

# Physical and Mechanical Properties of Clay After Heating in Different Oxygen Levels and Cooling With Different Methods

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## Research Article

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# Abstract

Clay is a vital industrial raw material, and its physical and mechanical properties undergo significant changes when heated at high temperatures. A good understanding of those properties can provide details on the best use of clay, and may eventually facilitate new application areas. In this paper, the physical and mechanical properties of clay after exposure to high temperatures at two different oxygen levels and cooled in two different ways are studied. In the experiment, the clay samples are heated at high temperatures in anoxic conditions and air. The anoxic conditions are facilitated by a closed crucible. After the sample reaches the target temperature (400°C, 500°C, 600°C, 700°C, 800°C and 900°C), it is cooled to room temperature slowly in the furnace. The samples that are heated in air are either cooled in the furnace or quenched in water. Finally, the appearance of the clay samples in terms of their surface color and development of cracks, as well as their shrinkage, mass loss, hardness, strength and deformation, and the color of the core are examined and measured. The results show that the color of the surface of the clay samples heated in anoxic conditions and air significantly differs, and the color of the core is grayish black at temperatures that are below 800°C, which is possibly related to the oxidation and reduction of iron ions. Cracks in the clay samples partially offset shrinkage. The clay samples quenched in water show larger cracks and lower compressive strength. The mass of the samples does not change at temperatures that exceed 800°C. The hardness also does not change at temperatures that are higher than 700°C. In summary, temperatures between 700°C and 800°C have the most impact on clay because many of the physical properties (including their surface color and color of the core, shrinkage, mass, hardness, and strength) vary greatly at this temperature range.

## 1. Introduction

Clay is a vital industrial raw material and utilized in the ceramics, paper, paint and petroleum industries, as well as a catalyst, etc. It is generally believed that clay is a complex mixture of several different types of minerals (Geng and Sun, 2018). Clay may contain feldspar, quartz, mica, chlorite, smectite, etc., with exchangeable ions such as alkali metal ions (iron) and alkaline earth metal ions which are found in the internal clay structure or interlayers of clay minerals (Prudêncio et al., 2002; Escalera et al., 2014). The applications of clay very much depend on its structure, composition, and physical properties (Aramide, 2011). The physical properties of clay are highly temperature dependent (Sun et al., 2016b). High temperatures can impact the physical and mechanical properties of clay (for e.g., surface color, density, porosity, permeability, shear wave velocity, strength and compactness, and elastic modulus), thermal properties, and structure and appearance of clay (Milheiro et al., 2005; Sun et al., 2016a). A good understanding of these properties can provide details on the best use of clay, and may eventually facilitate new application areas (Aramide, 2011).

In different oxygen levels (anoxic conditions and in air), the surface color of clay bricks will differ when heated at high temperatures (Cultrone et al., 2004; Monteiro and Vieira, 2004; Aramide, 2011). Moreover, the center of a fired clay brick often contains a black or grey core (Gredmaier et al., 2011). due to carbonaceous matter which was added to the clay prior to the firing process. Small quantities are left

unburnt, thus promoting the formation of a black core. However, it is now known that the black core is not directly caused by unburnt carbon residue because it is still present even when clay with low amounts of carbon are fired. The black core is due to a reaction promoted by reduced iron species (Brosnan and Sanders, 2003; Sveda, 2006). Monteiro and Vieira (2004) found that there is an increase in linear shrinkage with increases in the firing temperatures of clay ceramics. Aramide (2011) found that the increase in the Brinell hardness of clay after subjected to a high temperature of 950°C, which might have been due to the presence of a glassy phase and mullite formation. Akinshipe and Kornelius (2017) found that clay heated in extremely high temperatures (> 1000°C) loses weight. The mass loss is due to the following reasons: 1) carbonaceous matter and combustible sulfur which are found in the clay are ignited and burned until eliminated; 2) carbonates which are in the clay break down and release carbon dioxide; and 3) water vapor is released from the clay. The mass loss of clay therefore indicates the amount of carbonaceous matter and carbonates that are found in the clay itself. For example, a low mass means that there is a small amount of carbonaceous matter and carbonates. Sun et al. (2016a) found that differences in the uniaxial compressive strength of clay are related to the transformation of minerals at high temperatures; for instance, dehydroxylated kaolinite is transformed into kaolinite at a temperature range of 400°C to 650°C. Kadir and Mohajerani (2015) found that while different rates of heating can impact the clay brick properties, the impacts are not substantial. However, exposure to higher temperatures has good environmental impacts, in terms of a shorter time of firing, more efficient use of energy, and less emitted gas.

Nevertheless, the properties of clay after exposure to high temperatures at different oxygen levels and then cooled in different ways are not well studied. Therefore, this study has conducted an experiment in which clay is heated in anoxic conditions and air, and then cooled in a furnace or quenched in water, to observe the changes in the surface color and color of the core, shrinkage, mass, hardness and strength after heating. Finally, thermogravimetric (TG) and derivative thermogravimetric (DTG) analyses are carried out and plotted, and the observations are discussed in order to understand the physical and chemical changes associated with heat.

## **2. Materials And Methods**

### **2.1 Clay and sample preparation**

The clay for testing was collected from Chengwu County in Heze City of Shandong Province in China. Locally, the clay is used to fire red bricks. The clay was then broken into small blocks of 1–3 cm in size and dried in a drying oven (BPH-120B, Shanghai Bluepard Instruments Co., Ltd.) at 70°C for 72 h. Then the clay blocks were grounded into a 200 mesh powder by using a comminutor. Finally, the clay powder was dried again in the drying oven at 70°C for 72 h to completely remove the free water.

The clay was first prepared with a moisture content of 15%, then placed into a mold to produce samples of  $\Phi 50 \times 100$  mm (diameter x height), and compacted by using a WES-D1000 electro-hydraulic servo universal testing machine (Shenzhen SUNS Technology Stock Co. Ltd.; also used for testing the uniaxial

compressive strength). During the compaction process, the load was increased at a rate of 0.5 kN/s until a load of 25 kN, after which, the load was maintained for 5 min to ensure that the sample was completely compacted. After compaction, the samples were removed from the mold and weighed, and their length was measured. Finally, the samples were tightly wrapped in plastic wrap and placed into a sealed bag to prevent moisture loss and kept at an ambient temperature of about 25°C. A total of 38 clay samples were prepared for the experiment.

