Mechanisms of Strength Loss during Wetting and Drying of Pierre Shale

Mécanismes de la perte de force pendant humidification et séchage de *Pierre Shale*

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ABSTRACT: The physio-chemical and morphological role on the residual strength of Pierre Shale during wetting and drying cycles was investigated. Ring-shear tests were conducted on intact and cycled material to assess the residual strength. The mineralogy and chemistry were determined from x-ray diffraction and x-ray fluorescence results. Minor mineralogical changes were observed during the cycling process. The material degraded from a firm, dense shale to a massive, clayey material after three to four cycles. Gypsum concentrations decreased during the wet-dry cycles. The low residual friction angles of 6.1 to 6.8 degrees decreased an additional 0.8 to 1.4 degrees during the wet-dry cycles. A significant fabric contrast was apparent after three cycles as the material's structure became more massive. The material with higher amounts of montmorillonite in the mixed-layer clay mineral showed little change in the liquid limits with cycling, in contrast to the illite materials. A decrease in residual strength was observed for the first two wet-dry cycles, but little change for successive wet-dry cycles. The results suggest the disintegration of particles during slaking is the main determinate of strength loss. The initial mineralogy was also observed to be a factor on the slaking rate and the residual strength behavior.

RÉSUMÉ : Le rôle physio-chimique et morphologique sur la résistance résiduelle de *Pierre Shale* au cours des cycles de mouillage et de séchage a été étudié . Les essais de cisaillement annulaires ont été effectués sur ce matériau intact et recyclé pour évaluer sa résistance résiduelle. La minéralogie et la chimie ont été étudiées à partir de diffraction des rayons X et de rayons X de fluorescence. Des modifications minéralogiques mineures ont été observées au cours des cycles hydriques. . Le schiste dense a subi une dégradation après 3 à 4 cycles et a été transformé en une argile. La concentration en gypse a également été diminuée. Les faibles angles de frottement interne résiduels de 6,1 à 6,8 degrés ont diminué d'environ 0,8 à 1,4 degrés supplémentaires au cours des cycles de mouillage-séchage. Une modification importante de la structure interne et une densification ont été notées après trois cycles. Les limites de liquidité du matériau avec un pourcentage élevé de montmorillonite n'ont pas été modifiées contrairement au matériau contenant de l'illite. Une diminution de la résistance résiduelle a été observée pour les deux premiers cycles hydriques, mais peu de changement a été observé pour des cycles ultérieurs de mouillage-séchage. Les résultats suggèrent que la désintégration des particules pendant l'humidification est la principale cause de perte de résistance. La minéralogie initiale a également été considérée comme un facteur important influençant cette désintégration et la résistance résiduelle.

KEYWORDS: Shale, weathering, strength, clay mineralogy, residual friction angle

1 INTRODUCTION

The tendency for clay shales to weather, soften, and slake upon drying and rewetting has been well documented. The degradation causes the material to soften and lose strength, possibly leading to slope failures. Skempton (1964) noted strength losses of up to 80% in some deposits after softening. The slaking rate has been observed to be dependent on the mineralogy and physico-chemical behavior, especially in materials with high activity clay minerals (Perry and Andrews 1984). While the mineralogy mechanisms are well known, very few studies on clay shales have been conducted to analyze the role of physico-chemical occurrences on the strength loss.

Weathering in overconsolidated clays and clay shales has been observed to be a significant process due to the mode of deposition and the bonding from diagenesis, especially in outcroppings. One of the largest and most problematic clay shales in the United States is the Pierre Shale Formation (Fleming *et al*. 1970). The drop in strength from the peak to residual strength is a source of the stability problems associated with these materials. Initial fissuring results from rebound in clay shales and leads to clay swelling, strain softening and weathering (Brooker and Peck 1993). Much of the past work on residual strength has focused on the mechanical aspects with less emphasis on the role of the more dynamic, physico-chemical effects on residual strength.

This paper reports the results of tests conducted to relate the strength of a series of laboratory wetting and drying cycles on unweathered Pierre Shale to its chemistry, mineralogy, and micromorphology. The study analyzes the role of the initial mineralogy, breakdown of particle size, and the overall changes in the mineralogy.

