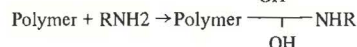
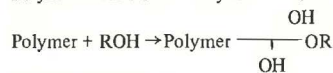
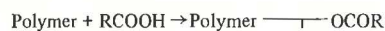


served that lack of adhesion is a major deficiency in a polymer that is to be used for soil stabilization. Adhesion between polymer molecules and a polar substrate such as the quartz surfaces of most sandy noncohesive soils depends, among other things, on the flexibility and polarity of the polymer molecules. Water, a polar material, spreads on quartz surfaces very quickly. Similarly, natural rubber, which has very flexible molecules, is a very good adhesive. Thus a polymer that is relatively flexible, like an elastomer, and is also polar has good properties for stabilization for erosion control. Due to the polarity of molecules, there will be a strong electrical force between them and the polar substrate. This will give rise to strong adhesion. In addition, if the molecules are flexible, a large area of contact and better adsorption will result. This will increase the van der Waals forces, which in turn will improve adhesion. Flexibility in a polymer can be increased by incorporating a suitable plasticizer. Similarly, polarity can be induced or increased by combining an acid group in the polymer molecule. These functional groups can be incorporated, even after polymerization, by treating the polymer with carboxylic acid, alcohol, or amine. The resulting modifications are indicated below:



The main disadvantage to adding an acid group is that it makes the polymer more sensitive to the effects of water.

Most of the elastomers, such as butadiene-styrene and butadiene-acrylonitrile, have quite flexible molecules and consequently are good adhesives. These copolymers are also less water sensitive. If such copolymers can be made polar, their adhesive and cohesive strength can be further increased. The most common acid group that can make these elastomers more polar is the carboxylic group (COOH). By incorporating this group into an elastomer, a strong and good adhesive polymer can be produced. The amount of carboxylic group in the elastomer will affect the overall properties of the polymer. It has been reported (8) that the addition of up to 20 percent of an acid group in an elastomer increases both the adhesion and the cohesive strength of the

polymer because of the increased intermolecular and intramolecular forces. When the acid group exceeds 20 percent, the polymer becomes rigid and its adhesive properties decrease. Moreover, it becomes somewhat water sensitive. Thus, for controlling the erosion of noncohesive sandy and silty materials, a carboxylated elastomer of butadiene-styrene or butadiene-acrylonitrile in the form of latex or emulsion, which is dilutable in water, would be a promising polymer.

#### ACKNOWLEDGMENT

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## Pore-Size Distribution and Its Relation to Durability and Strength of Shales

M. SURENDRA, C.W. LOVELL, AND L.E. WOOD

Shale durability is measured by resistance to slaking in a standard laboratory test. All slaking mechanisms (namely, air-pressure breakage, differential swelling, and dissolution of cementing agents) require that water penetrate the pore space of the shale pieces. Since it is now possible to measure the magnitude and size distribution of these pores by mercury intrusion, correlation of slaking and pore-size distribution is feasible. Testing of slake durability, pore-

size distribution, and point-load strength was undertaken on eight Indiana shales of varying durability and strength. It is proposed that the shales be classified as to performance in compacted embankments by slake-durability and point-load-strength indices and that either index can be estimated from parameters of the pore-size distribution. Parameters from the pore-size distribution study (namely, cumulative porosity, median diameter, and spread

factor) were correlated with the slake durability and the point-load strength by linear regression. These Indiana shales were then classified into performance categories based on durability and strength values predicted from the measured pore-size parameters. Thus, the pore-size measurements appear to have both conceptual and practical value with respect to the design and construction of compacted shale embankments.

Use of excavated shale from cuts and borrow areas in Indiana for compacted embankments as a rock fill [in lift thicknesses of about 1 m (3 ft)] has led to various problems, namely, excessive settlement and slope failures. This initiated an extensive research program at Purdue University through the Joint Highway Research Project to study the behavior of Indiana shales. Deo (1) proposed a classification system that is currently being used by the Indiana State Highway Commission (ISHC). Chapman (2) investigated additional laboratory classification tests. Bailey (3) and Hale (4) investigated the factors relating to degradation of shales during the compaction process, and van Zyl (5) prepared a statistical analysis of the data provided by ISHC for the shales tested in their laboratory. Abeyesekera (6) investigated the stress-deformation and strength characteristics of compacted New Providence shale, and Witsman (7) investigated the effect of compacted prestress on compressibility of compacted New Providence shale. Surendra (8) investigated the potential for stabilizing compacted shales by using salts and lime. He also measured pore-size distributions of shale aggregates and developed statistical relationships among pore-size distribution parameters, slake durability, and point-load strength (PLS). The latter relationships suggest a new engineering classification for midwestern U.S. shales for use in compacted embankments.

