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Time-Dependent Behavior of Coal Measure Rocks: Adsorption Rate and Strength Degradation

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

h	hour	MPa	Megapascal
m	meter	pct	percent
mm	millimeter	psi	pound per square inch

TIME-DEPENDENT BEHAVIOR OF COAL MEASURE ROCKS: ADSORPTION RATE AND STRENGTH DEGRADATION

By Sathit Tandanand[1]

ABSTRACT

This report presents a Bureau of Mines study on the time-dependent behavior of coal measure rocks for future implementation in stability analysis and safety evaluation of in-mine rock structures. This initial study focused on moisture adsorption rate and strength degradation of shales in Pennsylvanian coal-bearing strata in the Illinois Basin. A reaction of clay minerals in Illinois shales with atmospheric water vapor was hypothesized as the adsorption and strength degradation mechanism. Part of energy exchange between shales and the environment was attributed to a free energy controlled by pore pressure of the rock and partial pressure in the environment. The relationships between adsorption versus time and strength degradation versus moisture content were derived and correlated with the experimental data. Partial saturation at various levels of relative humidity occurs within approximately 2 weeks at room temperature. The strength of shales tested decreases linearly with increasing moisture content. When the moisture exceeds 7 pct of the dry weight, these Illinois shales become unstable. To this end, the moisture content is a good predictor of rock strength for shales.

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INTRODUCTION

Strength degradation of shales caused by moisture and water gives rise to persistent roof falls in underground coal mines. Although the need for geologic and engineering data for improving ground control procedures has been emphasized (1),[2] at present no specific design methods for moisture-sensitive rocks and swelling ground have been advanced beyond the state of the art (2). To develop methods for stability analysis and safety evaluation of in-mine rock structures, data on the material properties, stress-strain relationship, failure mechanisms, and failure criterion are necessary. Lack of this information currently is a stumbling block in advancing stability design for reliable solutions to ground control problems.

In the past decade, the Bureau of Mines has conducted studies on the effects of moisture on shales in the laboratory and in the mine (3-7). However, no constitutive relations have been derived to express time-dependent behavior of shales. As part of research efforts at the Twin Cities Research Center, the behavior of shales was further examined. In addition to the Bureau's work, related work conducted elsewhere (8) has contributed to a better understanding of weak rocks. However, knowledge of shales is still incomplete.

In this report, adsorption rate and strength degradation models for shales are determined. The moisture adsorption rate is formulated by introducing the laws of thermodynamics into the consideration. A new discipline in material science, continuum damage mechanics (9-11), is also introduced to describe the effects of moisture on reductions of elasticity and strength. Correlations and verifications of the models are made with experimental results. This study was conducted subsequent to that of the constitutive relations of coal measure rocks under dry conditions (12).

TIME-DEPENDENT BEHAVIOR

Time-dependent behavior of engineering materials is due hypothetically to the response of dissipative elements that produce a viscous deformation. Such behavior has been idealized and simulated by mechanical models that can be translated into constitutive models describing the stress-strain relations. In a simple idealization to help understand the time-dependent behavior, a spring and a dashpot connected in series or in parallel are used as basic elements. The spring represents an elastic response, whereas the dashpot represents a slow, viscous response in materials. The deformation of the system is controlled by the spring stiffness and the viscosity constant of the dashpot. The spring and the dashpot connected in series called the Maxwell model (fig. 1) is adopted for discussion. If the system is stretched by a force and the total elongation e is held constant, the dashpot absorbs the energy from the spring and slowly moves as time goes on until the energy in the spring vanishes. A mathematical model for stress relaxation can be derived from the deformation and the deformation rate in the system. Likewise, the strain-time relationship for creep can be determined from this mechanical model.

Describing complicated responses of a material to other influences requires further consideration of the interaction between the material and influential factors. In the present study, the interaction between rocks and moisture in the atmosphere, under isothermal condition, is taken into consideration. Creep and relaxation that occur simultaneously with

[2]Underlined numbers in parentheses refer to items in the list of references preceding the appendix.
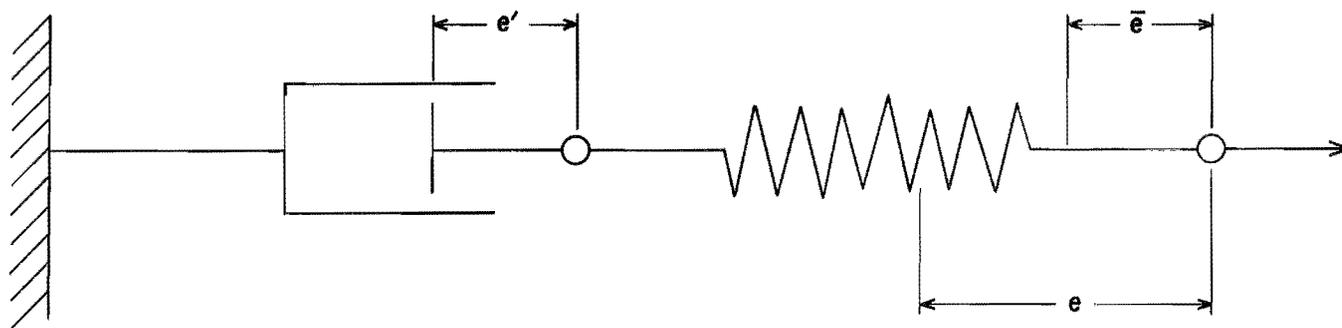
FIGURE 1.—Maxwell model. See appendix for explanation of symbols.

moisture are excluded. Physically, moisture adsorption is dependent upon several variables such as pressure, temperature, and volume of water vapor. In developing a unique relationship to describe the adsorption rate, the parameters that uniquely define the equilibrium condition of adsorption are selected. This primary phase of time-dependent behavior of rocks is treated in detail as follows.

MOISTURE ADSORPTION AND WATER ABSORPTION

Adsorption and absorption are different according to the substance assimilated by a solid. Absorption refers to suction of water by rocks directly in contact with water. This phenomenon is attributed to the pore pressure differences between two points. Absorption causes capillary rise and water movement in the pores, but at a low velocity. Absorption capacity of a rock is dependent upon surface area, pore volume, and initial water content, which can be measured by the pF scale based on the value of the common logarithm of the measured height in centimeters of water column (13). Water absorption is outside the scope of this report and is addressed only to distinguish it from moisture adsorption, on which this report is focused.

