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A COMPARATIVE STUDY OF SIMULATED AND NATURAL WEATHERING OF SOME OKLAHOMA SHALES

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Abstract—Shales from six locations in Oklahoma were subjected to natural weathering for 2 yr. Simulated weathering of these shales was effected in the laboratory by subjecting them to ultrasonic treatment in a tank type device. Both treatments produced disaggregation. X-ray diffraction patterns for the ultrasonically treated and weathered shales indicated no major changes in the types of clay minerals. However, natural weathering in the field produced degradation of the clay minerals in addition to disaggregation of the shales. Ultrasonic treatment appears to be a good predictive test for determining the durability and weatherability of the shales; however, it can simulate field weathering only so far as the engineering index properties of the shale are concerned. It is not a predictive test where the mineralogical characteristics are of significance.

INTRODUCTION

This study determines how well tests with short term ultrasonic treatment of fresh shale can predict the properties of the shale weathered naturally for a much longer period of time.

MATERIALS

Shale samples were obtained from six different locations in Oklahoma. Pertinent information is presented in Table 1.

For the purposes of this study, there are three distinguishable sets of samples. Fresh shale refers to samples obtained freshly from a sampling location; such are identified as field samples. Field weathered samples are obtained from shale, contained in boxes, which has been exposed to weathering for 24 months. Ultrasonically treated samples are fresh samples which have been treated ultrasonically in the laboratory.

PREPARATION OF SAMPLES

Field weathering

The fresh shale samples obtained from the field were put in Plexiglass boxes 12 × 12 × 8 in. Provision was made for drainage at the base by 3/8 in. dia. perforated pipes embedded in a $1\frac{1}{2}$ in. thick layer of clean gravel and sand. The boxes were covered only by 3/8 in. mesh screen. The boxes were set outside, $2\frac{1}{2}$ ft above the ground surface, in the open and away from buildings and trees.

Laboratory weathering

Weathering of the shale samples in the laboratory was accomplished by ultrasonic treatment. Ultrasonic disaggregation of sandstones and silt-stones has been reported by Savage (1969); of shales by Gipson (1963) and Alguire (1969).

In comparing probe and the tank type ultrasonic equipment, these investigators found that the latter is

Table 1. Description of shales

Shale No.	County	Geologic unit	System	Predominant clay minerals
8	Mayes	Chattanooga	Mississippian	*I*
13	McCurtin	Washita	Cretaceous	*M, K*
15	LeFlore	Stanley	Mississippian	*I, K, M*
21	Stephens	Claypool	Permian	*ML, M, I, K*
22	Carter	Springer-Gottard	Pennsylvanian	*ML, K, M, I*
24	McIntosh	Senora	Pennsylvanian	*I, K, M*

I = Illite; *K* = Kaolinite; *M* = Montmorillonite; *ML* = Mixed Layer Illite–Montmorillonite.

more suitable for shaley rocks and rocks with high percentage of clay. Since the six shales under study contained large amounts of clay, it was decided to use tank type equipment (Westinghouse Mini Magnapak). The tank used has a capacity of 1·5 gal and the generator an average output of 200 W at a frequency of 20 kc.

The field shale sample was air dried and pulverized to pass a U.S. Std. No. 10 sieve. A sample was then placed in a beaker and soaked with distilled water for 12–18 hr. Then, the beaker and its contents were placed in the sonifer and subjected to ultrasonic treatment for time durations of 1/4, 1/2, 1, 2, 4 and 8 hr at a temperature of 70 ± 2°F. During the treatment period, the shale was prevented from settling by means of mechanical stirring. Minor modifications of this method were effected, depending on the specific laboratory test to be run; such modifications are indicated herein.

LABORATORY TESTS AND DATA

Particle size distribution, liquid limit, plastic limit and X-ray diffraction analysis were set as tentative criteria for studying the effects of natural weathering and ultrasonic treatment.

Particle size analysis

This test followed ASTM:D422. For the field and field-weathered samples, a 50 g portion of the −10 mesh material was soaked in 125 ml of Calgon solution, prepared according to the standard, for 12 hr and the dispersion was transferred to a hydrometer jar. The dispersion was made up to 1000 ml with distilled water and the hydrometer test performed.

