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To evaluate the composition and effect of temperature on mass decrease of clay using TGA and XRD techniques

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ABSTRACT

The aim of the current research was to study the mass decrease with temperature using Thermogravimetry (TGA) Technique and composition of the clay used in the manufacturing of bricks using X-ray Diffraction (XRD). Moreover, the bricks formed is further studied and analyzed using the above mention techniques. It is found that the strength of bricks depends on porosity and composition. Mass decrease and temperature distribution are irregular and from 629^{0}C to 708^{0}C a rapid decrease in mass occurred due to dehydroxylation.

Introduction

Brick is defined as a rectangular block shape of clay hardened by drying in natural air and sunshine or ignited in a kiln which is mainly used as a construction material. It is considered as one of the durable, cheap and easily available construction material used throughout the history dating back to 7000 BC [1] However, the analysis of the mineralogical and the thermochemical variations of clay throughout the firing treatment begun at the end of the 20th century[2]. Bricks are made from the mixture of clay minerals and non-clay minerals [3]. Clay minerals are chlorite, kaolinite, smectite, mixed- layer clays and illite [4] and some non-clay minerals are quartz, feldspar, calcite, dolomite, goethite hematite [5]. Common clays have different physical properties such as plasticity, dry strength, firing shrinkage, color and verification. These properties vary with the products depend on the ratio of mixing water and minerals [6].

The microstructure, phase, water absorption and porosity of bricks dependent upon the temperature. Heating destroys the crystal structure of clay, which results in pozzolanic amorphous substances like metakaolin when the heating temperature is roughly between 450 and 800 °C depending on the type of clay mineral [7]. When temperature changes up to 1250oC, the porosity of bricks decreases as a result strength increase [8]. Clay sciences have permitted researchers to study the consequence of clay materials and the heat behavior on the material nature so as to develop effective products with controlled properties [9]. It was found that the mechanical power and thermal conductivity intensely depend on the mineralogical composition of the raw materials used in the manufacturing of bricks [10]. Both industry and academia's people need optimum properties and used of a material[11].

In the current study the phase and microstructure of locally available bricks and the material used for manufacturing of bricks have been investigated by using Thermogravimetry (TGA) and X-ray Diffraction (XRD) techniques.

Key words

Bricks, Clay, TGA , XRD

METHODS

The phase and microstructure of the bricks and clay used for manufacturing of bricks have been investigated by using Thermogravimetry (TGA) and X-ray Diffraction (XRD) techniques

THERMOGRAVIMETRY (TGA)

Thermogravimetry (TGA) is a technique in which change in mass of the substance is measured as a function of temperature[12]. Thermogravimetric analysis (TGA) has emerged as a less expensive, easier, alternative and fast technique which measure the change in mass of a material with time at a given temperature, or over a temperature range using a predetermined heating rate[13]. TGA consist of a microbalance within a furnace interfaced with a computer and trace the change in mass (gain or loss) and at a constant heating rate, it plots the curve between weight and time or temperature which may single or multiple stages depends on the nature of the material. Reactions investigated by TGA depend upon the nature, mass, geometry in particle of the sample. DTA traces the useful information to differentiate the type of particular transformation.

X-RAY DIFFRACTION (XRD)

X-ray diffraction (XRD) analysis is used to measure the phase identification, quantitative analysis, residual strength, crystallinity and texture of the materials[14]. When X-rays are allowed to pass through a sample of a material then these X-rays are diffracted only from those planes for which the Braggs law satisfied and rise of a diffraction pattern of different intensities[15].

$$2\,d\,Sin\;\theta = n\;\lambda$$

Here "d" shows the inter planer spacing, "n" is the order of the diffraction pattern and λ is the wave length of the X-ray. The inter planer spacing & the corresponding intensities are compared with standard powdered diffraction data files such as JCPDS and ICDD cards. In the present study powder XRD was carried out using the press cavity mount technique. This powdered was placed in the Al cavity sample holder and pressed with a glass slide and then inserted into the X-ray diffractometer. All the samples were scanned from $2\theta^0 = 5^0$ to 60^0, using X-ray diffractometer with a Cu kα-radiations of $\lambda = 1.541838\,\overset{o}{A}$, operating at 40 KV & 30 mA with the scanning speed 1^o/min with a step angle of 0.02^o.

