Surface area and elemental composition of clay under elevated temperatures

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ABSTRACT: Micro-scale studies can provide useful insight to the macro-structural behaviour of unsaturated soils. Limited information exists about the effects of temperature, particularly extreme temperature levels, on micro-structural characteristics of unsaturated soils. In this study, various microscopic techniques were employed to investigate the micro-structural characteristics of a highly plastic clay, referred to as Buckshot clay, under elevated temperatures. Micro-structural characteristics of the tested clay first were examined using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectrometry (EDX) to characterize the minerology, micro-structure, and elemental composition, respectively. The soil was exposed to various temperatures ranging from the ambient room temperature up to 1000 ºC and were examined using Brunauer–Emmett–Teller (BET), and thermal gravimetric analysis (TGA) to determine the specific area and mass loss under elevated temperatures. Temperature-induced changes in the surface area can affect several properties of unsaturated clays such as the shrink-swell potential, the soil water retention curve, and mechanical-chemical coupling characteristics, among others. The SEM micrographs showed non-continuous flaky-like morphological properties of the clay. The results show that extremely high temperatures can have considerable impacts on micro-structural characteristics of the clay. The BET results indicated that the elevated temperatures have a notable negative impact on the soil surface area. Furthermore, the TGA data provides insight to the changes in the weight of the soil with varying temperature.

1 INTRODUCTION

The response of geomaterials to temperature changes has been extensively studied in geotechnical engineering (e.g., Demars and Charles 1982, Thomas and He 1997, Uchaipichat and Khalili 2009, Rajeev et al. 2012, Coccia and McCartney 2016, Ng et al. 2017). Several studies have been performed to examine the effects of temperature on soil's engineering properties such as strength, compressibility, compaction using numerical methods and prototype-scale laboratory tests (e.g., Cekerevac and Laloui 2004, Mon et al. 2014, Alsherif and McCartney 2015, Gao et al. 2015, Yavari et al. 2016, Zhou and Ng 2016).

Recently, studying the effects of temperature in the context of unsaturated soil mechanics has gained increased interest among researchers. This interest has primarily arisen in an attempt to deal with several emerging applications and issues, which involve non-isothermal conditions. Such non-isothermal problems include, but are not limited to, soil-atmospheric interaction under a changing climate, nuclear waste disposal, radioactive barriers, buried high voltage cables, ground-source heat pumps for geothermal heating/cooling systems, thermal energy storage systems, and thermally active earthen structures (e.g., Brandon et al. 1989, Laloui et al. 2006, Gens and Olivella 2001, McCartney et al. 2013, Vahedifard et al. 2015, Alsherif and McCartney 2015, Vahedifard et al. 2016, Robinson and Vahedifard 2016).

The majority of aforementioned applications generally do not expose soil to temperatures over 100 ºC. However, there can be certain applications that involve much higher temperatures. For example, NASA's Cassini spacecraft ended its 20-year voyage of exploring Saturn's moons and the atmosphere around it (Brown et al. 2017), which provided astrophysicists with a wealth of information about the cosmos. If a rover were going to land on a hot planet such as Venus, where the soil is similar chemically to that of volcanic rock found on Earth which consists largely of bedrock surfaces with a small amount of soil-like porous material (Basilevsky and Head 1988), high temperature ranges of up to 500 ºC may be applicable. In addition, smouldering remediation can apply heat ground surfaces to 500 – 1000 °C (Switzer et al. 2015), and ceramic firing applies temperatures upwards of 1400 °C to clays (Grim and Bradley 1940, Allegretta et al. 2017).

Most of the previous studies focus on the effects of temperature on the macro-scale behaviour of soils. Micro-scale studies can augment macro-scale tests by providing useful insight to the microstructural behaviour of unsaturated soils. Several attempts have been made to employ microscopic tests to explain macro-structural characteristics of unsaturated soils (e.g., Gen and Alonso 1992, Romero et al. 1999, Saba et al. 2014, Burton et al. 2015). Romero and Simms (2008) provide a wide-ranging review of the state of the art in this area. The results of a micro-structural or micro-mechanical study can be used for various applications inducing, 1) to improve constitutive models, 2.) to relate to macro-structural properties, 3) to aid in the interpretation of experiments where macro-structural measures cannot be observed, and 4) to provide insight to local hydro-chemo-mechanical phenomena in unsaturated soils (Walsh et al. 2007, Romero and Simms 2008).