## 2.2 Experimental procedure

In the experiment, the clay samples were heated at temperatures of 25°C, 400°C, 500°C, 600°C, 700°C, 800°C, and 900°C. The samples were first heated at different oxygen levels in a benchtop high temperature muffle furnace (KSL-1700X, Hefei Kejing Materials Technology Co., Ltd.), and then cooled in different ways. There were two oxygen levels during heating which were facilitated as follows: heating the sample in a closed crucible which did not allow the permeation of oxygen (therefore, an anoxic condition) and directly heating the sample in air. There were also two ways used to cool the sample: cooling in the furnace to room temperature (25°C) which allowed the sample to cool slowly, and quenching the sample in water to room temperature (25°C). Among the samples, those that were heated in the crucible were not quenched in water. Therefore, the samples in the experiment were divided into three groups: those that were heated in the crucible and cooled slowly in the furnace (labeled as NA); samples that were heated in air and cooled in the furnace (labeled as TA); and those that were heated in air and quenched in water for cooling (labeled as TS).

During the heating process, the rate of heating of the samples was 10°C/min. After the target temperature was reached, it was maintained for 1 h to ensure that the samples were fully heated. Next, the samples were dried in a drying oven at 70°C for 72 h to remove the free water. Then the samples were weighed again and their length was measured. The surface color and color of the core, hardness and uniaxial compressive strength of the samples were measured. Finally, TG and DTG analyses were carried out to measure the physical and chemical changes associated with heat.

## 2.3 Measuring color

The surface color and color of the core of the samples were determined by using a TES-135A color meter (TES Electrical Electronic Corp.). The color was assessed by using the CIE- $L^*a^*b^*$  system (Karaman et al., 2006b), in which color is quantified based on three chromatic coordinates: the  $L^*$  parameter represents lightness or luminosity ( $L^* = 0$  dark;  $L^* = 100$  white); the  $a^*$  parameter is the red–green axis ( $a^* > 0$  red;  $a^* < 0$  green); and the  $b^*$  parameter is the yellow–blue axis ( $b^* > 0$  yellow;  $b^* < 0$  blue).

## 2.4 Measuring hardness

The hardness of the samples was measured by using an SW-6230 hardness tester (Dong Guan Sndway Co., Ltd.) which measures the Leeb hardness value (HL). The hardness tester measures the hardness of a material as follows: by applying a force with the use of an elastic spring, a hammer with a specific weight is accelerated towards the surface and hits the sample at a certain speed. The hardness value is then

calculated the ratio of the rebound and impact velocities at a distance of 1 mm from the surface of the sample. The equation for calculating the hardness is as follows:

$$HL = 1000 \times \frac{V_B}{V_A}$$

where HL is the Leeb hardness value,  $V_B$  is the rebound velocity, and  $V_A$  is the impact velocity.

During the uniaxial compressive strength, a loading rate of 0.2 kN/s was used.

## 2.5 Measuring uniaxial compressive strength

A WES-D1000 electro-hydraulic servo universal testing machine (Shenzhen SUNS Technology Stock Co. Ltd.) was used to test the uniaxial compressive strength.

## 2.6 Measuring physical and chemical changes

TG and DTG analyses were carried out by using HCT-3 thermal analyzer (Beijing Henven Scientific Instrument Factory).

# 3. Results

## 3.1 Changes in sample appearance

The surface color of the clay samples changed significantly after exposure to high temperatures, and the colors differed based on the heating temperature (Fig. 1). It can be observed in Fig. 1 that when the samples are heated at temperatures higher than 500°C, the TA and TS samples are brick red in color, while the NA samples are bluish-gray. At a temperature of 500°C, the NA samples appear to be particularly dark in color because organic matter in the samples has carbonized to form carbon residue (Sun et al., 2016b).

Figure 2 plots the relationship between color and temperature. Compared with the  $L^*$  value of 34.7 of the untreated sample, the  $L^*$  value of the TA and TS samples increases with an increase in temperature, and the highest  $L^*$  value of the TA sample is 55.7 while that of the TS sample is 49.7 at 700°C; see Fig. 2(a). After that, the  $L^*$  value of the TA and TS samples remains almost constant but fluctuates at a value of about 52 and 46, respectively. However, the  $L^*$  value of the NA sample is lower than the untreated sample at temperatures less than 600°C. When the temperature exceeds 600°C the  $L^*$  value is greatly increased with the highest value of 53.9 at 900°C. In general, the whiteness of the TA and TS samples increases at higher temperatures, while that of the NA sample is first reduced, then greatly increased, and finally approximately equivalent to the  $L^*$  value of the TA sample.

Figure 2(b) shows the relationship between the  $a^*$  value and temperature, where the  $a^*$  value of the untreated sample is 9.2. At 400°C, the  $a^*$  value of TA and TS is 5.6 and 6.6, respectively, which is less

than 9.2. The  $a^*$  value then increases with higher temperatures, but increases more slowly between 500–700°C. The highest  $a^*$  value of the TA and TS samples is 20.1 and 22 at 800°C, respectively. However, the  $a^*$  value of the NA sample increases steadily from 3.1 at 400°C to 4.7 at 900°C. This indicates that exposure to a high temperature will initially reduce the redness of the NA sample. Then, both the TA and TS samples increase in redness at temperatures that exceed 500°C, but not the NA sample.

Figure 2(c) shows the relationship between the  $b^*$  value and temperature. At 400°C, the  $b^*$  value of the TA, TS and NA samples of 8.8, 11.4 and 3.7 is less than that of the untreated sample, which is 12.4. As the temperature is increased, the  $b^*$  value of the TA and TS samples greatly increases, and the highest  $b^*$  value of the TS sample is 26.8 at 700°C, while that of the TA sample is 27.5 at 800°C. However, there is a relatively smaller increase in the  $b^*$  value of the NA sample, and the highest  $b^*$  value of this sample is 9.4 at 900°C. It can be seen that exposure to a high temperature initially reduces the yellowness of the samples, but with increases in the temperature, the yellowness of the samples becomes higher. Compared to the TA and TS samples, the NA sample is less yellow in color.

In addition to the changes in the surface color of the samples, another area that is worth noting is the development of cracks on the surface of the samples at high temperatures. In Fig. 3, it can be observed that as the temperature increases, the surface cracks of the samples appear to grow in size. Among the samples, the NA samples appear to have relatively fewer and smaller cracks while the TS samples have more and larger cracks. This suggests that the clay samples have fewer cracks when heated in anoxic conditions.

