

### To investigate Phase and microstructure of locally available bricks.

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#### Abstract

The aim of the current research was to study phase and microstructure of different bricks and clay used for bricks. Phase and microstructure of locally available bricks and the material used for manufacturing bricks have been investigated by using Energy dispersive spectroscopy (EDS) or X-ray electron spectroscopy (EDX) and Differential thermal analysis (DTA) techniques. EDS data for different bricks and clay used for bricks shows the presence of Al, Si, Fe, Mg, K, Ca and Na as major elements with little variations whereas clay contain some organic materials which upon firing emits moisture and carbon contents. Dehydration occurs before 200°C, removal of organic material before 550°C, dihydroxylation around 708°C till 900°C no exothermal have been observed to show crystallization of new phases.

**Keywords:** Bricks, Energy dispersive spectroscopy, X-ray electron spectroscopy, Differential thermal analysis.

#### I. INTRODUCTION

Clay brick is an old and the first material used and manufactured by human for construction, began its history in Egypt. Ancient Rome used for the construction of complex structures, like arches and vaults. Brick at that time was square and flat with name plinfa, which was translated as "a brick[1]. Bricks called burnt bricks were used in Byzantium (Istanbul today) for a long time as constructive

and decorative building material along with lime mixture or crushed brick crumb[2]. In Germany, brick is used as architecture of a specific style like brick Gothic till 12-16 centuries [3]. Human used this material in this technological era because of its structure, properties, availability, low cost and architectural reasons. Traditionally, clay bricks are strong and durable material in all weathers [4].

The common clay deposits are used for bricks are easily available and bricks can easily be manufactured locally. The colours and surface texture of clay bricks are different and attractive which makes the brick acceptable for all [5]. Clay bricks used for construction require, material properties, design, constructing technique, weather conditions and maintenance. The properties of bricks depend on its phase and microstructure, which in turn related depend on the chemistry of the material used for manufacturing of bricks and temperature for firing during the manufacturing process [6].

In the current study the phase and microstructure of locally available bricks and the material used for manufacturing bricks have been investigated by using Energy dispersive spectroscopy (EDS) or X-ray electron spectroscopy (EDX) and Differential thermal analysis (DTA) techniques. Some properties like chemical composition, dried and fired densities were also determined.

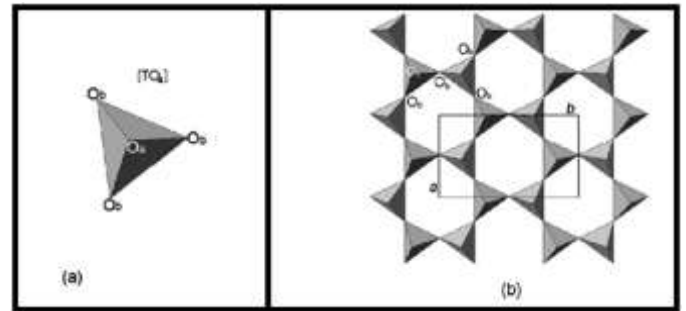
## II. LITERATURE REVIEW

Clay is defined as “a fine grained, natural, earthy and argillaceous material which includes shales and argillites of the geologist”. Chemically clay consists of alumina, silica and water, while some time with the variation of the amount of clay, it also consists of iron, alkalis and alkaline earths metals [7]. Clay societies defined clay as “A naturally occurring material composed primarily of fine-grained minerals, which is generally plastic at appropriate water contents and will harden with dried or fired”[8].

The clay bricks are manufactured using processes of soft mud, dry press and extruded [9]. Fly ash is also used for the manufacture of brick from coal power plants [10]. Before Roman time to first four centuries, clay has been used as a building material in different shapes and forms. Clay was used to form bricks. Clay is a material derived from weathered rock, contains Kaolin (hydro aluminium silicates) like smectite and illite [11].

Till the mid-19th century, brick made by hands, drying in the sun and fired in primitive furnace [12]. At the end of 19th century bricks were fired in annular kiln, belt press or machine pressed and the processing of clay was introduced, which leads to a technological manufacturing of bricks. Today bricks are stronger, resistant to water and frost, durable and meet modern standards [13].