There are limited studies in the literature regarding the effects of temperature on micro-structural characteristics of soil. The main objective of this research is to investigate the effects of elevated temperatures on micro-structural characteristics of a highly plastic clay, referred to as Buckshot clay. Micro-structural characteristics of the clay were examined using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectrometry (EDX) to understand the mineralogical, structural and elemental composition of the soil. The soil was exposed to various temperatures ranging from the ambient room temperature up to 1000 ºC and were examined using Brunauer–Emmett–Teller (BET) surface area analysis, and thermal gravimetric analysis (TGA) to quantitatively access the changes in the specific surface area and weight as a function of temperature, respectively.

2 MATERIALS AND METHODS

2.1 *Material*

The tested soil, Buckshot clay, is a high plastic clay (CH) collected from a construction site in Vicksburg, MS, USA. It is a Sharkey soil, which is the dominant soil type in the Mississippi Delta region, comprising of about one million acres. Their clayey, sticky, and plastic nature earned them the colloquialisms "gumbo" and "Buckshot" when referenced by locals. When these soils dry, small round aggregates form at the surface that look like shotgun Buckshot (hence the popular name for Delta clay soils, “Buckshot”) (Pettry and Switzer 1996).

Table 1 shows physicochemical properties of the Buckshot clay used in the tests. Prior to testing, the soil was dried and mechanically ground into a fine powder until it passed a 2.38 mm mesh for the information shown in Table 1, as well as for the XRD, SEM, and EDX analyses.

Table 1. Physicochemical properties of Buckshot clay.

Property	Measurement	Unit
BET Surface Area	66.620	m^2/g
Pore Volume	0.0178	cm^3/g
Pore Size	10.715	Å
pH	8.0	--
CEC	45.9	meq/100g
Organic Content	4	--
Classification	CH	--

2.2 *Methods*

To determine micro-structural properties of Buckshot clay, various microscopic techniques were carried out. The XRD analytical technique was used to obtain mineralogical properties of the tested soil. A Rigaku Ultima III X-ray Diffraction System was employed for the analysis of the samples using a scan angle (2θ) of 5° to 90° with step size of 1°/min. Finally, a comparison was made between the resultant patterns and the standard dataset of the Joint Committee for Powder Diffraction Standards (JCPDS 1995).

Scanning electron microscopy (SEM) is a common technique used to determine the micro-structural properties of soil fabric, providing information on the size, shape, state of orientation and aggregation of soil particles. The machine used for this study was a Zeiss SUPRA 40 field emission SEM (FESEM). Prior to testing, each sample was sputtered with gold-palladium for 120 s at 20 mA under vacuum suction. Also, the energy-dispersive X-ray spectrometry (EDX) attachment to the SEM was used to determine the elemental composition of the soil particles.

In this study, the BET surface area method was used to determine the changes that occur on the surface area and micro-pores of the samples due to the various temperature imposed. In this method, the surface area was derived through physical adsorption of nitrogen gas by means of a Micromeritics TriStar II PLUS surface area analyser. A small amount of cured sample was placed in the sample container. The outer area value was estimated adopting the single point BET technique.

Sample preparation for BET and TGA testing consisted of trimming thin slices of unsaturated Buckshot clay from excavated clods, air-drying the thin slices, and grinding them to pass the No. 8 sieve but remain on the No. 10 sieve. Approximately 1g of the air-dried and sieved soil was heated to the desired temperature using a ramp loading of 10°/min. Once the desired temperature was attained, the soil remained under constant temperature for 1 hour before removal from the oven. The sample was then placed in a moisture tight can and allowed to return to room temperature. Prior to BET testing, each sample was degassed for 1 hour at 180 °C under vacuum suction and the mass was recorded.