## 3.2 Linear shrinkage

After heating at high temperatures, the clay samples have different lengths, and the decrease in the length is expressed by using linear shrinkage. Linear shrinkage is expressed as a percentage and calculated by using the following formula (Bories et al., 2014; Bodian et al., 2018):

$$\text{Linear shrinkage (\%)} = \frac{L_{\text{before heating}} - L_{\text{after heating}}}{L_{\text{before heating}}} \times 100$$

where  $L_{\text{before heating}}$  is the length of the sample before heating and  $L_{\text{after heating}}$  is the length of the sample after heating.

Figure 4 shows the changes in linear shrinkage with temperature. The linear shrinkage of the TA and TS samples is at least 1.9% and 1.3% at 600°C, respectively, and at most 2.9% and 2.4% at 800°C, respectively. The linear shrinkage of the NA sample is at least 1.6% at 400°C and at most 3.7% at 800°C. Overall, the linear shrinkage of the samples is slightly higher at higher temperatures, so the samples are not greatly affected by the shrinkage.

### 3.3 Mass loss

After exposure to high temperatures, the mass of the samples decreases, which is known as mass loss. In this study, the mass loss of the clay samples varies, and expressed as a percentage. Therefore, the mass loss is calculated by using (Bories et al., 2014; Bodian et al., 2018):

$$\text{Mass Loss (\%)} = \frac{W_{\text{before heating}} - W_{\text{after heating}}}{W_{\text{before heating}}} \times 100$$

where  $W_{\text{before heating}}$  is the mass of the sample before heating and  $W_{\text{after heating}}$  is the mass of the sample after heating.

Figure 5 shows that the mass loss of the clay samples gradually increases as the temperature increases. At a temperature range of 400–800°C, the mass loss of the samples rapidly increases, but at temperatures higher than 800°C, the increase in mass loss is almost linear. The highest mass loss of the TA, NA and TS samples is 27.4%, 27.9% and 27.3%, respectively. The mass loss is related to the loss of water, burning off of organic matter, and the decomposition of the minerals. Therefore, the composition of the clay samples can be considered to be basically unchanging at temperatures over 800°C.

Figure 5 Changes in mass loss with temperature

### 3.5 Hardness

The hardness of the clay samples also changed after exposure to high temperatures (Fig. 6). Their hardness increases with increases in temperature. At a temperature range of 400°C-700°C, the hardness of the samples increases rapidly, while at temperatures above 700°C, there is very little increase in hardness and there are basically no changes. The increase in the hardness of the clay samples after heating indicates the presence of a glassy phase and the formation of mullite (Aramide, 2011). Therefore, it can be inferred that the glassy phase of the clay samples in this experiment is initiated at temperatures over 700°C.

### 3.6 Uniaxial compressive strength and deformation

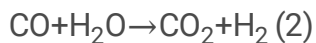
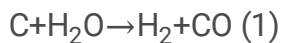
Figure 7 shows that the uniaxial compressive strength and deformation of the clay samples vary with temperature. At temperatures less than 700°C, the compressive strength of the samples increases with increases in temperature, and the highest compressive strengths of 15.5 MPa, 15.2 MPa, and 10 MPa for the TA, NA and TS samples are obtained, respectively. At temperatures higher than 700°C, the compressive strength of all of the samples is reduced. Besides, after exposure to high temperatures, the deformation of the clay samples is reduced. At temperatures over 400°C, the deformation of the samples increases slowly in general with increases in temperature, which means that the width of the cracks has increased. The generation of cracks reduces the strength of the clay samples. Moreover, the compressive strength of the TS sample is lower than that of the other two samples as a whole.

## 3.8 Color of the core

Figure 8 shows the color of the core of the clay sample after heated in high temperatures. At 400°C, there is a very thin grayish black layer on the clay surface, which is the carbon residue produced by the incomplete combustion of organic matter which covers the surface of the clay (Akinshipe and Kornelius, 2017). Moreover, at temperatures less than 700°C, the core is grayish black, but above 700°C, the core of the TA and TS samples become a light brick red color. The change in the color of the core is related to the environment in which the inside of the clay is formed at high temperatures.

## 4. Discussion

After exposure to high temperatures, clay undergoes a series of physical and chemical changes. The TA and TS samples which are heated in air become a brick red color at temperatures over 500°C because the iron-containing minerals in the clay are oxidized to  $\text{Fe}^{3+}$  (Murad and Wagner, 1998). At high temperatures, the  $\text{Fe}^{3+}$  cations can partially substitute for the  $\text{Al}^{3+}$  cations in the octahedral sites of the kaolinite structure (Manhães et al., 2002; Milheiro et al., 2005). A higher temperature means a more thorough reaction and a deeper red color. The NA sample which is heated in anoxic conditions is blue-gray in color because the organic matter in the clay is carbonized, and a series of chemical changes occur as a result, which is shown in Eqs. (1) and (2) (Toledo et al., 2004), thus resulting in a reducing environment in the crucible.

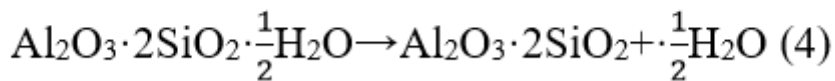
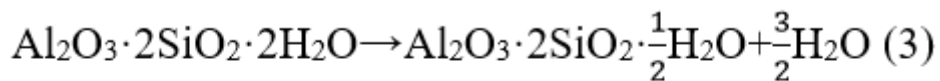


With a reduced firing temperature, the iron oxides are reduced to magnetite ( $\text{Fe}_3\text{O}_4$ ) and wustite ( $\text{FeO}$ ), both of which are dark in color (Houseman and Koenig, 1971). As the temperature rises, the blue-gray color begins to fade. This is because at a more rapid rate of heating, the  $\text{Fe}^{2+}$  cations react with a small amount of oxygen that has not yet been consumed, thus resulting in the formation of a small amount of  $\text{Fe}^{3+}$  cations which then partially substitute for the  $\text{Al}^{3+}$  cations in the octahedral sites of the kaolinite structure, so the  $\text{Fe}^{3+}$  ions are not reduced.