Adsorption refers to the physical and chemical attraction between gases or vapors and the surface area of rocks. Quantitatively, the process is expressed in terms of the amount of gas adsorbed per gram of adsorbent and the corresponding pressure in the gas phase, at a given temperature. The relationship between the adsorbed gas and pressure at a given temperature is referred to as adsorption isotherm. In coal measure rocks, especially shales, adsorption occurs because shales often contain montmorillonite and illite, which are sensitive to moisture. Attraction of water vapor to these clay minerals and interaction of their molecules are the basic mechanisms of swelling, shrinking, and strength degradation. In the presence of moisture, the rock adsorbs or desorbs water molecules through its surface. This process normally is reversible, especially when the pressure and temperature bounding the surface change. Capillary condensation of the adsorbed water vapor in the voids or in small solid capillaries may occur when temperatures fall below the normal condensation temperature. Chemisorption also takes place when the clay and water molecules mutually react. The microscopic attraction mechanism in the clay-water system, however, is not clearly known. Hydrogen bonding, cation hydration, attraction by osmosis, and the van de Waal forces are suggested mechanisms (14, p. 103).

MOSITURE ADSORPTION RATE

Moisture adsorption by shales is a rate-dependent process where pressure, volume, and temperature of the moisture are controlled parameters that can be described by a set of kinematical coordinates. The intrinsic properties, namely internal energy, entropy, and enthalpy, are thermodynamic quantities and dependent upon the controlled parameters. The first law of thermodynamics states that a small change in internal total energy dU is the sum of the small work done on the system δW and the small heat supply δQ such that

$$dU = \delta W + \delta Q. \qquad (1)$$

In a quasi-static process in which the temperature tends to be uniform and the pressure P is in equilibrium, work δW in a quasi-static volume change dV against a pressure P is determined by PdV; thus

$$dU = \delta Q - PdV. \qquad (2)$$

For a reversible process, the second law of thermodynamics provides the small quantity of heat in terms of a change of the entropy supply dS and the absolute temperature T at which δQ is transformed to give δQ = TdS. Hence, the following relationship is obtained:

$$dU = TdS - PdV. \qquad (3)$$

The other state variable of the system, called the free energy, Ψ, is introduced at this point. This quantity is the difference of the internal energy and quantity of heat, i.e., $\Psi = U - TS$, which provides the relationship

$$d\Psi = dU - TdS. \qquad (4)$$

It follows from equations 3 and 4 that

$$d\Psi = -PdV. \qquad (5)$$

In an isothermal, reversible process, $d\Psi$ equals the work done on the system. This relationship provides a theoretical background for considering the amount of moisture adsorbed by shales from the environment.

To formulate a relationship for the moisture adsorption rate, a rock sample of a finite volume is considered. The amount of moisture adsorbed is determined by the increasing weight of the sample. In the process, pressure, volume, and temperature of the water vapor in the pores may change. The change, however, is gradual and related to the change in free energy $d\Psi$. If the pore space is regarded as an isolated system, so that it has no heat content, the process will depend upon the difference between the partial pressure of moisture and the pore pressure.

Next, two assumptions are made: First, the free energy in the system is linearly proportional to the weight of adsorbed moisture w; i.e., $\Psi = \alpha w$, where α is the proportionality constant. At any time t it follows that the free energy diffusion rate is proportional to the moisture adsorption rate

$$\overset{\circ}{\Psi} = \alpha \overset{\circ}{w}, \qquad (6)$$

and at saturation

$$\Psi_s = \alpha w_s. \qquad (7)$$

The dot denotes the time derivative, and the 's' subscript denotes saturation.

Second, since the adsorption process is of physicochemical nature, the free energy diffusion rate is assumed to be proportional to the amount of free energy remaining; i.e.,

$$\overset{\circ}{\Psi} = c_o(\Psi_s - \Psi). \qquad (8)$$

Hence, one can find the adsorption rate

$$\overset{\circ}{w} = c(w_s - w) \qquad (9)$$

where c and c_o are proportionality constants.

The weight of moisture adsorbed at time t can be obtained by rearranging equation 9 and integrating $dw/(w_s - w) = c\,dt$, for $w = w_o$ at $t = o$, which gives

$$w = w_s [1 - (1 - w_o/w_s) \exp (-ct)]. \quad (10)$$

The weight of moisture approaches saturation w_s when t continuously increases, or $t = t_s$. This saturation time is determined experimentally.

ADSORPTION ISOTHERM

In an enclosed space, water vapor is saturated if the vapor pressure reaches an equilibrium and remains constant. The vapor pressure, however, is temperature dependent and can increase with increasing temperature. To determine a relationship between the amount of moisture adsorbed and the vapor pressure at saturation under a given temperature, the water vapor in the air is regarded as an ideal gas so that work, PV, is constant at a constant temperature. If there is a change in pressure and volume at saturation due to a change of energy, then

$$-PdV = VdP.$$

It follows that, at saturation,

$$-PdV = \alpha dw_s = V_s dP. \quad (11)$$

The saturated volume of vapor V_s is determined by the weight of moisture w_s and the vapor density m, from which

$$dw_s = \frac{w_s}{\alpha m}\, dP', \quad (12)$$

where P' is the adsorption pressure.

The quantity αm may be regarded as the energy density in adsorption. The solution of equation 12 is obtained by

rearranging and integrating with the initial value $w = w_o$, when $P' = 0$:

$$w_s = w_o \exp (P'/\alpha m). \quad (13)$$

Note that the adsorption pressure P' is the difference between the partial pressure of water vapor in the air P_a and the initial pore pressure P_i in the rock in which adsorption takes place; i.e.,

$$P' = (P_a - P_i).$$

Since the relative humidity in the air is measured by the ratio of the partial pressure of water vapor to the saturation pressure at the existing temperature, the adsorption isotherm at saturation can be expressed in terms of the relative humidity in the air as follows.

From equation 13,

$$w_s = w_o \exp [(P_a - P_i)/\alpha m]; \quad (14)$$

since $\phi = P_a/P_s$,

$$w_s = w_o \exp [P_s(\phi - P_s - P_i)/\alpha m], \quad (15)$$

where P_s is the saturation pressure at the existing temperature, and ϕ is the relative humidity.

STRENGTH DEGRADATION

Shales contain structural defects in the form of pores, cracks, joints, and bedding planes, which affect their strength. The tangent modulus and uniaxial compressive strength are experimentally found to be dependent on the relative humidity (3) and moisture content (15). To determine the effect of moisture on the elasticity and strength, the concept of continuum damage mechanics (CDM) is introduced. This concept was originated (9-11) to model the strength of materials by introducing a variable to define damage characterized by the density of cracks on a plane cross section.