RESULT AND DISCUSSION

Raw brick materials as clay and processed bricks were collected from several places of Khyber Pakhtunkhwa, Pakistan. Finely divided brick samples were obtained by grinding them in a mortar and pestle system.

The TG analysis of the clay sample used in local bricks is shown in Figure 1 and the corresponding weight and Wt% loss is shown in Table 1.

S.no	ΔT (^{0}C)	Δm (mass)	%Δm
1	31-200	0.12	1.59
2	31-550	0.24	3.09
3	550-750	0.47	6.12
4	31-750	0.71	9.02
5	31-900	0.72	9.22

Table 1. Weight loss during TGA analysis

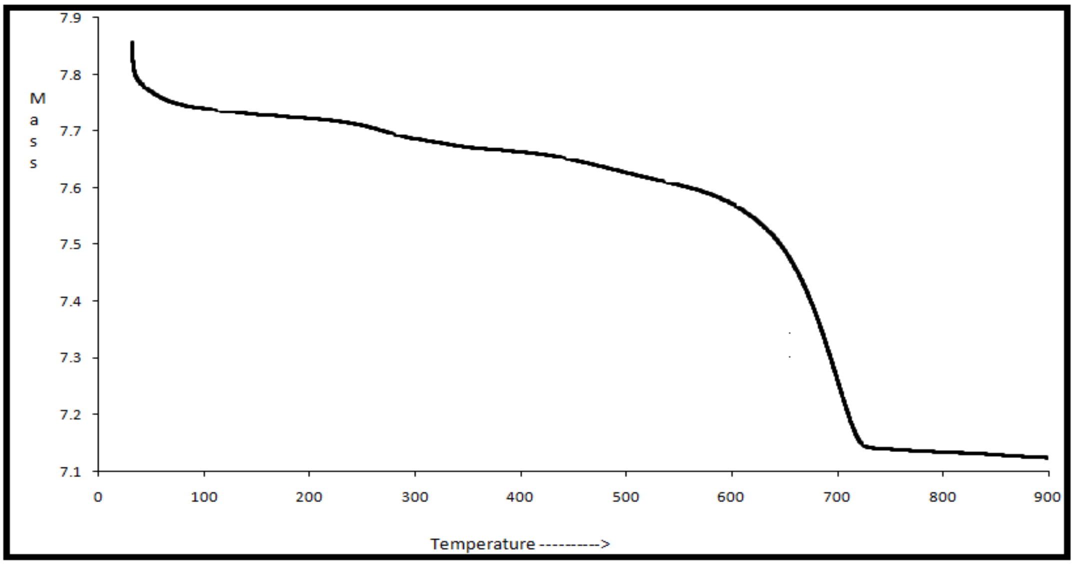

Figure 1. TGA of the clay used for bricks

Downward slope in Figure 1, before 200^{o}C shows the removal of surface water, which is 1.59 Wt%. The slope of the curve is nearly constant till 550^{o}C showing the uniform decrease in mass. This decrease in mass is because of dehydration and organic contents. The total mass decrease from 31 ^{o}C to 550^{o}C is 3.09 Wt%. The decrease in mass is higher between 550 ^{o}C to 750 ^{o}C which is 6.12 Wt% because of the removal of constituent water during the process of dehydroxylation. The total weight loss from 31 ^{o}C to 750 ^{o}C is 9.02 Wt%. The negative slope then nearly zero and the weight loss from 31 ^{o}C to 900^{o}C is 9.22 Wt%.