1 MATERIALS AND METHODS

1.1 *Materials*

As described by Bjerrum (1967), the behavior of overconsolidated materials is strongly correlated to their geologic history. Pierre Shale is a heavily overconsolidated clay shale formed from a marine/non-marine environment sedimentation during the Cretaceous Period approximately 60 to 80 million years ago (Fleming *et al.* 1970). The formation extends throughout Canada and as far south as the Gulf of Mexico. Significant slope failures have been observed throughout the formation, but are mainly focused in the upper Missouri and South Saskatchewan River basins. The mineralogy of Pierre Shale is primary clay minerals, specifically smectite and mixed-layer clays.

Unweathered samples were obtained from a boring at the Oahe Dam site, South Dakota, at depths below 60 meters. Firm shale, without any weathering, has an average dry density of 1.71 g/cm^3, a moisture content of 25%, and specific density of 2.7 at the site. Unweathered Pierre Shale at the site exhibits unconfined strengths of 0.5 to 17.4 MPa and a Young's modulus between 137 and 965 MPa (Johns *et al.* 1963). The wide variation in strength shows the engineering complications in classifying Pierre Shale as either a rock or soil. Extensive problems were encountered during the construction of the Oahe Dam due to the rapid strength loss in the material (Knight 1963). After many failures, slopes had to be redesigned with ϕ'=8.5 degrees and 14.4 kPa for cohesion (Johns *et al.* 1963). Weathering was observed to cause the durable, brittle, rock-like shale to turn into a weathered, soil-like material at the site. The peak strength of the weathered material was found to be ϕ'=11.9 degrees and 24.9 kPa for cohesion, but due to the possibility of slip surfaces in the shale, the residual strength controlled the design parameters. In a laboratory slaking test, Botts (1986) observed nearly a 75% drop in the shear strength of Pierre Shale samples, or a 6-degree drop in the internal friction angles and a reduction of the cohesion from 848 kPa to 0 kPa, after one wetting and drying cycle.

1.2 *Sample Preparation*

1.2.1 *Residual Shear Strength*

The testing procedure for the Bromhead Ring Shear device used a modified version of ASTM D6467. The material was crushed and passed through a US standard #50 sieve, remolded with distilled water to their plastic limit and allowed to hydrate for 48 to 72 hours. Higher water contents are suggested by ASTM (2003) but Bromhead (1979) advises drier samples in the Bromhead Ring Shear device to prevent excess settlement of the top platen.

1.2.2 *Micromorphology*

Central to the quality of any fabric analysis in soils is a preparation method preventing disturbance. Dry samples are required for analysis in the vacuum environment of the scanning electron microscope (SEM). The freeze-drying technique was used to obtain relatively undisturbed samples. A sample from each weathering cycle was placed in an intermediate freezer for twelve hours at a temperature of -75 degrees Celsius and then placed in a VirTis Ultra 35 8-shelf model freeze dryer at a temperature of -25 degrees Celsius until a vacuum of <100 millitorr was achieved. The temperature was raised to 26 degrees Celsius and held constant for 48 to 72 hours. Tension fractured and placement on carbon stubs preceded the analysis in the SEM.

1.2.3 *Mineralogical and Chemical Analysis*

X-ray fluorescence (XRF) and x-ray diffraction (XRD) analyses used both bulk samples and size fractions to determine the composition of the material. Bulk, silt, and sand mineralogy was obtained from random-oriented mount samples mounted in the XRD unit and analyzed at a speed of 2^{o} two theta per minute with copper K-alpha radiation. Minerals were identified by a computerized catalog of the Joint Committee on Powder Diffraction Standards (JCPDS) Powder Diffraction File system (The International Centre for Diffraction Data® 2004). Particle-size separation was performed on the material on the basis of Stoke's law, where particles <2 μm equivalent spherical diameter were obtained. This fraction was further separated by the same method into the coarse clay fraction, particles less than 2 μm, and the fine clay fraction, particles less than 0.5 μm. The oriented-mounted samples intensify the (001) reflections and reduce (hk0) reflections by removing non-platy minerals, dispersing clay minerals into individual colloidal particles, and laying the clay particles flat (Moore and Reynolds 1997). The filter transfer method, as recommended by Moore and Reynolds (1997), was used to transfer the material to a filter. The type of clay mineral is identified by the characteristic expansion, contraction, or collapse of the clay mineral's d-spacing through five subsequent treatments: air drying, glycolation with ethylene glycol, heating to 400°C, and heating to 550°C. The methods described in Shultz (1978) were used to estimate the concentrations of the clay minerals in the mixed-layer.