## LITERATURE REVIEW

### Slaking Tests

Slaking is defined in the dictionary of geological terms (9) as follows: "Loosely, the crumbling and disintegration of earth materials when exposed to air or moisture. More specifically, the breaking up of dried clay when saturated with water, due either to compression of entrapped air by inwardly migrating capillary water, or to the progressive swelling and sloughing off of the outer layers." Slaking is measured in the laboratory by the percentage of weight retained or lost through a given sieve as a result of soaking the specimen in water. A number of tests following this concept have been developed by various investigators. A description of some of these slaking tests has been presented by Surendra (8).

### Slaking Mechanisms

Terzaghi and Peck (10, p. 146) attributed the slaking phenomenon to the compression of entrapped air in the pores as water entered these pores. This air entrapped in the pores exerts tension on the solid skeleton, which causes the material to fail in tension. The behavior can be recognized in the case of soil aggregates and poorly cemented (i.e., compacted) shales and mudstones. Moriwaki (11) found that slaking of compacted kaolinite can be attributed to this mechanism. There have been cases (12;13, pp. 1-14) in which this mechanism did not satisfactorily explain the observation.

Clay surface hydration by ion adsorption has been suggested as the second mechanism that causes slaking through swelling of illite, chlorite, and montmorillonitic clays (14). Differential swelling due

to hydration or osmotic swelling is reported to be the main cause of slaking in expansive materials (11). Tschepotarioff (15, p. 102) defines slaking as a surface phenomenon in the following way: "...the clay layer at the exposed surface swells first and therefore expands more than the adjoining inner layers; the induced relative displacements are liable to detach the surface layer and cause it to disintegrate and slough away. The process can then be repeated and gradually progress from the surface inward."

Removal of cementing agents in the case of shales, siltstones, and mudstones by the dissolving action of the moving groundwater is considered to be the third mechanism that causes slaking (11,12). The pH of the percolating groundwater and the presence of oxygen, carbon dioxide, and other minerals in the shales control the slaking due to this mechanism.

No single mechanism can be considered the dominant cause for slaking of shales. A combination of the above-mentioned mechanisms is most likely, either by one triggering the other or by each occurring independently. The composition and the environment in which the shale is placed determine the principal mechanism that causes the failure.

### Shale Classification

Earlier classification systems were based on visual observation of physical features, namely, fissility and breaking characteristics of shales in situ and of hand specimens in the laboratory (16-18). The more-recent classification systems take into consideration the results from durability tests and observed field behavior (1,18-22). These classification systems may also employ test results in the form of Atterberg limits, pH's, rate of slaking values, and PLS. A classification system proposed by Morgenstern and Eigenbrod (23) used the unconfined compressive strength of soaked specimens. A discussion of these classification systems has been presented by Surendra (8). A summary of these classification systems and their tests is presented in Table 1.

The principal features needed in a shale-classification system are (a) a measure of durability (i.e., resistance to environmentally induced slaking cycle during service) and (b) a measure of strength or hardness (i.e., resistance to construction degradation in the field, which determines the ease with which it can be placed in an embankment).

Classification systems thus developed use measured properties in the laboratory to predict field behavior. The slake-durability test is normally used to distinguish durable and nondurable shales. Additional tests for durability are sometimes necessary (20,22,24). Visual observation of the material at the end of the slake-durability test is also useful (2,20). ISHC uses the classification system developed by Deo (1).

Indiana shales may be placed in three categories, namely, hard and durable, hard and nondurable, and soft and nondurable, as shown in Figure 1 (8). In Figure 1, the durability is rated from the slake-durability test and the hardness from the PLS test. It can be seen that increasing durability is generally associated with increased PLS. The properties of shales represented in Figure 1 are given in Table 2.