Organic matter can retard the proper action on a clay particle surface due to the organic particle’s greater affinity in adsorbing pore water ions and their own complex nature. The organic content of soil together with its thermal properties can be determined by monitoring the soil's weight loss in a controlled heating process under specified gas atmosphere. Some clayey minerals like montmorillonite and kaolinite can reduce 20% and 3%, respectively, in weight at low temperatures. However, the latter is triggered by dehydration and thus does not belong to the organic fraction. In this study, a TA Instrument TGAQ50 TGA device was used where a small quantity of the soil sample was inserted into an aluminium crucible in an N_2 gas atmosphere with a flow rate of 10 mL/min. The temperature was set to 1000 ºC at a rate of 10 ºC/min.

3 RESULTS AND DISCUSSION

3.1 *Micro-Structural properties of Buckshot clay*

The morphological features of Buckshot clay are shown in Figure 1. The SEM images show 5,000 and 10,000 times magnifications. The clay has a discrete surface made up of flaky clay particles with noticeable pore spaces. In addition, larger clay aggregates composed of the flakes are visible. The micrographs confirm that the Buckshot clay is a highly porous clay. The measured pore volume and pore size are reported in Table 1.

The elemental composition of the Buckshot clay as determined by EDX analysis is provided in Table 2. As shown in the table, the dominant elements in the Buckshot clay are iron, silica and alumina. Also, other elements like magnesium and potassium are available in the soil but in low percentages. Figure 2 depicts the EDX test graph for the Buckshot clay. The EDX graph confirms the presence of high amounts of silica with tetrahedral coordination and weak molecular bonding compared to the octahedral alumina. This could be a reason for the expansive behaviour of the Buckshot clay.

As a popular fundamental investigation technique, XRD is widely used to determine soil minerals. The results of the XRD test are provided in Figure 3. As can be seen, the analysis of the XRD data indicate that the dominant minerals in Buckshot clay were quartz and illite. The peaks corresponding to the quartz and illite minerals are marked accordingly. The illite presence in the XRD result correlates well with the EDX result. Illite is composed of sheets of silica weakly bonded to aluminium. It can be concluded that, the expansive behaviour of Buckshot clay could be due to the presence of illite mineral in this soil. The temperature effects on illite based on XRD analysis show that illite remains structurally intact up to around 800 ºC, then the structure is destroyed and the mineralogy gradually changes into what appears to be a spinel (Grim and Bradley 1940, Araújo et al. 2004).