2 TESTING AND ANALYITICAL METHODS.

2.1 *Weathering Cycle*

ASTM standard C593 (ASTM 2003) was used as an outline for the saturation of the Pierre Shale. The vacuum-saturation strength testing procedure described in the standard was modified to allow for saturation of the soil samples. The test consisted of obtaining multiple two cubic-inch samples of unweathered Pierre Shale and placing them on a filter in a vacuum chamber. Samples were vacuumed at 24 inch Hg (11.8 psi) for one hour in the chamber; the chamber was then flooded; and the samples were soaked for one hour in the distilled water bath. The samples were removed from the bath and allowed to air-dry for 48 to 72 hours, completing one weathering cycle.

2.2 *Classification and Ring Shear*

Classification testing followed applicable American Society for Testing and Materials (ASTM 2003) standards. Atterberg limit and hydrometer analyses were performed on the samples. The Bromhead Ring Shear Device was used for measurement of the residual strength. The device was first proposed by Bromhead (1979) and has provided results that are in good agreement with back-calculations for slope stability analysis (Bromhead and Dixon 1986, Skempton 1985). The mold dimensions are 100-mm outer diameter, 70-mm inner diameter, and a thickness of 5-mm. Replicate testing was not conducted for the cycling studies due to sample limitations.

The prepared sample was molded into the ring at a water content near its plastic limit and placed in a distilled water bath. Multistage testing was used to obtain the residual strength. This technique uses progressive loading after the formation of a shear surface. The samples were initially consolidated at the normal stress of 100 kPa and then sheared at a rate of 0.16 degrees per minute (0.119 mm per minute) for a displacement of one revolution to form the shear surface. The rate was slowed down to 0.048 degrees per minute (0.036 mm per minute) and allowed to shear until the residual strength was reached, typically an additional 10 to 15 mm. The residual strength was obtained for 100, 200, and 400 kPa normal stress increments.

2.3 *Scanning Electron Microscopy*

Freeze-dried samples were analyzed with a Hitachi S-2460N VP scanning electron microscope (SEM) with energy dispersive spectrographic (EDS) and digital imaging capability. Two sources were used to obtain surface topography: backscattered electron (BSE) and secondary electron (SE). BSE mode was used for lower-resolution imaging and elemental analyses while SE mode was used for higher magnifications. Samples were coated with gold to prevent charging for samples analyzed in the SE mode. Digital elemental maps were produced using the EDS function on the SEM. Mineral contents were inferred by coinciding elemental maps.

3 RESULTS

3.1 *Engineering Properties*

Samples from three different depths below 60 meters were cycled through wetting and drying. Table 1 shows the index properties for the cycles. The material at the 63.0 and 70.3-meter depths consisted of approximately 2% sand, 50% silt, and 48% clay sized particles. The 63-meter samples exhibited a substantial increase in the liquid limit, as the material was wetted and dried. Minor increases in the liquid limit were observed in the 63.6-meter sample. The 70.3-meter sample showed insignificant changes in the Atterberg limits. The plastic limit increased by ten and five percent for the 63.0 and 63.6 meter cycles. The plastic limit remained almost unchanged for the 70.3 meter depth.

Table 1. Weathering cycle Atterberg limits.