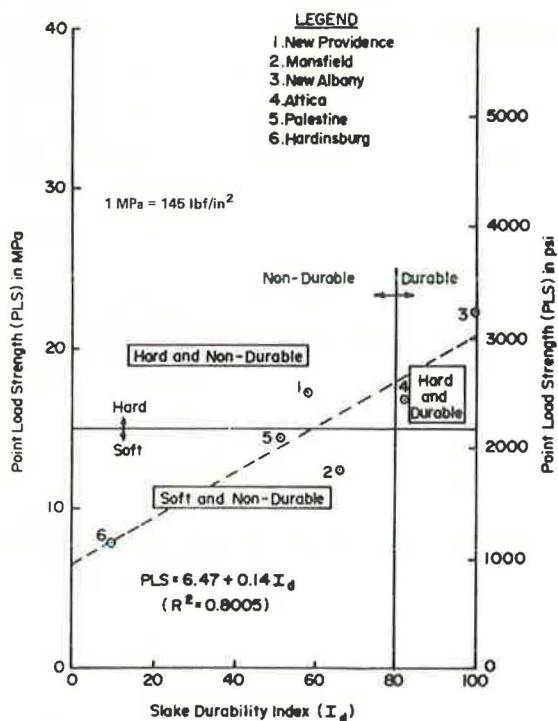
In the slake-durability test, the specimens (discrete pieces of shale) are subjected to a joint effect of abrasion caused by the tumbling action of the rotating drum and softening by water. The shale may be cycled in this test by removing the retained material from the drum, oven-drying it, and rein-



Table 1. Summary of classification tests.

Classification System	Test			Comments
	Durability	Strength	Other	
Gamble (18)	200-revolution, two-cycle slake-durability test	—	Atterberg limits ( $I_p$ )	
Deo (1)	500-revolution, one-cycle, wet and dry slake-durability test; slaking test; modified soundness test	—	—	
Morgenstern and Eigenbrod (23)	—	Unconfined compressive strength test	—	Loss of strength on soaking
Hudec (19)	200-revolution, five-cycle slake-durability test	—	—	
Strohm, Bragg, and Zeigler (20)	200-revolution, two-cycle slake-durability test; slaking test; rate-of-slaking test	—	pH; Atterberg limits ( $I_p$ )	Visual observation of fragmented material in slake-durability test
Andrews and others (21)	200-revolution, two-cycle slake-durability test	—	pH; cationic exchange capacity (CEC)	Preliminary classification based on pH and CEC determines need for any durability test
Franklin (22)	200-revolution, two-cycle slake-durability test ( $Id_2$ )	PLS	Atterberg limits ( $I_p$ )	For $Id_2 > 80$ , PLS is needed; for $Id_2 < 80$ , $I_p$ is needed

Figure 1. Variation of PLS with slake-durability index.



serting it in the drum. The breakdown is increased by increasing the number of cycles, as shown in Figure 2 [data from thesis by Gamble (18)]. Drying the sample prior to the slake-durability test does increase the degradation. Varying reasons for this are supplied by Nakano (13) and by Bailey (3). Degradation is also a function of the number of revolutions of the drum as shown in Figure 3 (data from ISHC).

#### LABORATORY TESTING

##### Materials

Table 3 gives a brief description of all the shales studied and the investigation during which these

Table 2. Properties of shales shown in Figure 1.

Shale	Slake-Durability Index $I_d$	PLS (MPa)	Plasticity Index $I_p$
New Providence	58.0	17.25	11
Mansfield	66.0	12.4	10.6
New Albany	99.1	22.3	5.6
Attica	82.1 <sup>a</sup>	16.8	5
Palestine	51.7	14.5	5.2
Lower Hardinsburg	9.8	8.30	15

Note: 1 MPa = 145 lbf/in<sup>2</sup>.

<sup>a</sup>Second-cycle slake-durability index was estimated [from Abeysekera and Lovell (25)].

shales were sampled. The slake-durability, PLS, and plasticity-index values were given in Table 2 for six of the shales studied.