For a uniform bar with a constant cross section under a uniaxial load, damage is assumed to initiate from void formation in a plane perpendicular to the loading axis. The ratio of damage to the intact cross-section area is regarded as damage factor, ω; thus,

$$\omega = \frac{A_d}{A_o} = \frac{A_o - A_e}{A_o}, \qquad (16)$$

where A_d is the damage area, A_o is the original area, and A_e is the effective, or load-transmitting, area of the cross section. Under an applied load, the nominal stress σ and the effective or true stress s are related by

$$\sigma A_o = s \, A_e,$$

from which

$$\sigma = s \, A_e/A_o = (1 - \omega)s. \qquad (17)$$

The relations between stress, strain, and damage can be developed as follows. For one-dimensional linear elasticity, the effective stress s is linearly proportional to the axial strain ε,

$$s = E\varepsilon,$$

and

$$\omega = s/D, \text{ for } S > o \qquad (18)$$

where D is defined as damage modulus.

It follows from equations 17 and 18 that

$$\sigma = (1 - \omega)s = E\varepsilon \, (1 - E\varepsilon/D) \qquad (19)$$

For nondamaged material, $\omega = 0$ and $D = \infty$; therefore $\sigma = E\varepsilon$.

The maximum load-carrying capacity or strength of the bar can be determined from the condition $d\sigma/d\varepsilon = 0$, which gives s, σ and ω at failure as follows:

$$s_f = D/2, \quad \omega_f = 1/2, \text{ and } \sigma_f = D/4.$$

Gradual degradation of brittle materials occurs as a result of a complex interaction of microcrack formation and plastic flow, especially under high hydrostatic pressure. Theoretically, the constitutive relations defining dissipation potential involving fracture and flow mechanisms are rather complicated (10-11). In applying the CDM to the current study, and for simplicity, the damage variable under a monotonic loading is considered to be related to the effective stress and the free energy produced by the clay minerals and water vapor. In this case, the damage factor is a function of the effective stress and moisture content, i.e., $\omega = f(s)$ and $\omega = g(w)$.

To determine a damage caused by moisture, it is assumed that the free energy in the rock is the mechanism that supplies an energy to assist crack extension. Consider a small element in rock of one unit thickness with a crack of length a. Theoretically, the resisting force of the element R_o equals the energy for crack extension per unit length dW/da. The energy W is the difference between work supplied by an external force F and an elastic energy in the element u. Hence,

$$R_o = \frac{dW}{du} = \frac{d}{du} (F-u). \qquad (20)$$

In the presence of moisture, the resisting force R_o is reduced as the result of dissipation of the free energy in crack extension, which in this case is $d\psi/da$. Therefore,

$$R_o - \Delta R = \frac{d}{da} (F - u) - \frac{d\psi}{da}. \qquad (21)$$

From equations 20, 21, and 6, one can find

$$\Delta R = \frac{d\psi}{da} = \frac{\alpha dw}{da}. \qquad (22)$$

An approximate relationship of $\omega = g(w)$ can be obtained by assuming that ω is a linear function of moisture content in the rock at a room temperature; hence let $g(w)$ be of the form

$$\omega = \zeta w/w_m \qquad (23)$$

where ζ is a nondimensional constant and w_m is maximum moisture content that the rock can hold. It follows that $\omega = 0$ for $w = 0$, and $\omega = \zeta$ for $w = w_m$. For an elastic rock, the tangent molulus (of elasticity) of rock with moisture E_t can be determined by equation 19, which gives

$$E_t = \frac{d\sigma}{d\varepsilon} = E_o \left(1 - 2\omega\right)$$

$$= E_o \left(1 - 2\frac{\zeta w}{w_m}\right), \qquad (24)$$

where E_o is the tangent modulus of dry rock.

Similarly, the uniaxial compressive strength of rock with moisture σ_c' can be expressed as

$$\sigma_c' = (1 - \omega_f) \sigma_c. \qquad (25)$$

In this case, ω_f is the damage factor defining the strength reduction due to moisture. In terms of moisture content

$$\sigma_c' = (1 - \zeta'w/w_m) \sigma_c, \qquad (26)$$

where ζ' is nondimensional constant; $\zeta' = 2\zeta$ (in equation 24) if the stress-strain relation is linear. For dry rocks $w = 0$, $\sigma_c' = \sigma_c$. When the moisture content reaches w_m, $\sigma_c' = (1 - \zeta') \sigma_c$. In this situation, the degradation reaches its limit.

DATA CORRELATION

Data published previously by Augenbaugh and Bruzewski (3) for the Bureau of Mines are used to examine the adsorption rate and adsorption isotherm of shales. Data obtained by Bauer (15) of the Illinois State Geological Survey are used to examine the strength degradation due to moisture.

ADSORPTION RATE

Table 1 lists shale samples obtained by Augenbaugh and Bruzewski (3) from mine roofs above the Harrisburg (No. 5) and Herrin (No. 6) seams in the Illinois Coal Basin. Empirical constants A' and B' and the correlation factor r, in the table, are discussed later. Each sample (5/8-in diam) was dried for 1 day, weighed, and placed in a separate dessicator. The relative humidity in the separate dessicators ranged from 10 to 100 pct. During the test, the temperature was kept constant. The results of the adsorption due to confining the samples are shown in figure 2.

To correlate the theory given by equation 10 with these test results, the ratio of the real time t to the saturation time t_s is used. The constant c in equation 10 becomes $c' = ct_s$, which is dimensionless. The saturation time, 288 h (12 days), was selected for t_s, and the moisture content at saturation corresponding to t_s was obtained from figure 2. The correlation of the theory and the experimental results is shown in figure 3. Empirically, the experimental results can be expressed as

$$\ln(1 - w/w_s) = \ln A - c't/t_s.$$

The values of constants A, c', and the correlation factor r at various relative humidity levels are summarized in table 2. The constant A may be interpreted as the ratio of adsorption potential to the saturated moisture content. The value of A implies that each rock sample contains some moisture.

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TABLE 1. – Shale samples and empirical constants

| Sample	Source	Location[1]	A'	B'	r
1	Little Dog Mine..........	Gillespie...............	1.847	0.016	0.985
2	Peabody River King.......	Freeburg................	1.308	.017	.963
3	Monterey.................	Carlinville.............	2.000	.013	.987
4	Old Ben Mine #26.........	Sesser..................	2.495	.009	.941
5	Hillsboro Mine #63.......	Coffeen.................	2.263	.010	.959
6	..do.....................	..do....................	.161	.038	.962
7	Inland Mine..............	Sesser..................	2.388	.007	.896
8	Orient Mine #4...........	Johnson City............	2.544	.008	.966
9	Sahara Mine #20..........	Harrisburg..............	2.423	.007	.932
10[2]	Peabody River Queen......	Central City............	.549	.024	.945
11[2]	Sahara Mine #20..........	Harrisburg..............	1.633	.010	.965
12[2]	Peabody Eagle............	Shawneetown.............	2.752	.003	.987