As for the core of the clay samples, the TA and TS samples which are heated in air at temperatures less than 800°C are grayish black. This is because the surface of the clay is first sintered, so that internally, it is a relatively closed anoxic system, and the water vapor inside cannot escape, thereby forming a reducing environment (Akinshipe and Kornelius, 2017), just like the NA sample. However, cracking penetrates the clay samples at temperatures that exceed 800°C, so that the inside of the clay samples is destroyed and exposed to the outside environment. Therefore, the core of the clay samples is no longer grayish black.



In order to understand the physical and chemical changes associated with heat, TG and DTG analyses were carried out by details of experiment here. The results are plotted in Fig. 9 which shows the TG curve of clay in a helium (He) atmosphere. At temperatures between 25-120°C, the TG curve slopes downwards rapidly which is mainly attributed to the rapid evaporation of the interparticle, adsorbed and interlayer water on the clay surface and interlayer space (Johari et al., 2010). At 300-600°C, the downwards trend of the TG curve is mainly due to the carbonization of the organic matter and the loss of the absorbed water in the clay samples. The absorbed water begins to evaporate when the temperature is above 300°C, and the organic matter begins to carbonize at temperatures between 400-500°C (Geng and Sun, 2018). At 600-730°C, an endothermic peak appears in the DSC curve, which is when the dehydration of the kaolinite occurs, as shown by Eqs. (3) and (4) (Sun et al., 2016b). At the same time, the melting of the flux components on the clay surface, such as potassium oxide (K<sub>2</sub>O), sodium oxide (Na<sub>2</sub>O) and calcium oxide (CaO), is initiated (Johari et al., 2010). Finally, at temperatures over 800°C, the clay mass does not change.



Temperature is a key factor that affects the shrinkage of the clay samples in this study. In general, increased temperatures result in an increase in shrinkage (Weng et al., 2003; Monteiro and Vieira, 2004). This is due to the loss of chemically and mechanically bound water (Karaman et al., 2006a). The capillary forces transport the liquid to the dried surface and shrinkage occurs as water is evaporated and the particles move closer together (Reed, 1995). At temperatures that exceed 700°C, the melting of the flux components such as K<sub>2</sub>O, Na<sub>2</sub>O, CaO on the surface of the clay is initiated; a liquid phase appears and the liquid fills the space between the solid particles (Johari et al., 2010). Due to the surface tension of the liquid phase, the unmelted particles are fused, which also causes volume shrinkage. There are two reasons for the formation of cracks. One is when the water evaporates, the rate of shrinkage on the sample surface is greater than the rate of internal shrinkage, which causes cracks in the sample. The second reason is that the thermal expansion of different minerals differs, so that thermal stress is produced between adjacent minerals, and deformation occurs between adjacent phases, which in turn evolves into cracks (Ahmad et al., 2008). Similarly, since the surface of the clay samples cools down at a faster rate and the inside of the clay samples cools down at a lower rate during the cooling process, the temperature differences between them produce thermal stress, which also promotes the formation of cracks. A higher thermal stress is produced when a sample is quenched in water, and therefore, the resulting cracks are much larger. In considering that some cracks will offset the shrinkage of soil samples, the rate of linear shrinkage is not entirely positively correlated with temperature in this study.

Shrinkage and sintering affect the hardness and compressive strength of clay (Manhães et al., 2002; Milheiro et al., 2005). The clay shrinks and the particles become more compact, so the hardness increases. At 700°C, the hardness of the samples increases due to melting and re-consolidation of the clay surface. As the temperature continues to rise, the clay surface is essentially unchanged, so the hardness does not change substantially. The shrinkage and sintering of the clay samples increase their compressive strength, while cracking reduces their compressive strength. At 700°C, cracking has not yet developed inside the clay samples, so the clay samples have the highest compressive strength when subjected to this temperature. With further increases in temperature, cracking develops inside the clay samples, and the compressive strength of the clay is reduced.

In addition, the temperature range of 700-800°C is an important range for clay because many of the physical properties (color, shrinkage, mass, hardness, and strength) greatly vary when the samples are exposed to this temperature range.

## 5. Conclusions

The properties of clay after exposure to high temperatures change at different oxygen levels and when subjected to different methods of cooling. Some of the key findings are summarized as follows.

(1) At temperatures higher than 500°C, the clay samples heated in air (TA and TS samples) are brick red in color, while the clay sample heated in anoxic conditions (NA sample) is blue-gray in color. At 800°C and higher, the clay samples heated in air no longer have a dark gray core. The changes in both the surface color and color of the core are related to the oxidation and reduction of iron ions. The oxidation of iron ions causes the brick red color while the reduction of iron ions causes the blue-gray color on the samples.

(2) In the process of evaporation, heating and cooling, the differences between the surface and internal properties of the clay samples contribute to cracking. Clay samples heated in anoxic conditions develop fewer cracks, while those quenched in water develop larger cracks. Water loss and sintering are the reasons for shrinkage. The cracks offset the shrinkage of the samples, so the rate of linear shrinkage is not entirely positively correlated with temperature.

(3) The mass loss is related to the loss of water, combustion and carbonization of organic matter, and the transformation of the mineral phases. The mass of the samples does not change at temperatures that exceed 800°C. Shrinkage and sintering increase the hardness and compressive strength of clay. The clay starts to sinter at 700°C, so the hardness does not substantially increase at temperatures over 700°C. Cracks reduce the compressive strength of the clay. The strength peaks at 700°C, after which the strength is reduced, and the strength of the clay sample quenched in water is overall the lowest.

## Declarations

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**Declarations of interest:** none.