In the case of ultrasonically treated shales, the sample soaked as before was subjected to ultrasonic treatment for 1/4, 1/2, 1, 2, 4 or 8 hr duration. Then, the sample was transferred to the hydrometer jar, made up to 1000 ml and hydrometer test was performed.

A typical pattern of particle size distribution curves for shale No. 24 is shown in Fig. 1. A similar trend was observed with the other shales. The increases in amounts of 5-μm clay size particles with ultrasonic treatment time, for all the shales, are reported in Table 2. The amounts of 5-μm clay for the field samples, ultrasonically treated samples and field weathered samples are reported in Table 3.

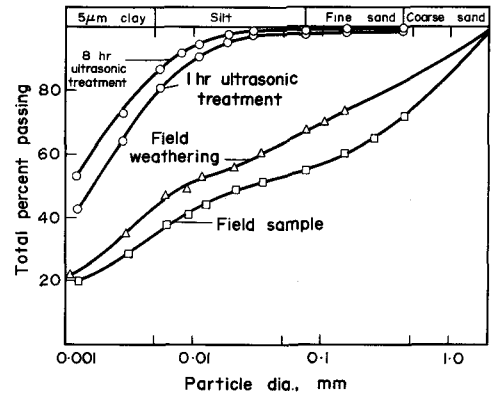

Fig. 1. Effect of ultrasonic treatment and field weathering on particle size distribution of shale No. 24.

Liquid limit and plastic limit

These tests were conducted on a −40 mesh sample of the pulverized shale material, in accordance with the standard ASTM procedures D423 and D424. For field and field-weathered samples, the material was air dried, crushed to pass 40 mesh and tested. For the ultrasonically treated shales, 125 g of the −10 mesh sample were soaked in 125 ml of distilled water for 12–18 hr and treated ultrasonically for 1/4, 1/2, 1, 2, 4 or 8 hr. Then the material was washed through a 40 mesh sieve, air dried, and again pulverized to pass 40 mesh. The liquid and plastic limits of this material were determined in accordance with the above standard procedures. The liquid and plastic limit values for all six shales are given in Table 2.

X-ray diffraction

For the three sets of samples, a portion of the material prepared for liquid and plastic limit tests was used.

Table 2. Effect of ultrasonic treatment time on the amount of 5-μm clay size particles

Shale No.	Treatment time (hr)						
	0	1/4	1/2	1	2	4	8
8	3	15	18	18	25	28	33
13	59	66	70	68	65	64	69
15	18	52	62	80	81	81	83
21	43	74	76	76	76	76	77
22	82	89	89	92	90	93	90
24	35	74	75	79	80	82	85

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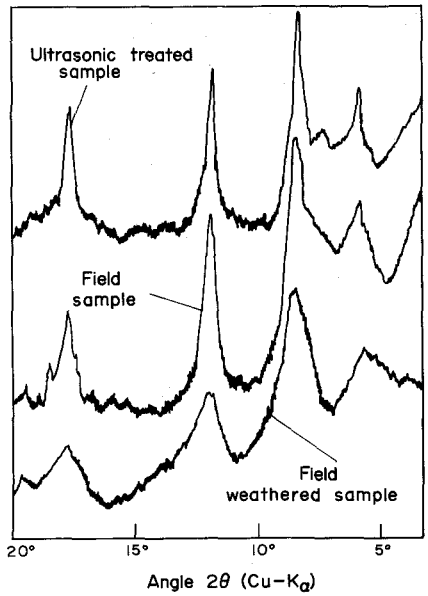

Fig. 2. Effect of ultrasonic treatment and field weathering on X-ray diffractograms of shale No. 24.

About 20 g of the −200 mesh material were mixed with 100 ml of distilled water in a 100 ml measuring jar and thoroughly shaken. A sample was removed after 15 min of settling so as to include 10–20 μm particles, placed on a glass slide and dried at 140°F. In the case of ultrasonically treated samples, such slides were prepared for each treatment time.