[1]All in Illinois.   [2]Coal seam 5.
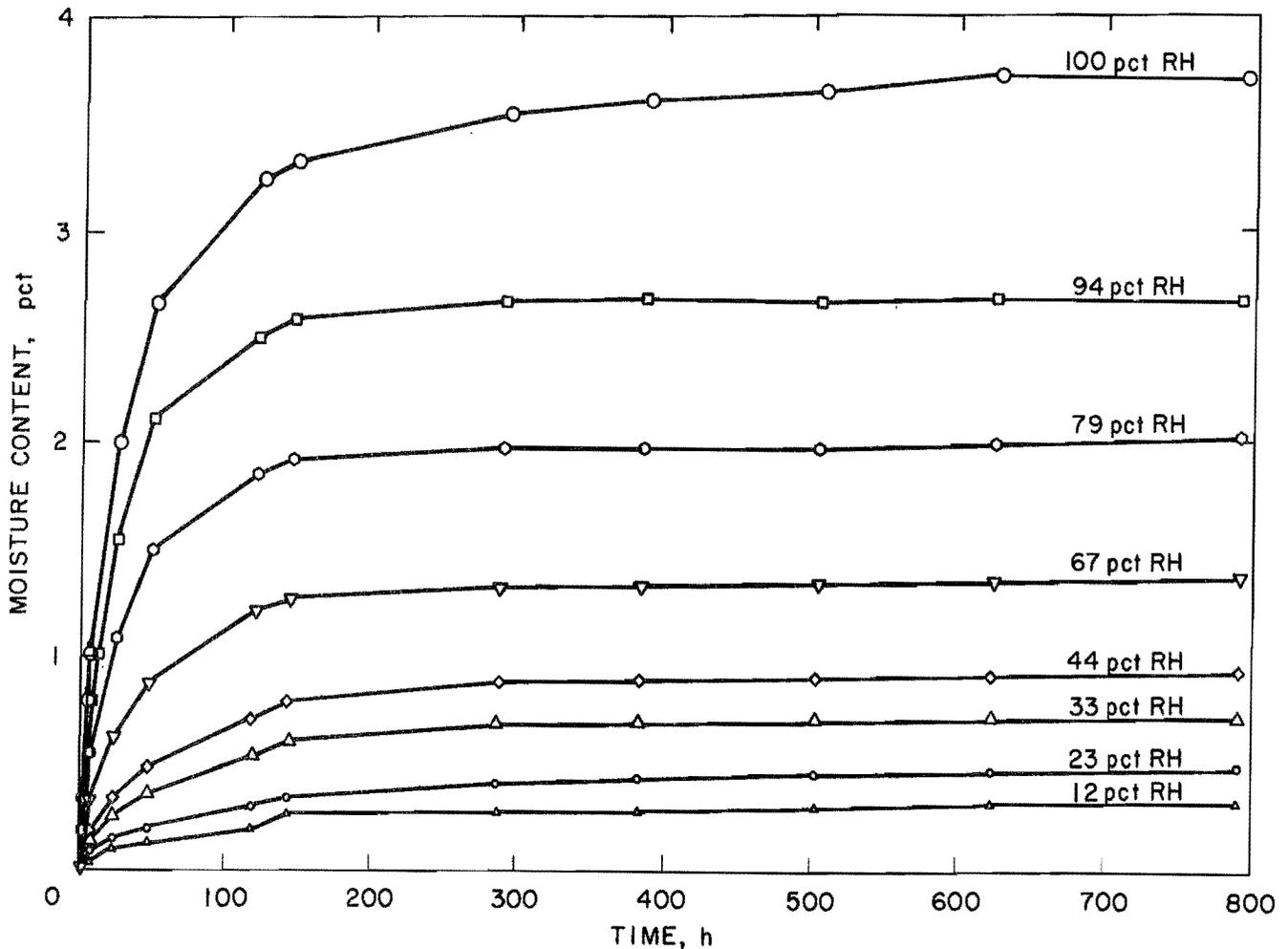
FIGURE 2.—Moisture adsorption rates by shale at various relative humidity levels and a constant room temperature.

ADSORPTION ISOTHERMS

Moisture contents in the samples at the equilibrium state, with respect to the relative humidity change, are shown in figure 4. These curves represent adsorption isotherms of the samples from the same coal seam, as expressed by equation 15. The "null point" at a certain relative humidity level is indicated by the zero gain where the adsorption is in an equilibrium state. Theoretically, the adsorption is dependent on the initial

TABLE 2. - Adsorption constants of Illinois shale

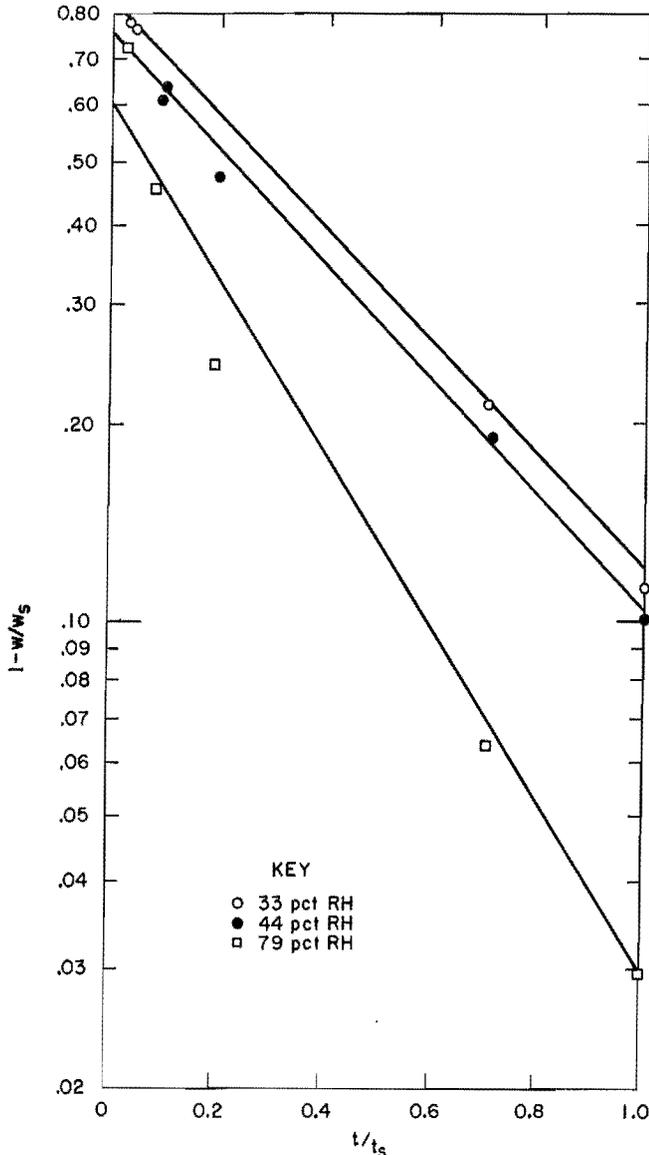| ϕ	w_s, pct	Empirical constants		r
		A	c'	
12	.29	0.88	2.19	0.95
23	.42	.75	1.41	.99
33	.70	.76	1.87	.99
44	.89	.76	2.00	.99
67	1.33	.69	2.83	.99
79	1.97	.60	3.06	.99

