower and Reeves, "The Effect of Various Exchangeable Cations upon the Physical Conditions of Soils." Proc. Soil Sci. Soc. of Amer., 20(3):325-327 (1956).
13. Seed, Woodward and Lundgren, "Closure on Prediction of Swelling Potential for Compacted Clays." ASCE Proc. Sep. 3528 (May 1963).
14. Casagrande, A., "Classification and Identification of Soils." ASCE Proc., 73:783-810 (1947).
15. Matsuo, S., "Character of Consistency of Soils as Shown in the Plasticity Chart." Rept. on Res. and Invest., Japanese Comm. on Soil Mechanics and Foundation Eng., pp. 9-11 (1952).
16. Seed, Woodward and Lundgren, "Prediction of Swelling Potential for Compacted Clays." ASCE Proc. Sep. 3169 (June 1962).
17. Lambe, T. W., "The Character and Identification of Expansive Soils." FHA Tech. Studies Rept. (Dec. 1960).
18. Li, M. C., "Discussion on Prediction of Swelling Potential for Compacted Clays." ASCE Proc. Sep. 3378 (Dec. 1962).
19. Rao and Wadhawan, "Plasticity of Heated Soils." Irrigation and Power Jour., India, 12(2) (April 1955).