X-ray diffraction patterns were obtained using a Norelco X-ray diffractometer operated at 40 kV and 20 mA. Copper K_α radiation was employed with an Ni filter. Glycolation and heating up to 600°C were used to confirm the type of clay minerals identified (Table 1). A comparison of X-ray diffractograms of the ultrasonically treated, field weathered and the field shale is presented in Fig. 2 for shale No. 24. A similar trend was observed for other shales.

DISCUSSION AND CONCLUSIONS

With the exception of small variations, inherent with all soil experiments, the various experiments showed a general trend for each type of weathering.

Ultrasonic treatment

The shale samples steadily assumed finer gradations with increase in ultrasonic treatment time (Fig. 1). In general, the changes in particle size distribution occurred rapidly at first but then tended to reach asymptotically stable values with time (Table 2). Most of the disaggregation took place within 1 hr. Beyond that period there is very little change in gradation characteristics for all shales except No. 8. Shale No. 8 is a highly indurated silty material which initially had a very low percentage of clay material. Significant disaggregation occurred after 1 hr, and would probably continue even after 8 hr of ultrasonic treatment.

As expected, the increase in the amount of clay size particles, which occurred with the ultrasonic treatment time, increased the liquid and plastic limits (Table 3). Due to disaggregation, greater surface area of clay platelets is exposed to the influence of external agencies,

Table 3. Data on the amount of 5-μm clay, liquid limit and plastic limit values of shale samples and their weathering products

Shale No.	8	13	15	21	22	24
5-μm clay (%)						
A	3	59	18	43	82	35
B	18	68	80	76	92	79
C	33	69	83	77	90	85
D	9	61	20	55	83	44
Liquid limit						
A	np	43	24	40	64	29
B	32	51	45	33	69	47
C	40	55	46	45	72	47
D	22	51	28	43	64	32
Plastic limit						
A	np	20	22	26	35	23
B	np	22	25	20	35	27
C	24	23	26	22	33	25
D	np	18	21	23	32	25

A = Original field sample; *B* = Sample treated for 1-hr by ultrasonic method; *C* = Sample treated for 8-hr by ultrasonic method; *D* = Sample afforded 24-month natural weathering.
np = Non plastic.

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especially water, thereby contributing to higher plasticity. No relationship, however, could be established between the liquid limit and the amount of shale particles smaller than a given size. The changes in liquid limit occurred more rapidly in the first one hour of treatment. The plastic limit, generally, changed at a lesser rate than the liquid limit. Also, any ultrasonic treatment time considered optimum from a particle size distribution viewpoint may not be so with regard to plasticity characteristics.

Clay mineral analysis by X-ray diffraction is generally qualitative and gives only semi-quantitative information. The relative proportions of clay minerals in a sample, however, can be estimated from the relative intensities of the peaks on an X-ray diffraction pattern (Gillott, 1969). For all six untreated and ultrasonically treated shale samples, the diffractograms did not reveal any alterations in clay mineral types. The patterns for a given shale at different ultrasonic treatment times looked almost the same. The patterns obtained for shales ultrasonically treated for 1 hr or more showed better-defined peaks than did the patterns for field samples (Fig. 2). Since aggregation causes weakened X-ray reflections (Gillott, 1969) it may be concluded that ultrasonic treatment effectively disaggregates the particles.

Field weathering

Field weathering altered the particle size distribution for all the six shales; i.e. the amount of 5-μm size clay particles was increased in all cases (Table 3). The changes produced in the particle size distributions by 24-month field weathering corresponded, in general, to those brought about by one hour or less of ultrasonic treatment.

The liquid and plastic limit values for the field weathered shales generally increased over the values for the field samples, and in a very general way, fell between the field sample and the 8 hr ultrasonically treated sample. One exception is shale No. 22 for which the values for the field weathered sample were the same as those of the original field sample. This particular shale is a heavy clay containing about 82 per cent <5-μm size clay particles, and appears to be already greatly weathered. Thus, neither the field weathering nor the ultrasonic treatment had noticeable effect on this shale sample. Within the normal accuracy range of these limit tests, the liquid and plastic limit values for all the other field weathered samples correspond to the values obtained for the material treated ultrasonically for 1 hr or less.