FIGURE 3.—Selected linearized time-dependent adsorption of shale at room temperature, at three humidity levels.
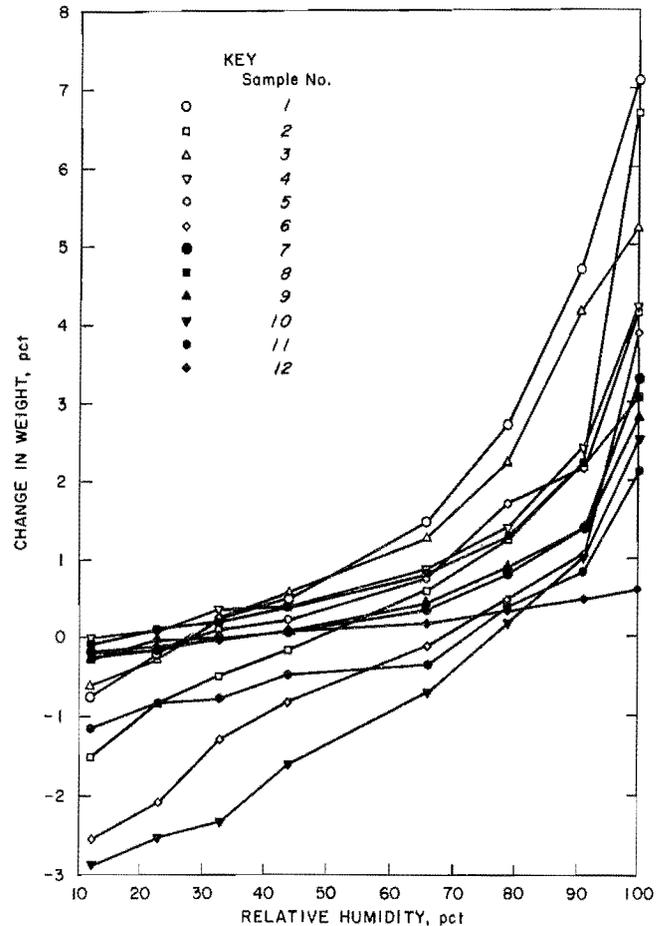

FIGURE 4.—Weight gain and loss of moisture content in the samples at the equilibrium state with respect to the relative humidity change.

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moisture content in the rock. As the relative humidity increases, the adsorption increases exponentially. To obtain the values of empirical constants, the null point was shifted to -3 to facilitate the semilogarithmic plot. Hence, $w_s' = w_s + 3$, and $w_s' = A' \exp B'\phi$, where A' and B' are the empirical constants for the samples shown in table 1. The average of percent change in weight of all samples versus the relative humidity is shown in figure 5. A good correlation between the theory and the experimental results is obtained, but the empirical constants are not explicit because the initial moisture content in each sample is unknown.
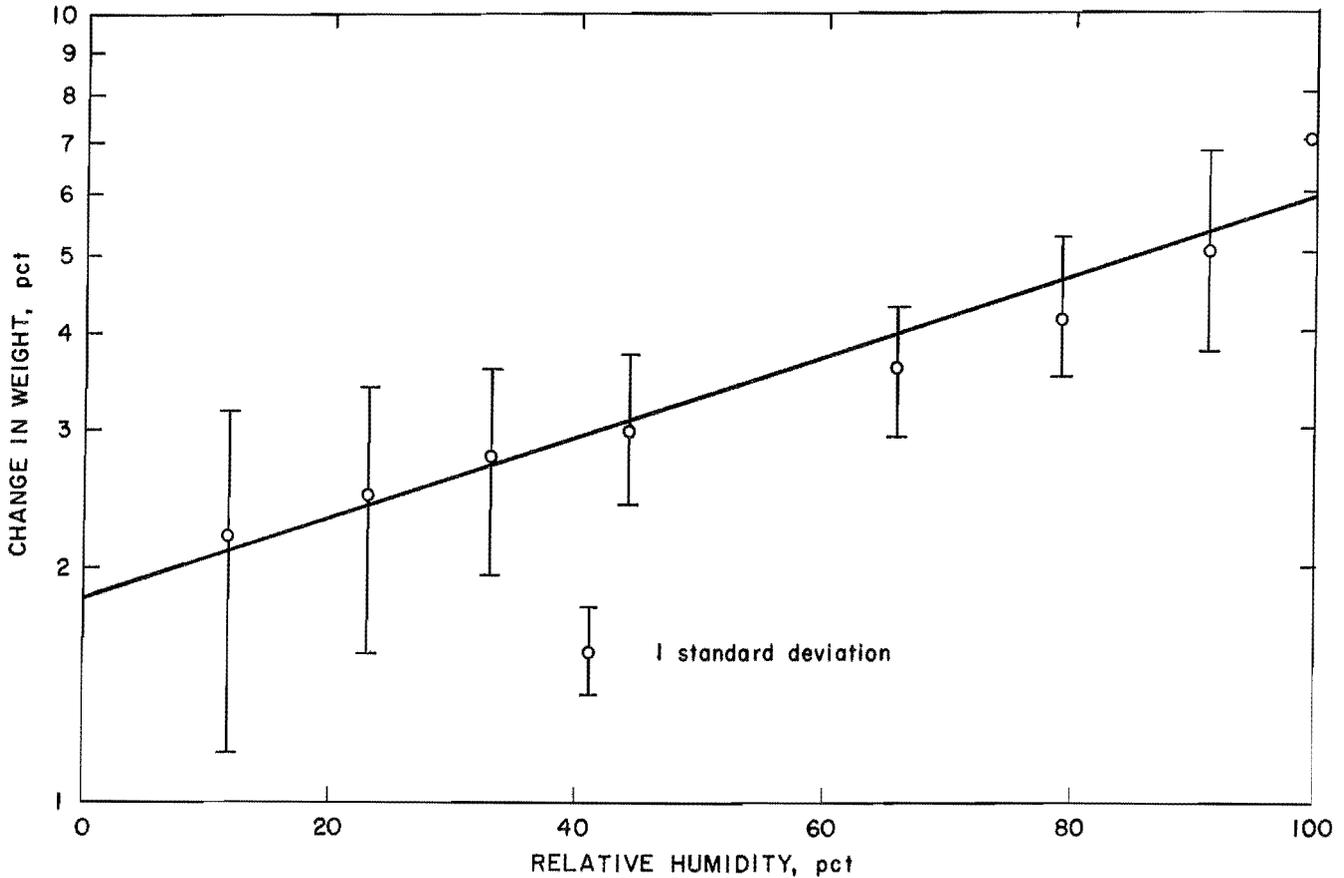

FIGURE 5.—Average of percent change in weight of all samples versus the relative humidity.