#### Slake-Durability Test

The slake-durability index was determined according to the procedure of the International Society for Rock Mechanics (26, pp. 32-36). The slake-durability apparatus, developed by Franklin (27), consists of a drum that has a screen opening of 2 mm (0.08 in) (no. 10 sieve). The drum is rotated by an electric motor in a bath of slaking fluid (usually water) at a constant rate (20 rpm). The slaking samples consist of 10 equidimensional pieces of shale that each weigh about 50 g (1.5 oz) and are oven dried at  $110 \pm 5^\circ\text{C}$  ( $225 \pm 10^\circ\text{F}$ ), cooled to room temperature, and placed in the drum of the apparatus. The drum is immersed in the tub that contains the slaking fluid and is rotated for 200 revolutions. At the end of the test, the material retained in the drum is oven dried and weighed. The retained material is then subjected to another cycle of slaking in the rotating drum. The slake-durability index ( $Id_2$ ) is calculated at the end of the second cycle as follows:

$$Id_2 = \left[ \frac{(\text{oven-dried weight of material retained at end of second cycle})}{(\text{oven-dried weight of sample before test})} \right] \times 100.$$

At least four tests were run for each shale, and average values are reported. The slake-durability indices presented in Table 4 are the average of six tests.

Figure 2. Influence of number of cycles on slake-durability index.

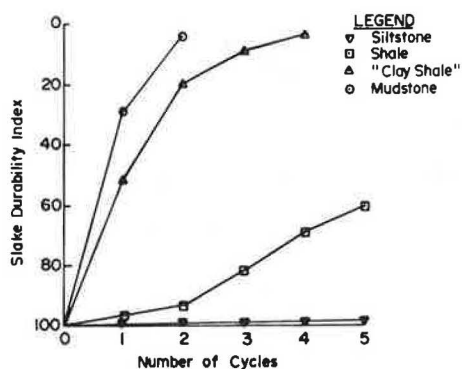


Figure 3. Influence of number of revolutions of drum on slake-durability index.

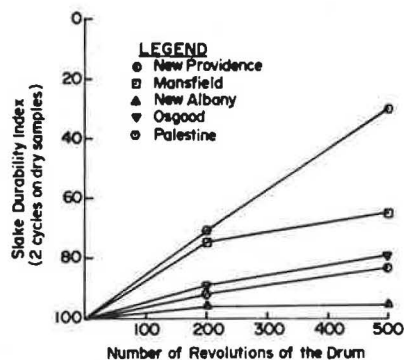


Table 3. Description of shales studied.

ISHC Laboratory No.	Shale	Deo's Classification (1)	Physical Nature	Study
75-55731	New Providence	Soil-like	Hard and nondurable	Abeyesekera (6)
79-55198	New Providence	Soil-like	Hard and nondurable	Hale (4)
74-54684	Mansfield	Soil-like	Soft and nondurable	Chapman (2)
74-54621	New Albany	Rocklike	Hard and durable	Chapman (2)
79-55199	Osgood	Soil-like	Hard and nondurable	Hale (4)
75-55564	Attica	Soil-like	Hard and nondurable	Bailey (3)
74-54716	Palestine	Soil-like	Soft and nondurable	Chapman (2)
79-55204	Palestine	Soil-like	Soft and nondurable	Hale (4)
73-51703	Lower Hardinsburg	Soil-like	Soft and nondurable	Chapman (2)
75-55315	Klondike	Rocklike	Hard and durable	Chapman (2)

Table 4. Durability and PLS properties.

Shale	Slaking Index (SI)	Slake-Durability Index (Id <sub>2</sub> )	Deo's Classification (1)	PLS (MPa)
New Providence	50.81	58	Soil-like	16.18
Mansfield	40.78	66	Soil-like	9.32
New Albany	0.14	99.1	Rocklike	23.68

Note: 1 MPa = 145 lbf/in<sup>2</sup>.