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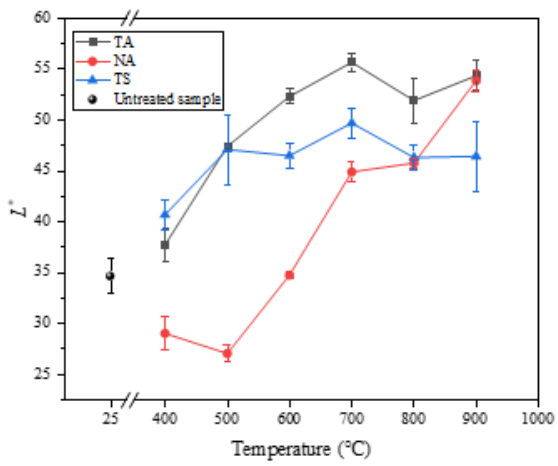
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## Figures

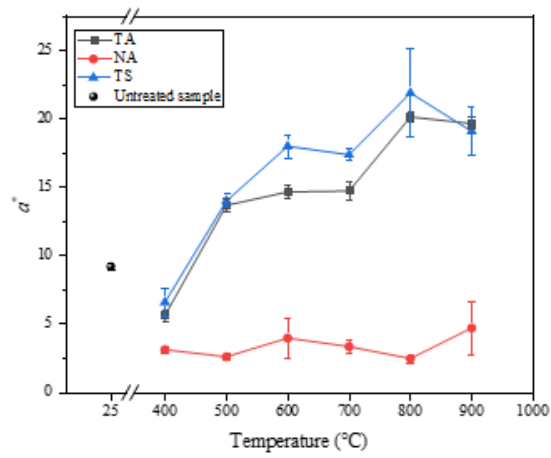


**Figure 1**

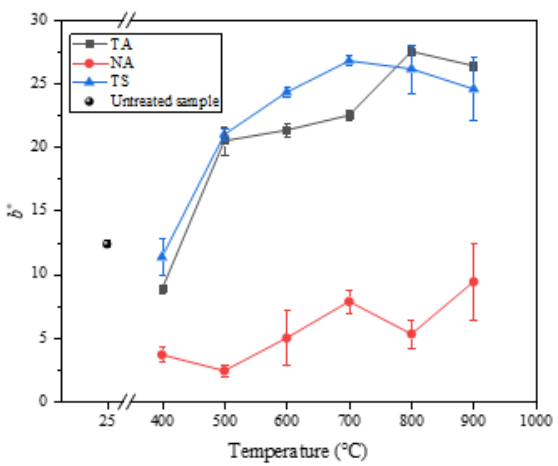
Changes in color of clay samples at different temperatures



(a)



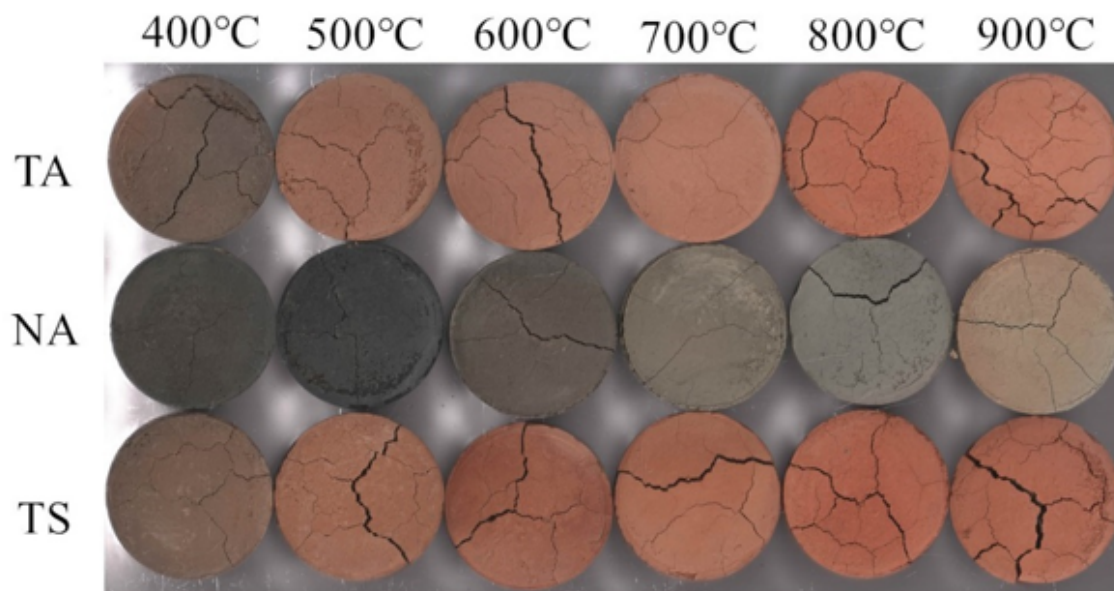
(b)



(c)

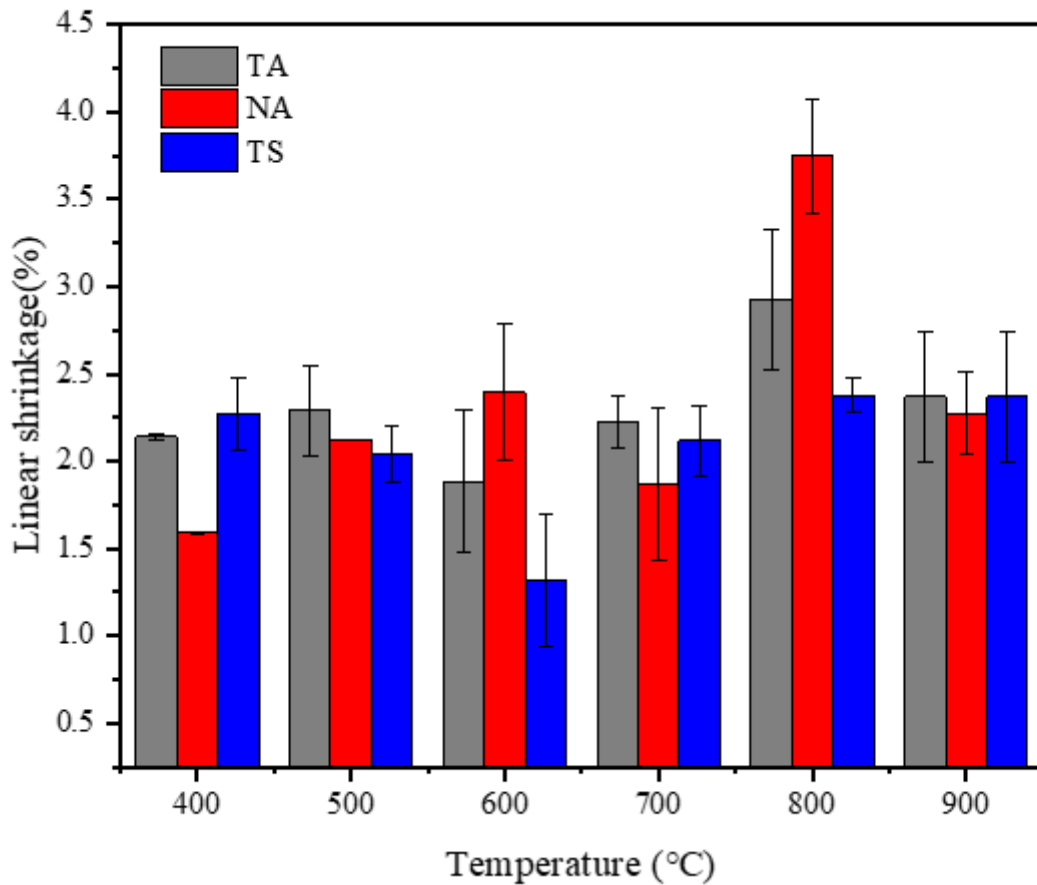
**Figure 2**

Surface color vs. temperature: (a)  $L^*$  vs. temperature, (b)  $a^*$  vs. temperature, and (c)  $b^*$  vs. temperature