STRENGTH DEGRADATION

Typical stress-strain curves of gray shale under uniaxial compression at various moisture contents are shown in figure 6. The shale samples were obtained from a surface mine in southern Illinois approximately 35 m below the surface. These 25.4- by 50.8-mm samples were placed in a dessicator containing 100 pct relative humidity. The adsorption time of each sample ranged from 4 to 18 days. The reductions of the tangent modulus and the strength are shown by figure 6. The decrease of uniaxial compressive strength is linear with the increase of the critical strain as shown by the dotted line representing the locus of strength degradation with respect to the adsorption time.

To show that the tangent modulus and the compressive strength of three Illinois shales is a linear function of moisture content, a linear regression was made using the experimental data obtained by Bauer (15) from the Illinois State Geological Survey. The correlation results are shown in figures 7-12. The intercept of the regression line gives the compression strength or tangent modulus for the dry state. The slope of the line gives the value of $2\zeta/w_m$ for the tangent modulus and the value of ζ'/w_m for the compressive strength. These two constants have units "per percent" of moisture content. The strength reduction of the shales can be determined from the slope of the reduction line. Energy, Purington and Farmington Shales reduce in

strength by 80 pct when the moisture content increases to approximately 5.6, 6.7, and 7.3 pct, respectively.
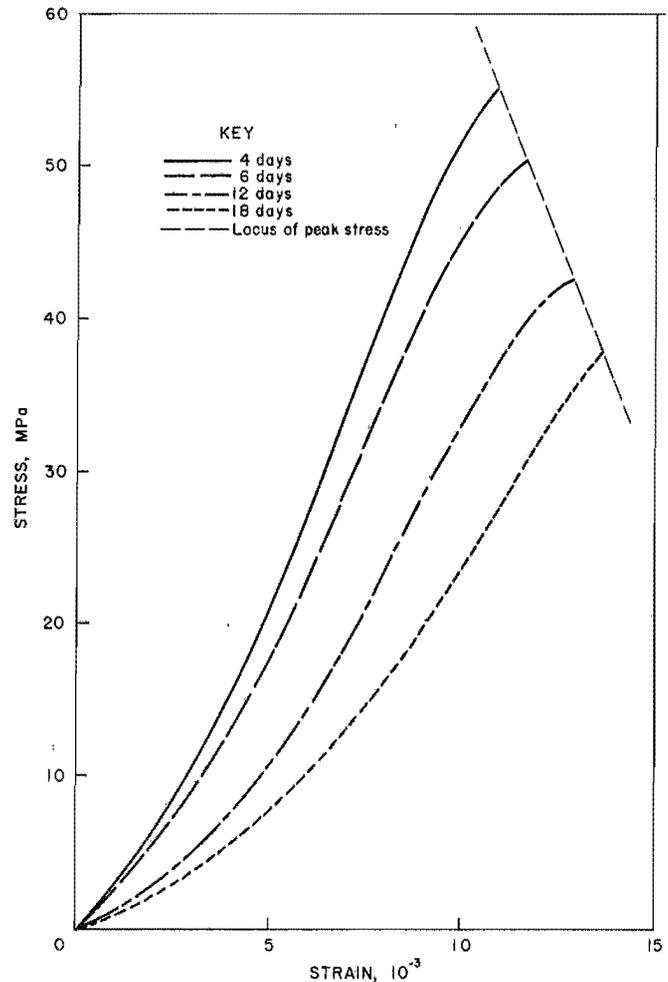

FIGURE 6.—Typical stress-strain curves of gray shale under uniaxial compression at various moisture contents.
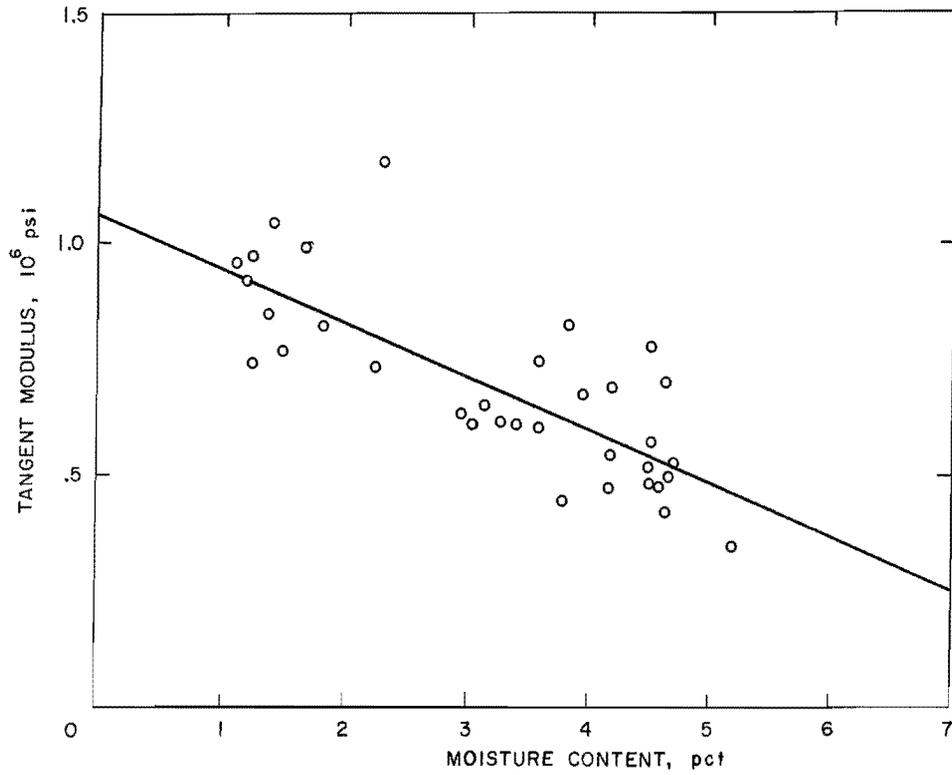
12