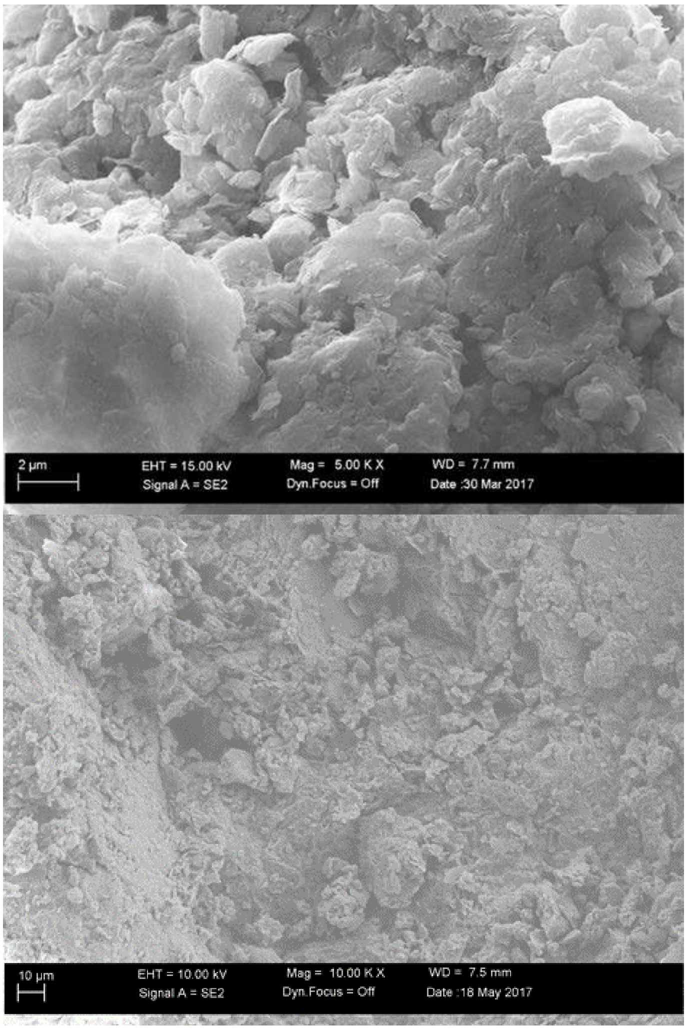

Figure 1. Micro-graphs of Buckshot clay at 5000x and 10000x magnifications.

Table 2. Elemental composition from EDX

Element	O	Mg	Al	Si	K	Ca	Fe
Percent Mass	35.11	1.25	9.99	36.21	2.53	1.35	13.57

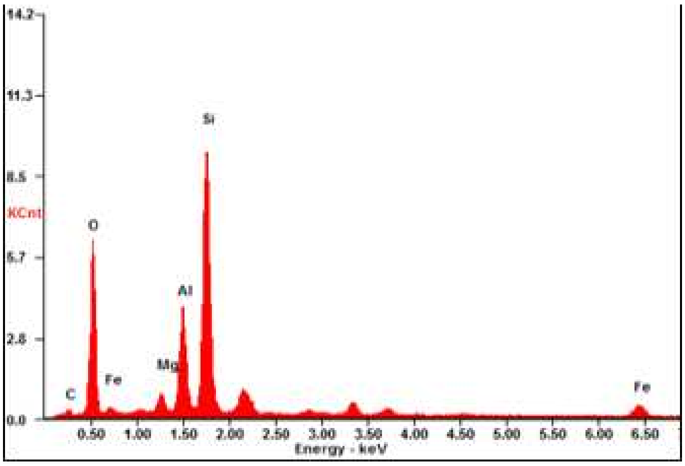

Figure 2. EDX results.

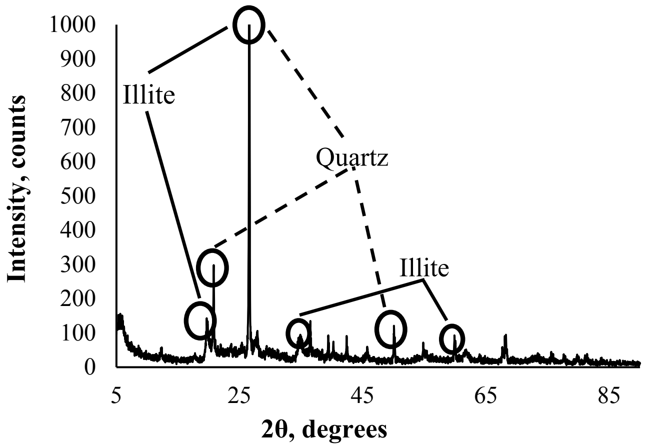

Figure 3. XRD results.

3.2 *Effect of temperature on Buckshot clay*

3.2.1 *Thermogravimetric Analysis*

The TGA analysis followed a ramp loading path of 10 ºC/min from room temperature to 1000 ºC. The results shown in Figure 4 show weight as a function of time throughout the temperature application. This test yields more information about the removal of moisture and organic materials of the Buckshot clay.

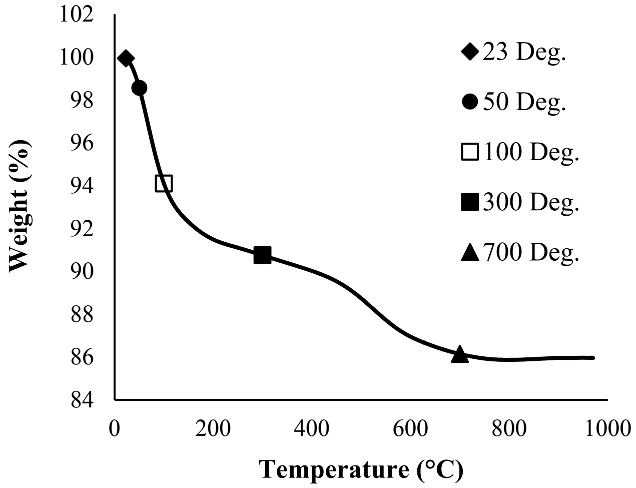

Figure 4. Weight versus temperature from TGA.