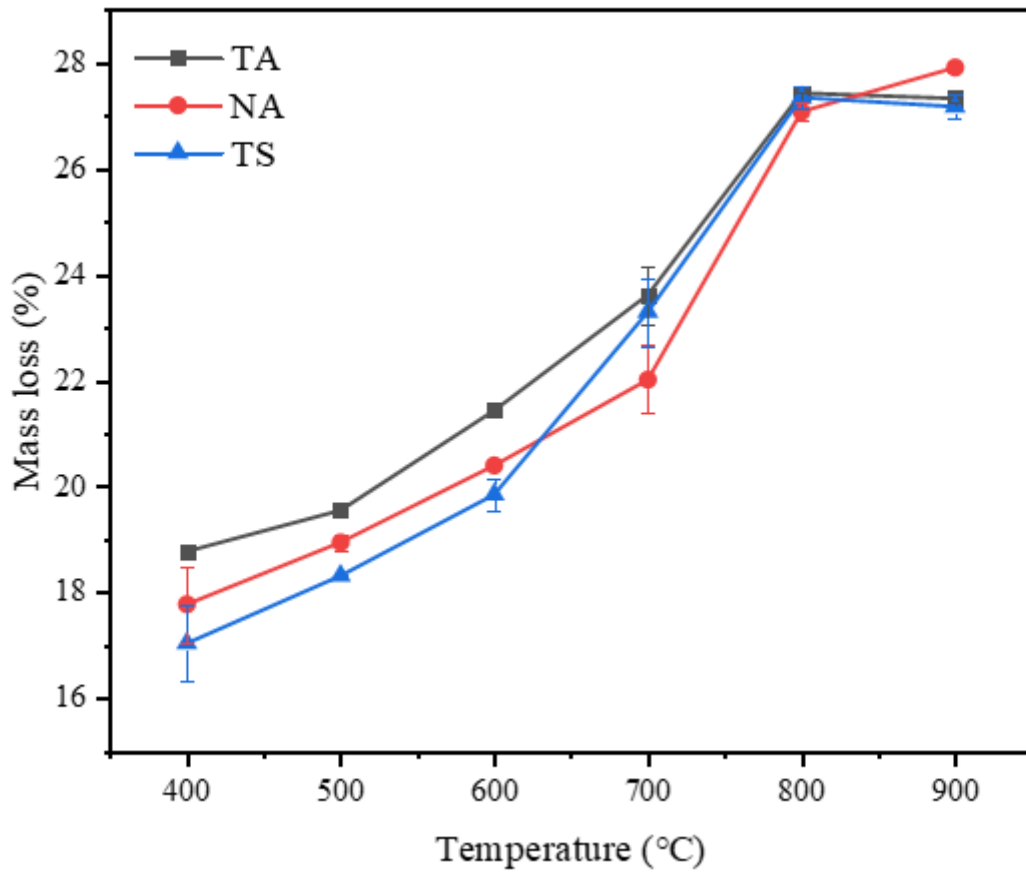
**Figure 3**

Evolution of cracking in clay samples at different temperatures and under different heat treatments