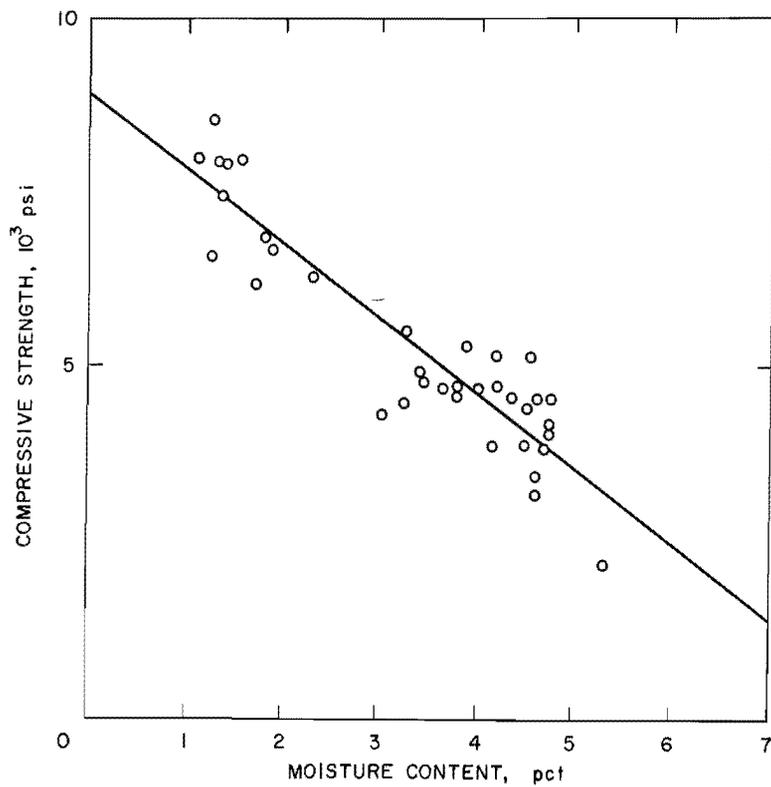FIGURE 7.—Linear relationship of tangent modulus of the Purington Shale versus moisture content.

FIGURE 8.—Linear relationship of compressive strength of the Purington Shale versus moisture content.
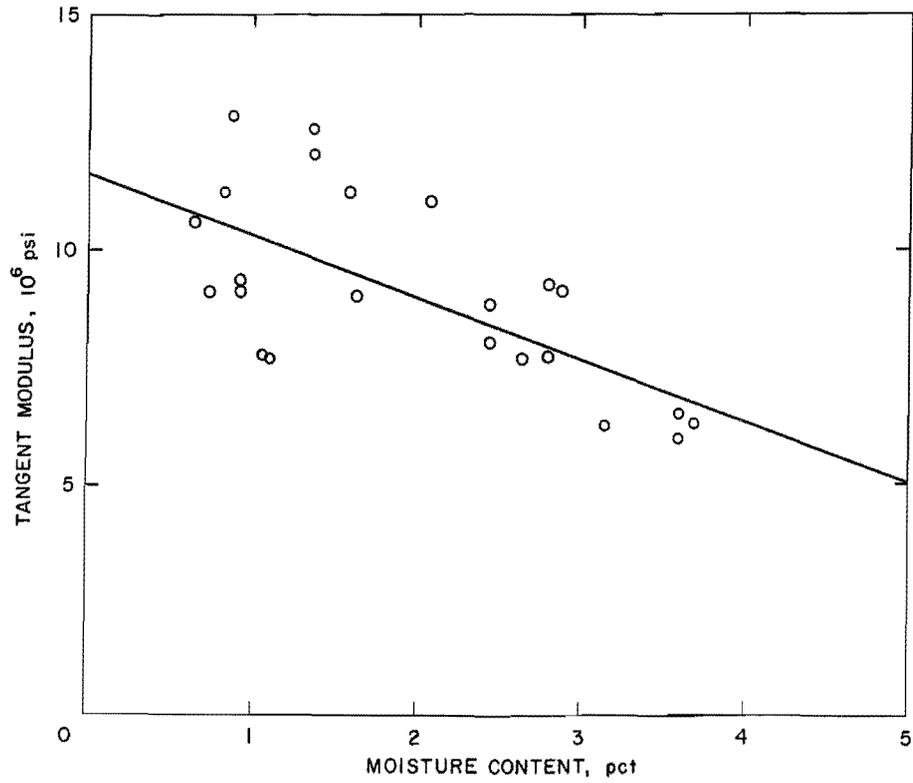
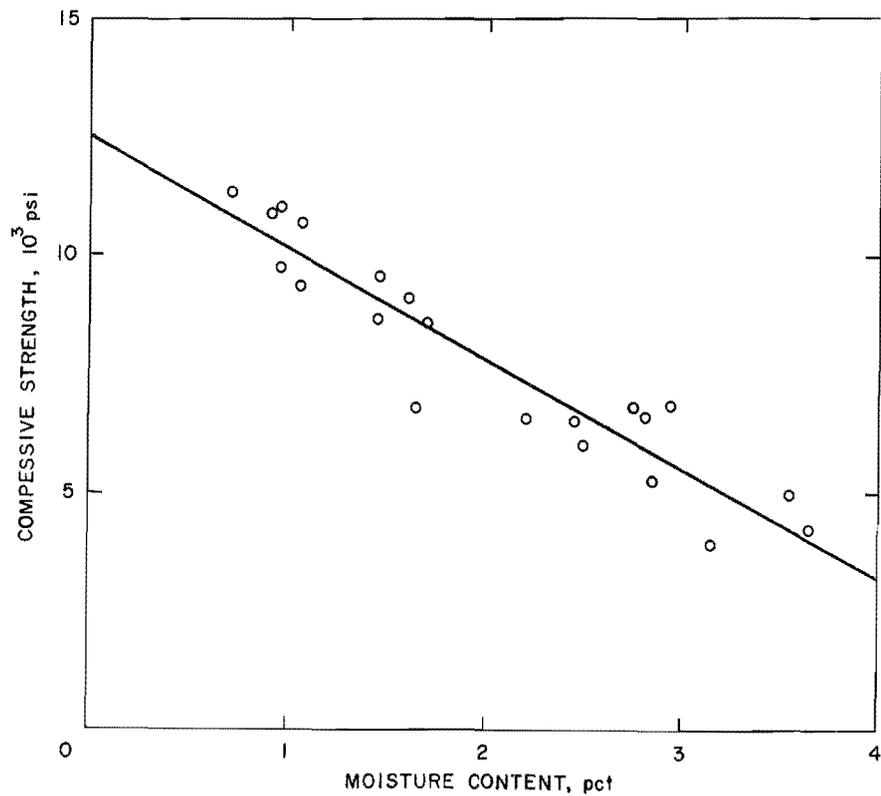FIGURE 9.—Linear relationship of tangent modulus of the Energy Shale versus moisture content.