From Figure 4 the weight loss between 23 and 300 ºC is approximately 9.3%. This weight loss was due to the evaporation of the adsorbed water on the surface and inter-layer of the illite mineral (the dominant mineral in Buckshot clay as confirmed by XRD). Between 300 and 700 ºC, the weight lost was approximately 4.6%. This is attributed to removal of organics from the soil. From previous organic content tests of the Buckshot clay, it is known that 4.6% for the mass loss is nearly the same value provided from ASTM D2974 (Table 1). In addition, the loss at approximately 650 ºC – 700 ºC observed was due to the dehydroxylation of illite mineral. The total weight loss in the specimen over the entire range of temperature was approximately 14%.

Figure 5 shows the derivative of weight verses temperature. The peaks on Figure 5 show the temperatures at which weight begins to change. The first and tallest peak in Figure 5 shows the temperature at which the soil begins to dehydrate. The Buckshot clay, which was previously air-dried, starts to lose mass in the form of water at 66.7 ºC in this heating regime. The second major peak, which occurs at 525.8 ºC, is presumably the point at which the organic matter in the specimen begins to incinerate. In a typical organic matter content test, the soil is subjected to a constant temperature of 440 ºC for 24 h. However, since the application rate of temperature for this sample is much faster and the specimens are much smaller (the specimen size was 43 mg), then the temperature required to burn the organic matter in the soil is higher.

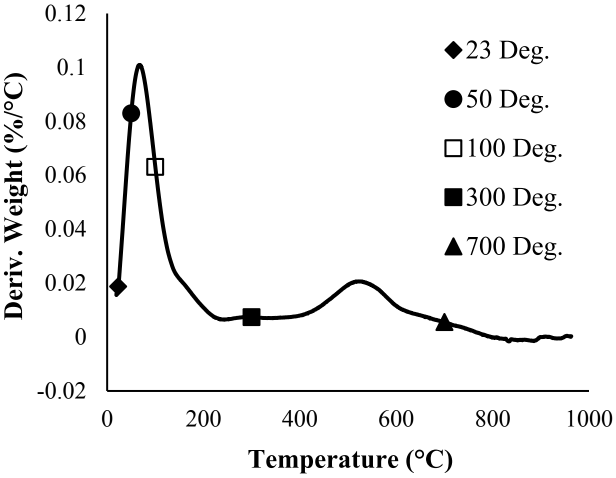

Figure 5. The derivative of weight verses temperature.

3.2.2 *BET*

For unsaturated clays, the surface area of the particles is a fundamental property that affects engineering behaviour. The shrink-swell potential (e.g., Ross 1978, Smith et al. 1985), Atterberg limits (e.g., Smith et al. 1985, Churchman and Burke 1991), the soil water retention curve (SWRC) (Khorshidi et al. 2016) and mechanical-chemical coupling characteristics (Santamarina et al. 2002) are all related to the specific surface area of the soil particles. Thus, understanding the effect of elevated temperatures on the specific surface area is important for a complete assessment of unsaturated soil behaviour.

Nitrogen-based BET adsorption analyses were carried out to find the effect of elevated temperatures on the surface area, pore volume, and pore size of the tested clay. The three points listed in the legend of Figures 4 and 5 are the points that were chosen for the BET analysis. Table 3 shows the results of the analysis for each temperature imposed to the soil. The results for the 1000 ºC data point were outside of the range of expected values required for initialization of the BET analysis, thus, only a pore size was attained from the analysis.

The trend in the results show a definite reduction in surface area, pore volume, and pore size of the soil. Figure 6 provides a plot of the surface area as a function of temperature showing a clear negative association between the two parameters. The form of Figure 6 also indicates the non-linear trend of data.