Depth (M)	Depth (Ft)	Weath-ering Cycle	Liquid Limit (%)	Plastic Limit (%)	Plasticity Limit (%)
63.0	208	0	79	33	46
63.0	208	1	85	35	50
63.0	208	2	88	36	53
63.0	208	3	100	40	60
63.0	208	4	113	40	73
63.0	208	5	125	43	82
63.6	210	0	63	32	31
63.6	210	1	78	34	44
63.6	210	3	79	35	44
63.6	210	5	86	36	50
70.3	232	0	72	31	41
70.3	232	1	72	30	41
70.3	232	2	72	31	41
70.3	232	3	74	32	42
70.3	232	4	68	29	39
70.3	232	5	71	31	40

The residual friction angle for each cycle for the three depths was determined from ring-shear tests. The formulated residual strength-normal stress plot for each sample was constructed using a spreadsheet program and analyzed for both the trend line through the origin and with a cohesion intercept. The residual friction angle (ϕ'_r) and residual cohesion are summarized in Table 2. The residual friction angle dropped almost 1.5 degrees for the 63.0 meter depth, nearly one degree for the 63.6 meter sample, and 1.3 degrees for the 70.3 meter depth during the wet/dry cycles. An unexpected increase in strength was observed in the later stages of the 70.3 meter cycles. Similarities in the 63.6 and 70.3 meter residual friction angle plots are apparent for the first two cycles.

3.2 *SEM Images and Visual Observations*

The unweathered material below 60 meters showed dense, high-laminae fabric. The three depths analyzed had similar fabrics. Various other fabrics and particle shapes were observed over the depths including burrows of pyrite framboids and cemented calcite accretions. Weathering created a more homogenous fabric. Weathering led to a more open fabric.

Visual observations of yellow, sulfuric, sand particles scattered over the sample between the laminae were made. Fabrics with large amounts of yellow particles showed a significant amount of degradation after one to two wet/dry cycles as well. After the third cycle, the material turned into a soil-like material. As the material dried, large cracks were observed, an indicator of the shrink/swell potential of the clay minerals in the material.

Table 2. Wet/dry cycle residual friction angles

Depth (M)	Depth (ft)	Weather -ing cycle	ϕ_r' (°)	c_r' (kPa)	ϕ_r' for $c'=0$ (°)
63.0	208	0	6.8	0.7	6.9
63.0	208	1	6.5	~0	6.5
63.0	208	2	6.5	~0	6.3
63.0	208	3	6.0	0.5	6.1
63.0	208	4	5.6	0.1	5.6
63.0	208	5	5.4	~0	5.3
63.6	210	0	6.1	0.2	6.1
63.6	210	1	5.7	0.8	5.9
63.6	210	3	5.4	1.1	5.6
63.6	210	5	5.2	1.3	5.5
70.3	232	0	6.7	0.2	6.7
70.3	232	1	6.4	0.2	6.4
70.3	232	2	6.3	0.3	6.4
70.3	232	3	5.4	1.2	5.7
70.3	232	4	6.3	1.9	6.6
70.3	232	5	6.6	1.0	6.8

Following each weathering cycle, the sample was fractionated in a sedimentation cylinder to obtain the clay fraction for XRD analyses. Observations of the time rate of settlement of the particles during this process provide insight into the soils' behavior. Settlement analyses showed a contrast in the suspension time for wet/dry samples. The fine fraction of the wet/dry cycled samples stayed in suspension for many weeks longer than the un-cycled samples. A sample after five wet/dry cycles continued to be in suspension after three months.

The elemental composition of the materials was inferred from elemental maps of the EDS analyses. Elemental maps of the unweathered Pierre Shale showed K- and Na-ions are dispersed over the material, a possible indicator of the adsorbed cation on the montmorillonite mineral. The material has high aluminum, silicon, and oxygen suggesting clay minerals. The particles with high reflection were determined to be pyrite due to the occurrence of iron and sulfur in the bright particles.

3.3 *Mineralogy and Chemistry*

Bulk mineralogy was determined from powder samples. Sand, silt, clay, and very fine clay fractions were divided to determine the mineralogy of the fractions. Sand and silt mineralogy was determined by random-mount samples and clay mineralogy was found from oriented-mount samples. Bulk mineralogy indicated quartz ($\mathrm{SiO2}$), gypsum ($\mathrm{CaSO4\cdot 2H2O}$), and pyrite ($\mathrm{FeS2}$). The mineralogy was similar over the depths except for higher concentrations of gypsum at the 70.3 m depth.