FIGURE 10.—Linear relationship of compressive strength of the Energy Shale versus moisture content.
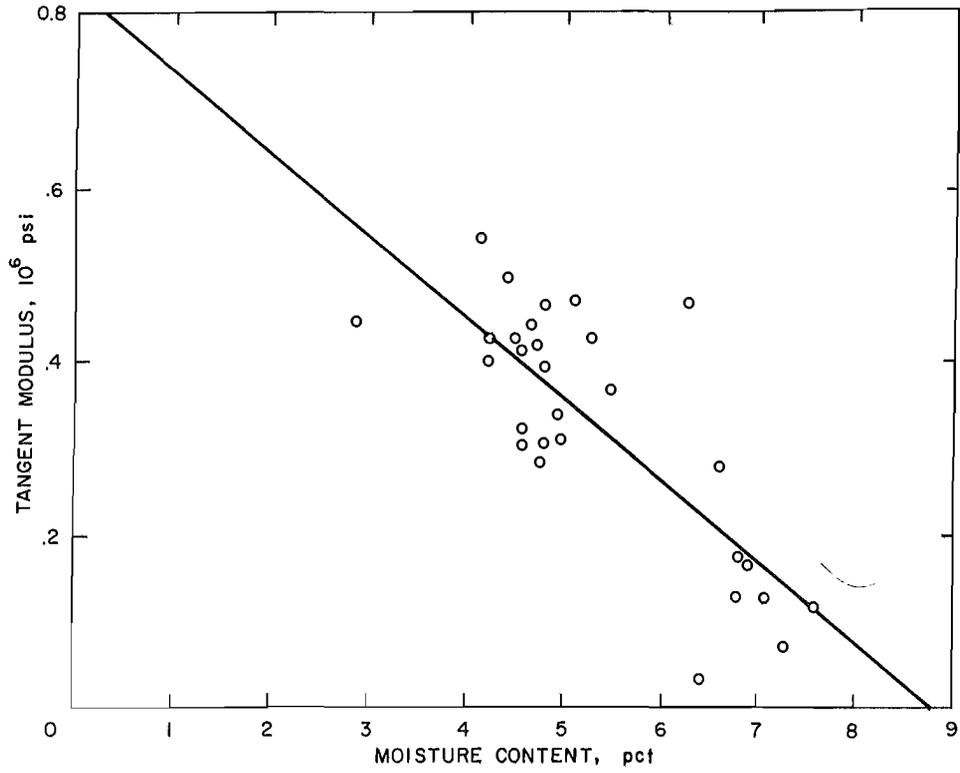
FIGURE 11.—Linear relationship of tangent modulus of the Farmington Shale versus moisture content.
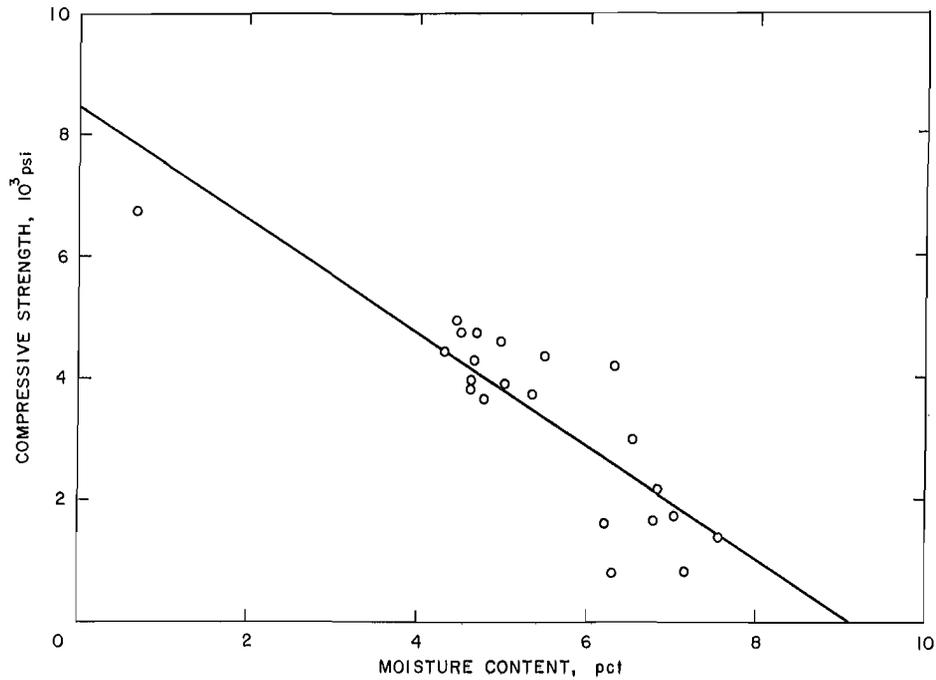

FIGURE 12.—Linear relationship of compressive strength of the Farmington Shale versus moisture content.

CONCLUSIONS

Equations describing the adsorption rate, adsorption isotherm, and strength degradation of shales are derived from an adsorption mechanism of energy exchange between clay minerals and moisture in the air that controls the adsorption-desorption process. The free energy generated in the process is a contributing factor to strength degradation. The equations used are restricted to the simplest case, i.e., the isothermal process in which the free energy is a function of pressure and volume of water vapor.

Experimentally, the adsorption period for shale samples is approximately 2 weeks, i.e., between 12 to 18 days to saturation at various relative humidity levels. Both the tangent modulus and the compressive strength of Illinois shales are shown to decrease linearly with increase in moisture content. Results show that strength of Illinois shales can be reduced by 80 pct if the moisture content is increased by about 6 to 7 pct over the dry weight. To this end, moisture content in shales is a good indicator of strength degradation. More fundamental studies of the time-dependent behavior of shales under various states of stress, temperatures, and sustained long-term loadings are suggested for developing constitutive relations for ground control applications.

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APPENDIX.—NOTATIONS

A	adsorption constant
A'	empirical constant
A_d	damage area
A_e	effective area
A_o	undamaged area cross section
a	crack length
B'	empirical constant
c, c', c_o	constants
D	damage modulus
d	small change
e	total elongation
e'	elongation of dashpot
e	elongation of spring
E_o	tangent modulus, dry condition
E_t	tangent modulus
F	work done by external load
f	failure
m	moisture density
P	pressure
P'	adsorption pressure
p_a	partial pressure of water vapor
p_i	pore pressure
p_s	saturated pressure
Q	quantity of heat

R_o	resistance of dry rock
r	coefficient of correlation
S	entropy
s	actual stress
T	temperature
t	time
t_s	saturation time
U	total energy
V	volume
V_s	saturated volume of vapor
W	quantity of work, energy
w	moisture content
w_m	maximum moisture content
w_o	initial moisture content
w_s	moisture content at saturation
α	proportionality constant
δ	small quantity
ε	strain
ζ, ζ'	dimensionless constant
σ	stress
σ_c	compressive strength
Ψ	free energy
Ψ_s	free energy at saturation
ϕ	relative humidity
ω	damage factor