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X-RAY DIFFRACTION (XRD)

The X-ray analysis of the clay used for local bricks showed multi-phases clay having clay and non-clay minerals as in Figure 2. The major phases are anorthite ($CaAl_2Si_2O_8$), Calcite ($CaCO_3$), quartz (SiO_2), muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) and montmorillonite ($KAl_2(AlSi_3O_{10})(OH)_2$). The phase chemical formula and the ICDD number are shown in Table 2. The muscovite and Montmorillonite are clay minerals, Anorthite, Calcite and quartz are non-clay minerals.

S.no	Mineral Name	Chemical formula	ICDD #
1	Anorthite	$CaAl_2Si_2O_8$	100360
2	Calcite	$Ca\ CO_3$	30596
3	Quartz	$Si\ O_2$	30419
4	Muscovite-1M	$KAl_2(AlSi_3O_{10})(OH)_2$	70025
5	Montmorillonite	$KAl_2(AlSi_3O_{10})(OH)_2$	30009

Table 2. Phases its chemical formula and ICDD numbers used in clay for bricks

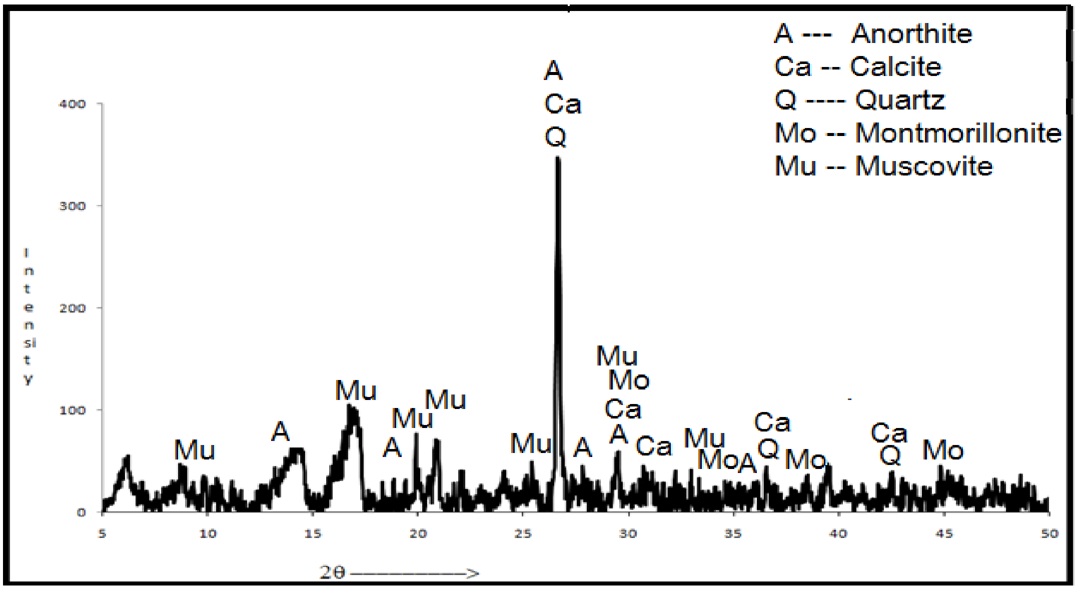

Figure 2. X-ray diffractogram of clay for bricks

The XRD analysis of the bricks also showed multi-phases having clay and non-clay minerals as in Figure 3. The major phases are anorthite with chemical formula $(Ca,Na)\ (Si,Al)_4O_8$, quartz with chemical formula SiO_2, muscovite with chemical formula $KAl_2Si_3AlO_{10}(OH)_2$, Albite with chemical formula $(Ca,Na)\ (Si,Al)_4O_8$ and essenite with the chemical formula $CaFeAlSiO_6$. The phases, chemical formula and the ICDD number is shown in Table 3. The muscovite is a clay mineral, Anorthite, albiteessenite and quartz are non-clay minerals. The removal of montmorillonite is due to firing, the crystal of which collapse before 800^{o}C. The presence of albite and essenite shows the selection of position clay selections which changes with the change of positions.