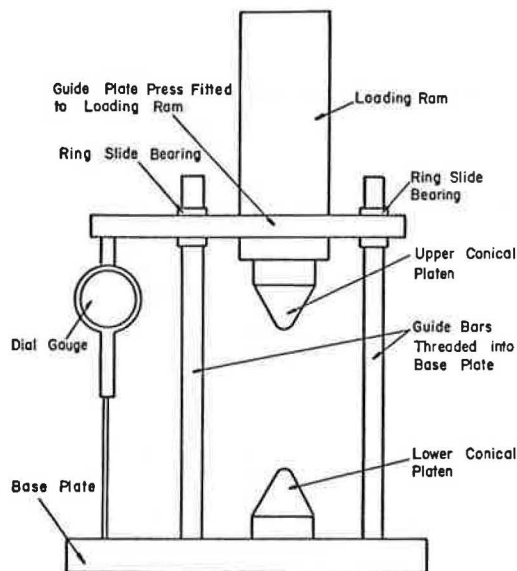
#### PLS Test

A diagram of the PLS test apparatus is shown in Figure 4 [data from reports by Bailey (3)]. The load was applied by an electrically driven compression-testing machine at a constant rate of deformation of 0.254 mm/min (0.01 in/min). The load was monitored through a 22.24-kN (5000-lbf) capacity, SR4-type load cell. The initial dial gage reading indicates the height of the guide plate, and from this the sample thickness (D) can be obtained. The sample of shale consisted of platy pieces approximately equidimensional for the plan area. The sample was loaded perpendicular to the bedding planes and the initial sample thickness of the shales tested varied from 2.56 to 13.41 mm (0.10–0.53 in). The samples were oven dried to constant weight at  $110 \pm 5^\circ\text{C}$  and cooled to room temperature before testing (i.e., all the samples were tested at near-zero moisture content). The PLS index was computed by taking the ratio of maximum compressive load (P) to the square of the initial sample thickness (D). Table 4 presents the PLS of the New Providence, the Mansfield, and the New Albany shale samples; these are averages of several tests.

#### Pore-Size Distribution

The procedure used in this test, the assumptions

Figure 4. Side view of PLS test apparatus.



made in the study, and the appropriate precautions and corrections have been described by Surendra (8). The determination of pore-size distribution is described briefly below.

The oven-dried sample of shale (sometimes consisting of two to three discrete pieces) is initially evacuated and surrounded by mercury, the pressure is raised in small increments, and the volume of mercury that enters the sample after each increment is recorded. With each pressure increment, the mercury is forced into the accessible pores in the sample of a diameter larger than or

Figure 5. Differential and cumulative pore-size distribution for Mansfield, New Providence, and New Albany shales.

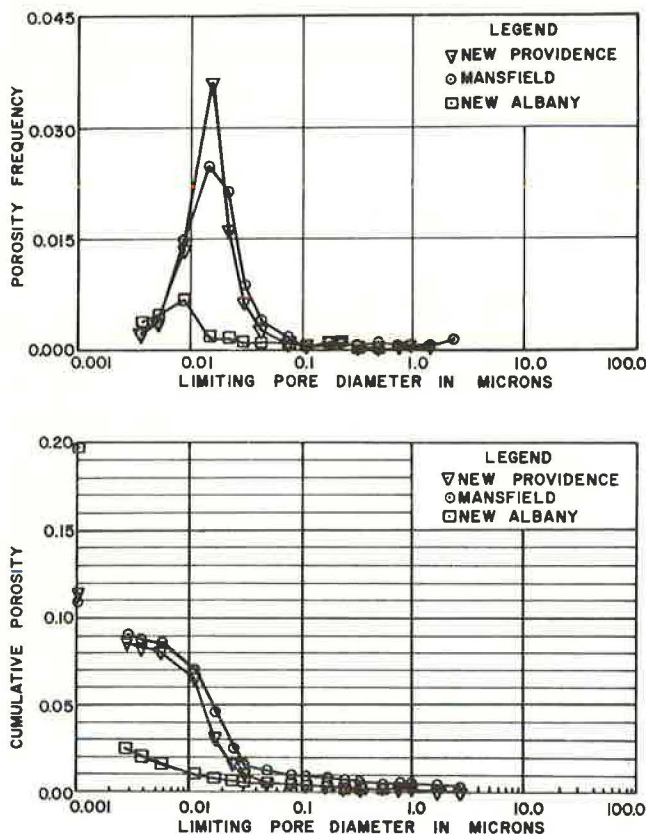


Table 5. Pore-size parameters for shales studied.