The differences in the values of Table 3 are thought to be more than that of the natural variance of the soil, however, more tests would need to be ran to verify. Some evidence to validate the previous comment for the BET surface area comes from Dogan et al. (2006) who performed BET characterization studies for *The Clay Minerals Society's* source clays. They show that the natural variation seen in the source clays range from approximately 0 - 6 m^2/g, and the variation seen in the special clay minerals (Dogan et al. 2007) is between approximately 0 – 4 m^2/g if the outliers are removed. These ranges are far less than those seen in Table 3, which implies that the temperature is the main cause of the reduction trends.

Table 3. BET results at various temperatures.

Temp. (ºC)	Surface Area (m^2/g)	Pore Volume (cm^3/g)	Pore Size (Å)
23	67.019	0.0178	11.415
50	67.202	0.0182	10.826
100	62.338	0.0166	10.666
300	61.856	0.0164	10.611
700	50.746	0.0126	9.893
1000	--	--	4.767

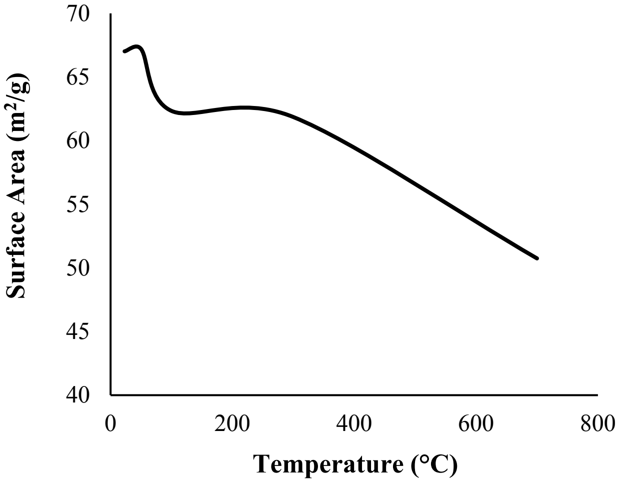

Figure 6. Surface area as a function of temperature.

Another point to consider is the effect of the organic matter within the soil that was removed at the higher temperatures but was not removed at the lower temperatures. The organic content of the Buckshot clay was measured to be approximately 4.6%. The BET surface area of organic matter is very small (approximately 1 m^2/g) in comparison to that of the clay particles (Chiou et al. 1990). Therefore, it is deduced that the removal of the small amount of organic content at the elevated temperatures for this soil doesn't affect the BET surface area calculations in this case. Therefore, it is thought that the downward trends in the BET data are caused primarily by the temperature variation in the soil.

It is noted that for the temperature imposed during degassing, it is assumed that only moisture was removed from the room temperature sample, and no organic matter or minerology was affected in the specimen. This is validated by the TGA results. However, degassing does impose an additional temperature cycle on the soil but is unavoidable in BET testing.

4 CONCLUSIONS

The objectives of this study were to study the micro-structural properties of Buckshot clay and evaluate the effect of elevated temperature on the micro-structural characteristics. A series of micro-structural tests were carried out. The result of SEM tests confirm that the tested clay has a porous structure EDX results indicated that the Si and Al are the dominant elements in the Buckshot clay and are the main reason for the expansive nature of the soil. The latter is also confirmed with the XRD test result, which indicated that illite is the main mineral in this soil. The BET test results on the clay samples under various temperature shows that by increasing the temperature, the surface area, pore volume, and particle size of the soil reduced significantly. In addition, the result of the TGA test confirms that by increasing the temperature, the soil thermal properties are changed.

Generally, the results indicate that for the given soil and testing conditions, there is a noticeable change in the micro-structure due to temperature variations. As demonstrated by previous studies, the surface area of the of the particles is a key property affecting unsaturated soil's engineering behaviour such as the shrink-swell potential, the soil water retention curve, and mechanical-chemical coupling characteristics, among others. Further tests are desired to quantify the effects of temperature-induced reductions in the surface area of the particles on the characteristics of unsaturated soils.

5 ACKNOWLEDGEMENTS

This material is based upon work supported in part by the National Science Foundation under Grant No. CMMI-1634748.

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