**Figure 4**

Changes in linear shrinkage with temperature



**Figure 5**

Changes in mass loss with temperature



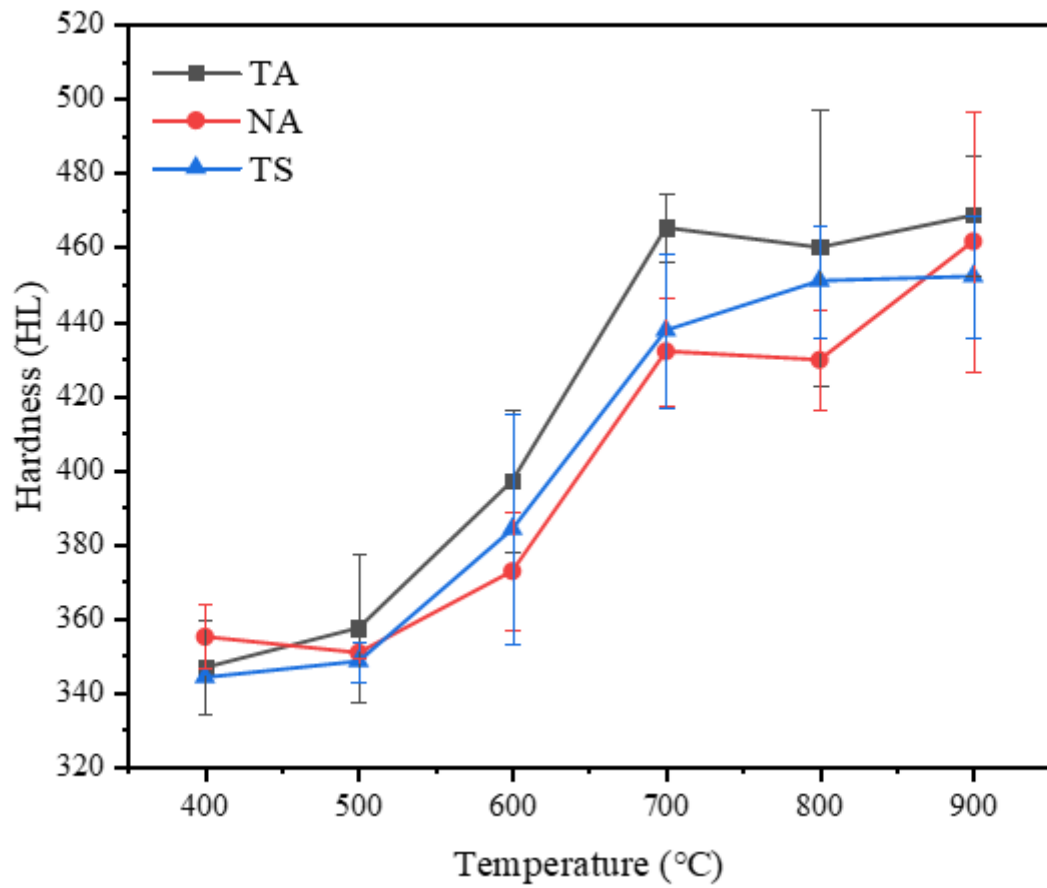


Figure 6

Variations in hardness of samples with temperature

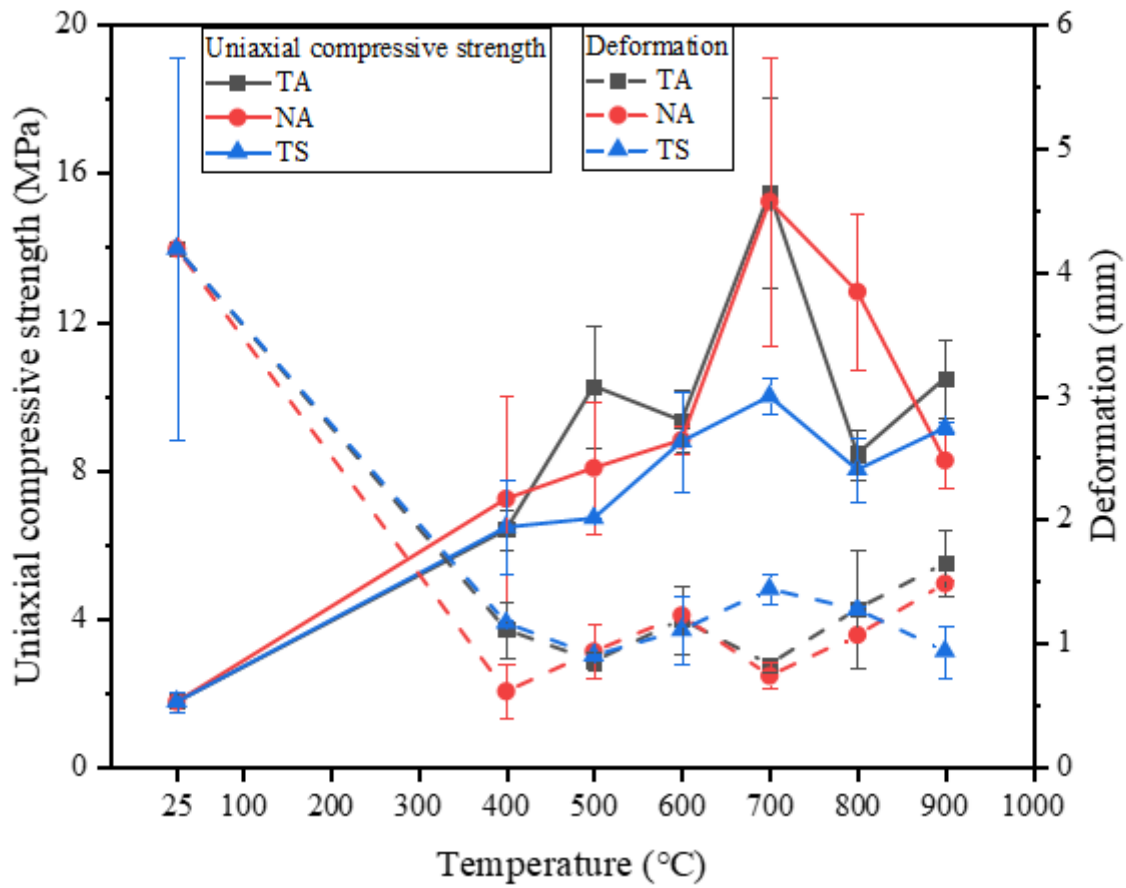
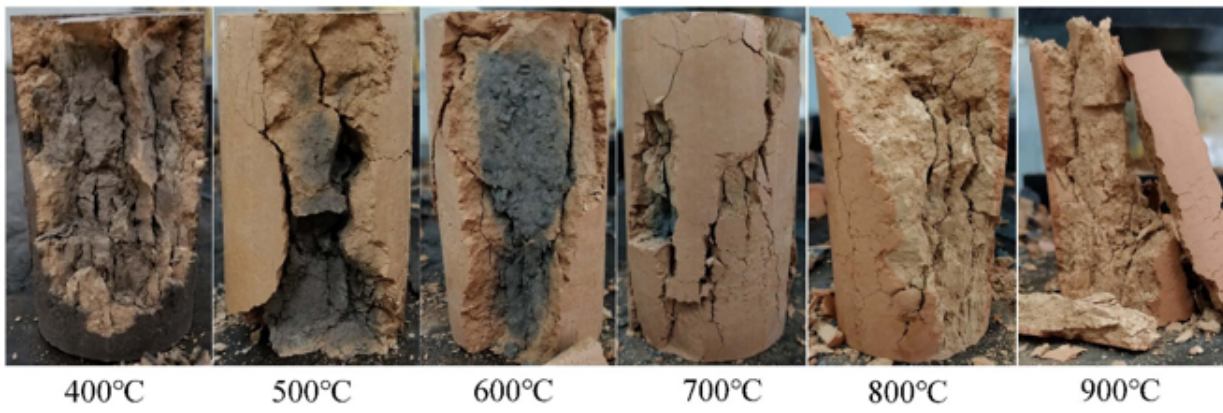
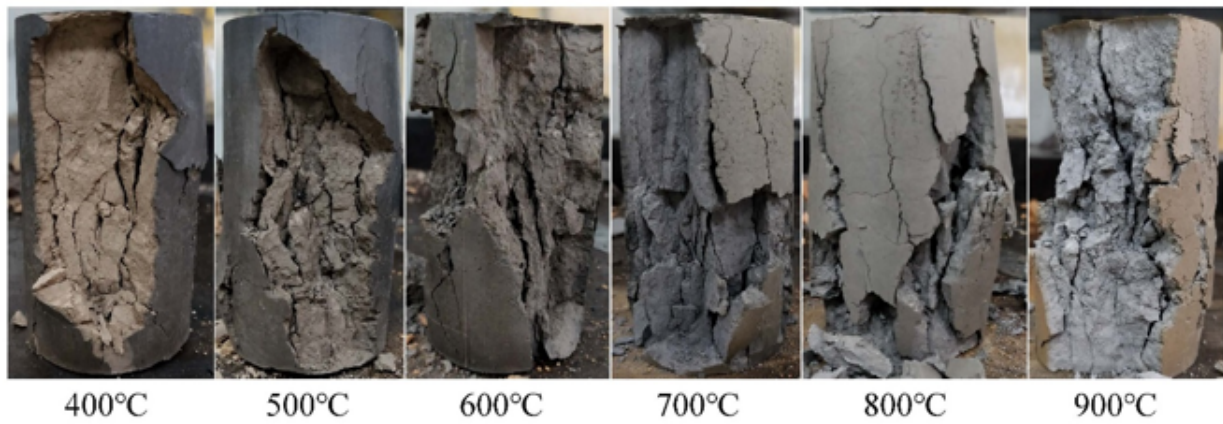