Clay minerals contain a continuous tetrahedral sheet. Each tetrahedron consists of a cation (T) coordinated to four oxygen atoms and linked to adjacent tetrahedron by sharing three corners the basal oxygen atoms ( $O_b$ ) to form an infinite two-dimensional ‘hexagonal’ mesh pattern along the a, b crystallographic directions [14].



**Figure 1. (a) Tetrahedron [TO4] (b) tetrahedral sheet**

When clay heats it loses the inter particle water, constitutional water and some organic materials due to breaking of crystals [15]. The material becomes amorphous in XRD analysis, microscopic structure changes [16], some new phases appear like mullite and cristobalite and at very high temperature the clay melts and fuses at fusion temperature [17].

## III. EXPERIMENTAL SETUP

Bricks are one of the local products used for construction purpose. The provider processes the local available common clay through pressing and heating for bricks. Three qualities of bricks are locally available, namely, quality No-1, quality No-2 and Concker on basis colour and finishing. In the present study quality No.1 bricks are selected for analysis.



**Figure 2. Collected sample, fired brick, dried brick and mud and clay used for bricks.**

## SAMPLE PREPARATION

The powder sample were sieved through 270 US Mesh for particle  $\leq 53\mu\text{m}$ . After grinding and pestle the unprocessed samples are used for phase transformation temperature using

a “Thermo-Gravimetric and Differential Thermal Analyzer (TG/DTA), Diamond Series TG/DTA Perkin Elmer, USA, installed in Centralized Resource Laboratory (CRL) University of Peshawar, Khyber Pakhtunkhwa, Pakistan.

Energy dispersive spectroscopy (EDS) or X-ray electron spectroscopy (EDX).

X-ray fluorescence spectroscopy is one of the non-destructive analytical technique used for the chemical analysis of the solid samples. In this technique a beam is directed at the surface of the sample as a result, atoms are excited when de-excitation occurs radiations are emitted.

The characteristics of X-ray spectrum depends on the atomic number and the intensities of the incident beam which is used for the identification of the element present in the material sample [18]. The X-ray radiations are detected and analyzed due to which the atoms are identified. The intensity of the radiated beam is used for the quantification of the atoms of the sample. Energy Dispersive X-ray electron spectroscopy (EDS or EDX) is used for chemical analysis of grain or micro region in a sample. EDS detector installed is an accessory with JEOL JSM5910 SEM, operative at 30 KV, with resolution of 2.4 nm, in the CRL, University of Peshawar, Khyber Pakhtunkhwa, Pakistan.

#### DIFFERENTIAL THERMAL ANALYSIS (DTA)

Differential thermal analysis (DTA) is used for the determination of the dehydration, dihydroxylation and temperatures at which the significant phase transformation events occur. Thermo-Gravimetric and Differential Thermal Analyzer (TG/DTA) (Diamond Series TG/DTA Perkin Elmer, USA) Max temperature 1300°C. For this intention two small crucibles are taken which are usually formed of alumina/platinum used in DTA unit in which one of them is filled of suggested material (calcined alumina) and the other crucible is filled with a sample under test. Before taking the crucible inside the DTA chamber the clay sample were sieved to  $\leq 53 \mu\text{m}$ .

#### IV. RESULTS AND DISCUSSION

The clay samples used for bricks and bricks (both dried and fired) were collected from Ajab khan BahtiKhasht Bara Jalala, Mardan Pakistan. Water was mixed with the mixture of clay and sand and was put into the mould for giving proper shape. After proper shape the brick was dried in open air and sunlight. The colour of dried bricks was “Khaki” and changed to platinum (radish) when fired as shown in the Figure 3 and Figure 4. The colour of clay and dried bricks was nearly same and the small difference was due to the moisture contents.



**Figure 3. Sample of fired brick, dried brick and clay used in Bricks.**

The average size of dried bricks was  $221.78 \times 10774 \times 73.19 \text{ mm}^3$  and mass was 3030 gm. Similarly, of fired bricks was  $225.33 \times 109.76 \times 70.42 \text{ mm}^3$  and mass was 2475 gm.



**Figure 4. Clay used for bricks, dried bricks and fired bricks**

The overall energy dispersive spectroscopy (EDS) was used for chemical analysis of the clay and clay used for the bricks and fired bricks as in Figure 3 and 4. Both the EDS plots showed the presence of Al, Si, Fe, Mg, K, Ca and Na with a

little variation. In contrast the EDS of clay showed C while EDS of bricks showed no C, this is because of firing that cause the emission of moisture and carbon content.