The sand fraction was observed to be highly heterogeneous, with a significant amount of quartz. Large calcite accretions were observed visually in the bulk sample and were found in the sand fraction mineralogy. These observations were verified by EDS analyses on SEM specimens. Pyrite, gypsum, and bassanite ($\mathrm{CaSO4\cdot 1/2H2O}$) were also observed in the sand fraction. Gypsum is a hydrated form of bassanite. Quartz and pyrite were found in the silt fraction with minor amounts of feldspars including orthoclase ($\mathrm{KAlSi3O8}$) and albite ($\mathrm{NaAlSi3O8}$).

The clay minerals were primarily mixed-layer clays as well as minor amounts of illite clay mineral. Shultz (1978) provides an outline for the determination of mixed-layer clay minerals in Pierre Shale. The 63.0 meter depth showed a mixed-layer clay mineral with montmorillonite, illite, and beidellite concentrations of 20, 45, and 35 percent, respectively. The 70.3 meter depth had a mixed-layer clay mineral closer to bentonite with montmorillonite, illite, and beidellite concentrations of 60, 15, and 20 percent, respectively. Data and diffractograms for the mixed-layer clay determinations are shown in Birchmier (2005). These values fall within the data compiled by Schultz (1978) for mixed-layer clay minerals in Pierre Shale. Overlays of diffractograms of bulk samples for the wet/dry cycles of the 70.3 meter depth showed little change over the weathering cycles except for the reduction of gypsum particle size during the initial cycles. The combination of the decrease in the gypsum peak intensity at 7.63 Å, and the constant 4.28 Å and 3.8 Å peak intensities during cycling suggests the breakdown of the gypsum particles.

4 DISCUSSION

Residual friction angles for the wet/dry cycles decreased from 6.8° to 5.4°, 6.1° to 5.2°, and 6.7° to 5.4° for the depths 63.0, 63.6, and 70.3 m, respectively. A one degree drop in a material with an initial residual friction angle of 6.5° is significant. For a given normal stress and negligible residual cohesion, the factor of safety would be reduced by a ratio of 1.2. Exposure and removal of confining stresses during construction activities in Pierre Shale could cause wetting and drying to occur and lead to slope failures.

The residual friction angle for each cycle for the three depths was determined from ring-shear tests. The residual friction angle dropped almost 1.5 degrees for the 63.0 meter depth, nearly one degree for the 63.6 meter sample, and 1.3 degrees for the 70.3 meter depth during the wet/dry cycles. An unexpected increase in strength was observed in the later stages of the 70.3 meter cycles. Similarities in the 63.6 and 70.3 meter residual friction angle plots are apparent for the first two cycles.

Residual friction angles for the wet/dry cycles decreased from 6.8° to 5.4°, 6.1° to 5.2°, and 6.7° to 5.4° for the depths tested. A degree drop in a material with an initial residual friction angle of 6.5° is significant. For a given normal stress and negligible residual cohesion, the factor of safety would be reduced by a ratio of 1.2. Exposure and removal of confining stresses during construction activities in Pierre Shale could cause wetting and drying to occur and lead to slope failures.

5 CONCLUSIONS

Minor mineralogical changes were observed in the wet/dry cycles similar to the weathering occurrences in Pierre Shale. Gypsum concentrations decreased initially in the wet/dry cycles. The low residual friction angles of 6.1° to 6.8° decreased an additional 0.8° to 1.4° during the wet/dry cycles. A significant fabric contrast was apparent after three cycles as the material's structure became more massive. The most noticeable difference in the cycles was the particle settlement rates. Excessive cycling caused particles to stay up in suspension for weeks to months longer than the un-cycled material. This observation indicates clay aggregates are becoming smaller and going towards their unit-cell size. The reduction in size increased the clay fraction, contributing to the residual strength decrease.

The mechanical behavior varied for the samples analyzed. The material with larger amounts of illite in the mixed-layer clay mineral showed a decrease in residual strength following Stark and Eid's (1994) curves. The material with higher amounts of montmorillonite in the mixed-layer clay mineral showed little change in the liquid limits, a contrast to the other sample. A decrease in residual strength was observed for the first two cycles but increased thereafter. The contrasting behavior shows the heterogeneity of the material and the difficulties in determining design parameters. The mineralogical changes and the disintegration of aggregates during wetting and drying are concluded to be more influential than physico-chemical effects.

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