Figure 7

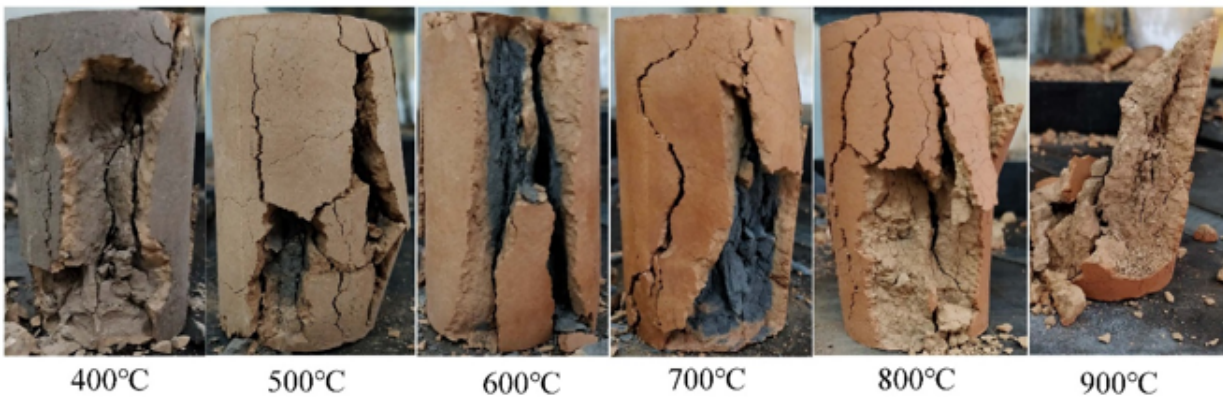
Variations in uniaxial compressive strength and deformation with temperature



(a)



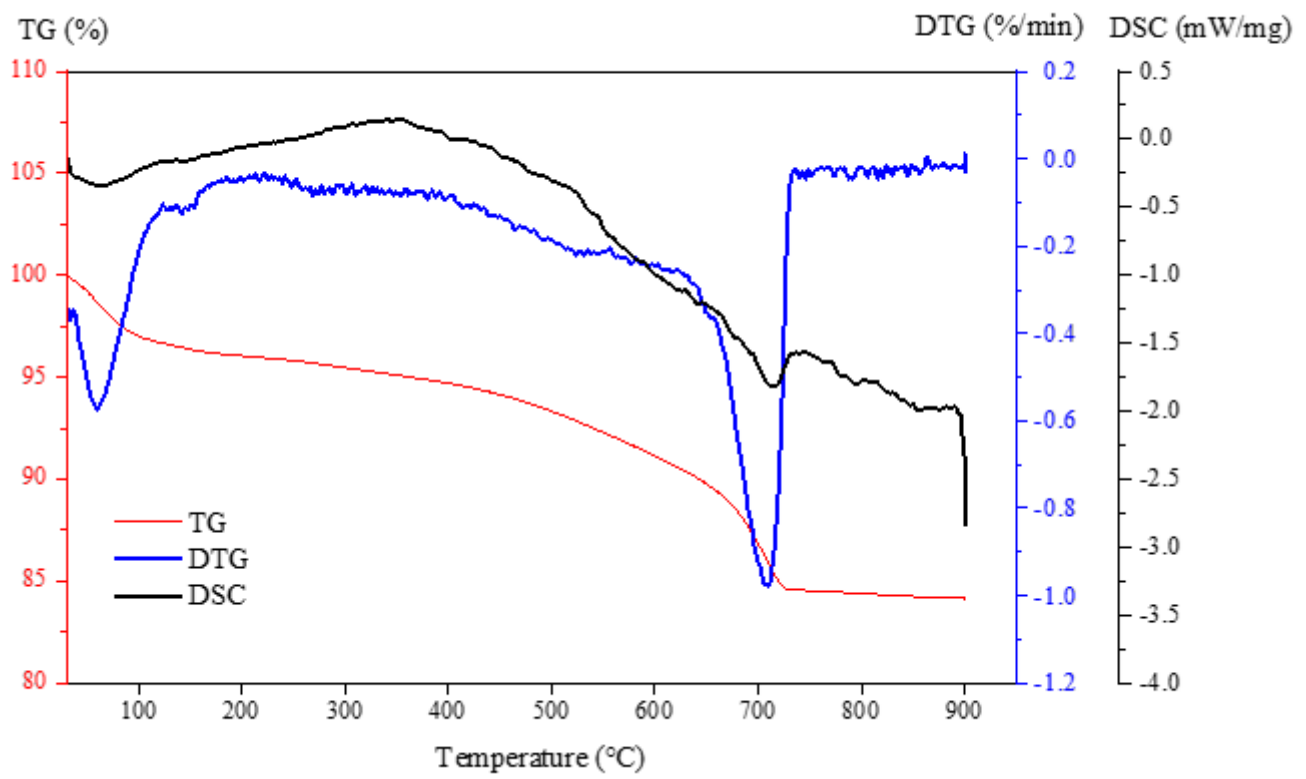
(b)



(c)

**Figure 8**

Color of core of samples: (a) TA, (b) NA, and (c) TS samples



**Figure 9**

Plotted TG, DTG and DSC values of clay in He atmosphere