Shale	1 ÷ Cumulative Porosity	1 ÷ Median Diameter (μm)	Spread Factor
New Providence	11.83	71.43	2.00
	11.30	71.43	2.14
Mansfield	11.09	62.5	1.56
	11.16	52.63	1.42
New Albany	52.55	114.94	2.64
	55.09	100.00	3.00
Osgood	14.86	50.00	10.25
	14.94	83.33	2.58
Attica	9.29	37.04	1.52
	9.06	38.46	1.54
Palestine	8.12	3.57	2.93
	5.57	8.33	4.00
Lower Hardinsburg	12.67	125.00	1.75
	12.41	108.70	1.96
Klondike	7.69	22.20	1.78
	6.33	27.03	2.38

equal to that calculated by the Washburn (28) equation:

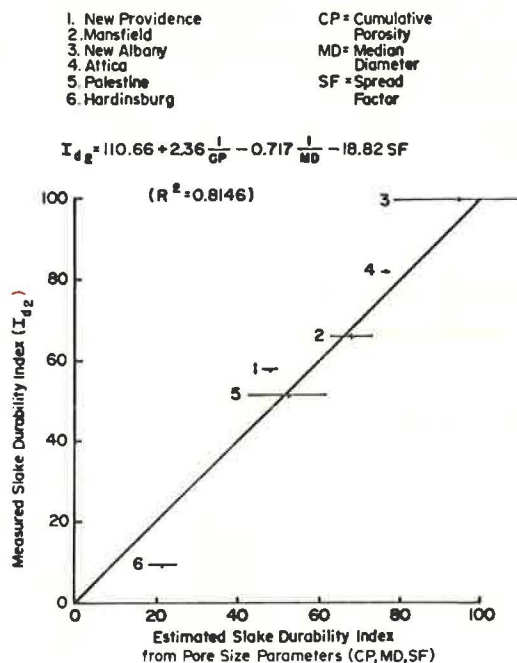
$$P = (4 T_g \cos \theta) / d \quad (1)$$

where

$P$  = absolute pressure required for intrusion,  
 $T_g$  = surface tension of intruding liquid,  
 $\theta$  = contact angle between solid and liquid, and  
 $d$  = limiting pore diameter.

The volume of pore space between pressure increments is recorded, and from this the limiting pore

Figure 6. Estimated slake-durability index ( $I_{d2}$ ) from pore-size parameters versus measured values.



diameter is computed and the pore-size distribution is generated. The pore-size distribution is presented in the form of differential-distribution and cumulative-distribution curves for this study. Figure 5 provides these curves for the three shales given in Table 4.

## RESULTS AND DISCUSSION

### Pore-Size Distribution

Pore-size studies were made on eight Indiana shales (Table 3) as described in detail by Surendra (8). The intrusion constant (Equation 1) used in this study was taken from Kaneuji (29) to be 160 (measured in microns times pounds force per square inch) or 1103.2 (measured in microns times kilonewtons per square meter). The same value was used for all the shales. Two tests were run on each shale. The parameters from these distributions used for correlation with the slake-durability index were as follows:

1. Cumulative porosity: ratio of intruded pore volume to the volume of the sample,
2. Median diameter: diameter of the pore corresponding to the 50th-percentile value of the intruded volume, and
3. Spread factor: ratio of pore diameter corresponding to the 25th-percentile value of the intruded volume to the median diameter.

The above parameters from the cumulative pore-size distribution curves for the shales studied are given in Table 5. The three parameters from the pore-size distribution study (except those for the Osgood and the Klondike shales) were successfully used in a linear regression to correlate with the slake-durability index, and a value of  $R^2 = 0.8146$  was obtained. It can be seen from Figure 6 that the slake-durability index can be estimated with good reliability from the pore-size parameters.



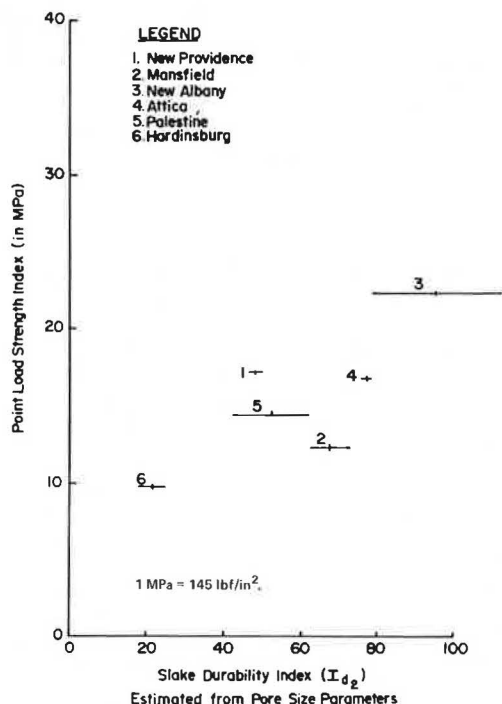
### PLS

PLS was determined in the laboratory as described earlier for six Indiana shales. All the samples tested were 6.6 mm (0.26 in) thick; they were oven dried prior to testing. The results of this test on

the shale samples investigated are presented below (1 MPa = 145 lbf/in<sup>2</sup>):