S.no	Mineral Name	Chemical formula	ICDD number
1	Anorthite, Sodian, O	$(Ca, Na)(Si, Al)_4 O_8$	90465
2	Albite, Calcian, dis	$(Na, Ca)(Si, Al)_4 O_8$	90456
3	Quartz	SiO_2	50490
4	Muscovite-1M	$K Al_2 Si_3 Al O_{10} (OH)_2$	70025
5	Esseneite	$Ca Fe^{+3} Al Si O_6$	400496

Table 3. The phases its chemical formula and ICDD numbers for bricks

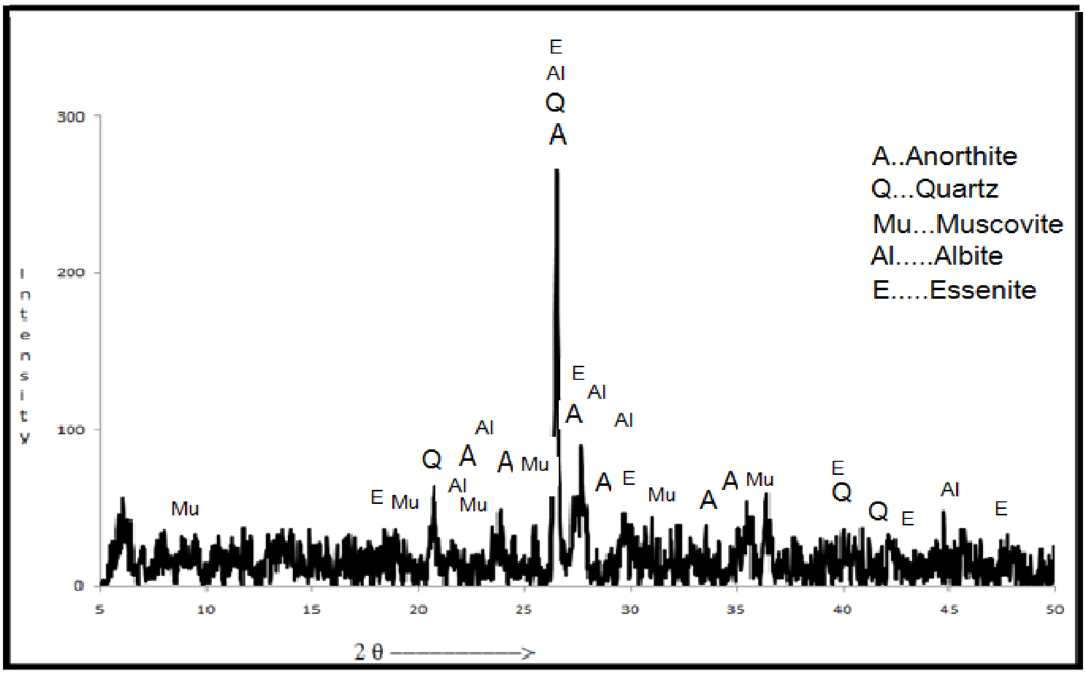

Figure 4. X-ray diffractogram of bricks

CONCLUSION

In the current work, a raw clay and its associated fired brick were studied. The results obtained from the test measurements showed that the industrial backing process changed the microstructure and the chemical properties of the bricks. Based on the obtained result the following points are concluded.

1. In TGA analysis, rapid decrease in mass occurred at 629^0C to 708^oC due to dihydroxylation of some clay minerals.
2. Porosity of the bricks decreased with increased in temperature as a result strength and durability of the bricks increases.
3. XRD analysis showed the presence of clay and non-clay minerals.
4. The removal of montmorillonite (phyllosilicate group of minerals) from clay is due to firing, the crystal of which collapse before 800^oC.

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