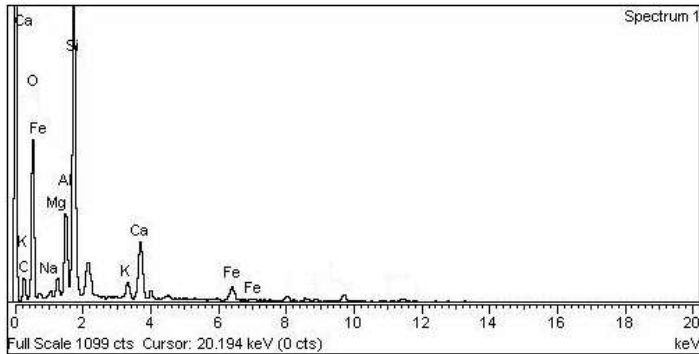


Figure 5. EDS of the clay used for bricks

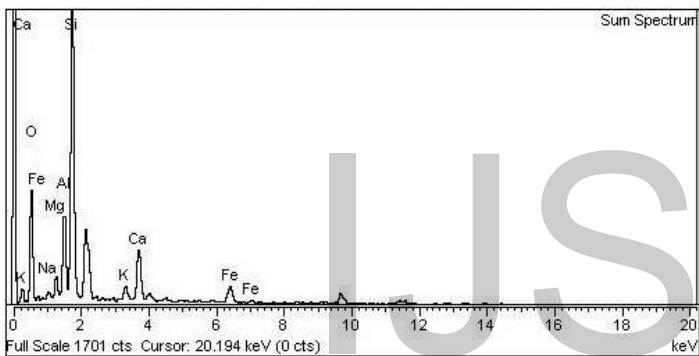


Figure 6. EDS of the bricks

Both the EDS plots showed an unmarked peak showing the coating of the sample with Au. The presence of Al, Si, Fe, Mg, K, Ca and Na shows the major oxides of clay, feldspar and silica. The highest peak of Si is because of the presence of quartz and sand.

#### DIFFERENTIAL ANALYSIS (DTA)

The differential thermal analysis plot showed the change in material with temperature. The endotherm showed the heat absorption while the exotherm showed heat emission during the thermal process. The DTA plot of the clay used for bricks is shown in Figure 7.

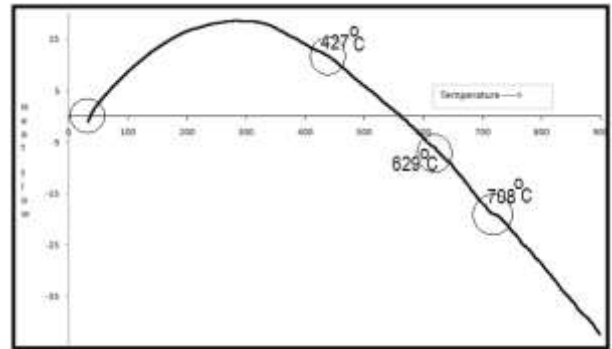


Figure 7. DTA of Clay used for bricks

The DTA curve showed the end of an endotherm before 30°C, indicating the removal of inter-particle water. Then the sample absorbs heat indicating by the positive slope of the curve. The plot comprises small exotherm at 427°C and endotherm at 629°C and 708°C. The exotherm is due to the change in phase of some organic material. The endotherm at 629°C and 708°C accompanied by the decrease in mass (by TGA analysis). This showed the dehydroxylation of some clay minerals.

#### CONFLICT OF INTEREST

The authors have no Conflict of interest.

#### CONCLUSION

Based on the above results the following conclusions are made.

- 1) Energy dispersive spectroscopy (EDS) analysis showed that clay and bricks contain Al, Si, Fe, Mg, K, Ca and Na with a little variation.
- 2) In contrast the EDS of clay showed C while EDS of bricks shows no C, this is because of firing that cause the emission of moisture and carbon content.
- 3) The endotherm at 629°C and 708°C accompanied by the decrease in mass (by TGA analysis) indicating the dehydroxylation of some clay minerals.

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