Shale	PLS (MPa)
New Albany	22.30
Mansfield	12.40
New Providence	17.25
Attica	16.80
Palestine	14.50
Lower Hardinsburg	8.30

Figure 7. Estimated slake-durability index from pore-size parameters versus PLS index.



### SUMMARY--NEW SHALE CLASSIFICATION

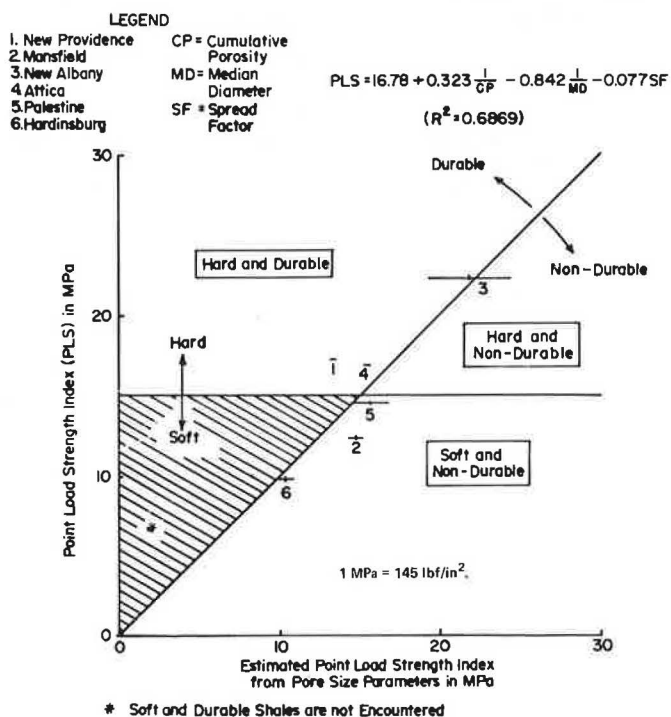
Parameters from the pore-size distribution study--namely, cumulative porosity, median diameter, and spread factor--were correlated with the slake-durability index by linear regression. The R<sup>2</sup>-value for six shales was 0.8146. The results of this regression were presented in Figure 6.

When the predicted slake-durability index was plotted against the PLS index (Figure 7), it was found that most of the shales plotted in a narrow range of strengths and had varying durabilities. The implication is that durability is more sensitive to pore-size distribution than is strength.

PLS indices are plotted against the measured values in Figure 8. Durable shales plot above the diagonal line and nondurable shales fall below it, except in the case of the New Providence shale. The R<sup>2</sup>-value is low, approximately 0.7. An arbitrary strength (indicated by the field behavior) can be selected to designate the shale as hard or soft. In this case, a value of 15 MPa (2175.5 lbf/in<sup>2</sup>) is used. The results from this classification and those of Deo (1) are given below (we found the New Providence shale to be nondurable since it has a slake-durability index of less than 80 from a two-cycle test):

Shale	This Classification	Deo's Classification
New Providence	Hard and durable	Soil-like
Mansfield	Soft and nondurable	Soil-like
New Albany	Hard and durable	Rocklike
Attica	Hard and durable	Soil-like
Palestine	Soft and nondurable	Soil-like
Lower Hardinsburg	Soft and nondurable	Soil-like

Figure 8. Estimated PLS index from pore-size parameters versus measured PLS index.



Thus, the parameters from the study of the pore-size distribution of shales--namely, cumulative porosity, median diameter, and spread factor--can be used to predict the durability of these Indiana shales. The pore-size parameters and the PLS index can be used to classify the shales with regard to durability and strength, respectively (Figure 8). Correlation with field performance is of course needed.

### ACKNOWLEDGMENT

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