The X-ray diffraction analysis of the field weathered samples indicated, in general, all the clay minerals present in the original field sample. The peaks were, however, less well-defined and broader than for the field sample, which suggests the presence of imperfections in the crystal lattice of the minerals. These imperfections could possibly be due to degradation caused by weathering. These might also be due to environmental impurities adhering to the broken surfaces of clay platelets.

Comparison

Both ultrasonic treatment and field weathering produced disaggregation of the shale particles. The entire sample was reduced to a mass containing smaller sized particles. In both cases, the types of clay minerals were the same before and after treatment.

Field weathering, in addition to disaggregation, produced degradation of the clay particles. Field weathering for a much longer period of time could conceivably alter the clay mineral types.

However, based on the limited experimental evidence, it is concluded that ultrasonic treatment simulates field weathering so far as the engineering index properties are concerned. Thus, ultrasonic treatment may serve as a good predictive test for determining the durability and weatherability of the shales.

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Résumé—Des schistes provenant de six localités de l'Oklahoma ont été soumis à une altération naturelle pendant deux ans. L'altération simulée de ces schistes a été effectuée au laboratoire en les soumettant à un traitement par ultrasons dans un montage du type réservoir. Les deux traitements ont entraîné une désagrégation. Les diagrammes de diffraction *X* des schistes traités aux ultrasons et des schistes altérés n'indiquent pas de différence majeure dans les types de minéraux argileux. Cependant, l'altération naturelle

sur le terrain entraîne une dégradation des minéraux argileux, en plus de la désagrégation des schistes. Le traitment aux ultrasons apparaît comme un bon test pour prédire la durabilité et l'altérabilité des schistes; toutefois, il ne peut simuler l'altération sur le terrain que dans la mesure où les propriétés technologiques repères des schistes sont concernées. Ce n'est plus un test prévisionnel lorsque les caractéristiques minéralogiques ont une importance.

Kurzreferat—Schiefertone von sechs Lokalitäten in Oklahoma wurden für zwei Jahre der natürlichen Verwitterung ausgesetzt. Im Labor wurde die Verwitterung dieser Schiefertone durch Behandlung mit Ultraschall in einer aus einem Behälter bestehenden Versuchsanordnung nachgeahmt. Beide Behandlungsarten riefen eine Aggregatzerstörung hervor. Die Röntgenbeugungsdiagramme der ultraschallbehandelten und der verwitterten Schiefertone lassen keine wesentlichen Veränderungen in der Art der Tonminerale erkennen. Die natürliche Verwitterung im Gelände rief jedoch zusätzlich zur Aggregatzerstörung der Schiefertone einen Abbau der Tonminerale hervor. Ultraschallbehandlung scheint sich gut für die Vorausbestimmung der Widerstandsfähigkeit und Verwitterbarkeit der Schiefertone zu eignen. Sie kann jedoch die Verwitterung im Gelände nur soweit nachahmen, als technische Kenngrößen betroffen sind. Sie ist hingegen dort kein für eine Vorhersage geeignetes Testverfahren, wo mineralogische Eigenschaften von Bedeutung sind.

Резюме — Сланцы, из шести местностей в Оклахоме, подвергались естественному выветриванию в продолжение двух лет. А в лаборатории проводилось исскуственное воспроизведение выветривания этих сланцев при помощи ультразвуковой обработки в бакообразном приспособлении. Обе обработки повели к дезагрегации. Рентгенограммы сланцев подвергавшихся ультразвуковой обработке и естественно выветрившихся сланцев не показали заметных изменений в типах минеральных глин. Однако естественное выветривание кроме дезагрегации сланцев повело к распадению глинистых минералов. Ультразвуковая обработка, кажется, является подходящей обработкой для предсказочных испытаний с целью определения долговечности и выветриваемости сланцев, однако, этот способ может моделировать выветривание в естественных условиях только по отношению к техническим указателям характеристик сланцев, что же касается минералогических характеристик это испытание не является прогностическим.

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