

Influence of Sintering Temperatures on Physico-Mechanical Properties and Microstructure of Refractory Fireclay Bricks

Job Ajala Amkpa^{#1}, Nur Azam Badarulzaman^{#2}, Abu Bakar Aramjat^{*3}

[#]Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn, Malaysia

¹jobitoh@yahoo.com

²azam@uthm.edu.my

^{*}Department of Ceramic Technology and Processing,

Kolej Kemahiran Tinggi MARA, 78300, Masjid Tanah, Melaka, Malaysia

³abubakararamjat@gmail.com

Abstract—This research emphasized on the influence of sintering temperatures on the microstructure and physico-mechanical properties of Kpata fireclay brick and Qua'an Panfireclay brick were assessed for suitability and application as refractory bricks. The clay samples were sintered at varied temperatures of 900 °C, 1000 °C, 1100 °C and 1200 °C. The clay samples were subjected to X-ray diffractometer to analyze the phase transformation after being sintered. The XRD results indicated the finger prints of the phase changes in the Kpata and Qua'an Pan fireclay bricks. At the best sintering temperature of 1200 °C, phase changes were quartz, mullite, rutile, corundum and cristobalite phases, while Qua'an Panfireclay brick had phase changes of quartz, cristobalite and mullite. SEM analysis was carried out to observe the surface morphology. The refractoriness of Kpata and Qua'an Pan bricks were 1621 °C and 1564 °C respectively. Their cold crushing strength and apparent porosity were investigated.

Keyword-Sintering Temperatures, Phase Transformation, Fireclay Brick, Refractoriness.

I. INTRODUCTION

The up surge application and request for refractory bricks had motivated this research to investigate the raw clay materials used for production of refractory bricks. The raw clay materials were subjected to standard refractory fireclay brick tests requirement through the tests procedures for chemical, thermal, physical and mechanical properties [1]. Clay as a raw material varied considerably in terms of workability, structure, particle-size distribution, plasticity and mineralogical composition. These differences paved way for clays being categorized or classified into plastic clays, flint clays, kaolin clays, fireclays and ball clays [2]. In the refractory industries, they are grouped and referred to as illite, Montmorillonite and kaolinite [3]. Refractories are clay materials that are generally capable of enduring very high temperatures without deformation, structural changes or softening [4]. The type of refractories is determined by the type of refractory requirement for a specific purpose. The types are classified as acid, basic and neutral refractories [5]. Traditionally these clay materials contain $Al_2O_3 \cdot SiO_2 \cdot H_2O$ to produce aluminosilicates refractory fireclay bricks. They are further classified by temperature. When the temperature is between 1500-1700 °C, it is branded as refractory e.g. fireclay. The temperature between 1700-2000 °C is recognized as high refractory e.g. chromite. When the temperature is > 2000 °C, it is referred to as super refractory e.g. zircon [5]. The applications of refractories are employed in the construction of furnace linings, ladles, reactors, ovens and kiln [6]. This disposition proved that the raw clay material is a fireclay and belongs to the aluminosilicate subgroup. The uses for fireclay refractory brick comprise of porous refractory insulation behind furnace linings, refractory fireclay bricks and ladles [7]. Refractories are frequently consumed basically in the iron and steel industries [8]. Contamination of refractory clays are usually with limited amount of impurity as contained in the oxides which are CaO, MgO, TiO_2 , Fe_2O_3 , Mn, SO_3 , ZnO, Cl, K_2O , P_2O_5 , Cr_2O_3 and alkali oxides that acts as fluxing mediator at very high temperature [9]. In the process of iron and steel production, the use of basic oxygen furnace (BOF) for the molten iron that comes from the blast furnace is decontaminated from the scums. Refractories belonging to silica-alumina have heterogeneous microstructure with bulky grain size and reasonable rate of porosity [10]. In Nigeria, previous investigation work has been done on the characterization of the native refractory raw clay materials for the making of quality refractory fireclay bricks for furnace lining, ladles, ovens and kilns [11]. In many cases, the outcome and results of such previous work were inconclusive and not comprehensive enough to be used or adopted for industrial uses. They were not subjected to standard test requirement for refractory bricks production. The researches for an alternative using the locally sourced raw material throw a challenge which is surmountable. This development will in no small way conserve foreign exchange for the country [12].

Deplorably, \$2.9 billion was being invested and depleted annually on the importation of these refractories [13]. It was on these premises that necessitated and informed the choice of investigation on these clays mentioned.

II. MATERIALS AND METHODS

A. Materials

The clay samples used in this research were collected from Kpata in Bassa local government area of Kogi state and Qua'an Pan in Qua'an Pan local government area of Plateau state, all in North central geo-political zone of Nigeria. The clay samples deposit sites were dogged 2 meters deep into the earth using an iron digger; 20 kg of the clay samples each were packed with shovel into a plastic bag. The clay samples lumps had an average size of 12-25 cm. The collection and transportation of all the clay samples were according to ASTM-D4220/D4220M-14 [14].

B. Methods

1) *Sample preparation*: Clay samples were ball milled to form fine powder particles. Each of the specimen powder was pressed into pallets using Caver hydraulic pressing machine. A force of 4 tonnes was applied with a holding time of 60 seconds. The samples were then transferred into furnace and subjected to thermal treatment by sintering process at varied sintering temperatures of 900°C, 1000 °C, 1100 °C and 1200 °C for 8 hours as soaking time with heating rate of 2.5 °C/min. The apparent porosity test was performed using the sintered test samples and the test was conducted according to ASTM C20-2000 [15]. The refractoriness test was performed according to ASTM C24-09 [16].

2) *Specimen characterization*: The X-ray diffraction (XRD) patterns were achieved using a BRUKER D8 ADVANCE machine. The pattern was scanned in series of angles diffraction from 10° to 80° (2θ) in steps of 0.034° designed for clay using copper (CuKα) with a wavelength of 1.5406 Å as X-ray source. At various angle, the intensity diffracted are measured and recorded instantly on a chart where the suited (θ) and (d) values were generated and received. The chemical composition was determined using the Oxford X-Supreme 8000 (XRF) technique. This analytical method is non-destructive, employed to identify the concentration of elements existing in the clay samples. The Scanning Electron Microscopy (SEM), JEOL JSM-6380LA and energy dispersive X-ray spectroscopy (EDX) were used to determine the elements dominate in the fireclay brick specimens and to observe the surface morphology of the Kpata and Qua'an Pan fireclay bricks.

III. RESULTS AND DISCUSSION

In the analyzed results of chemical composition of the two clay samples in Table 1, the dominating oxides for the two specimens were SiO₂ and Al₂O₃. Kpata fireclay brick had SiO₂ and Al₂O₃ with weight values of 74.8% and 22.599 % respectively, while Qua'an Pan fireclay brick had SiO₂ and Al₂O₃ with weight values of 63.437 % and 32.673 % respectively. The sintering of refractory bricks can impact strength to a large extent in the bricks products as they become stronger when the pores are closed due to rise or increase in sintering temperature [17]. The Kpata and Qua'an Pan fireclay bricks fall within the range of temperature 1500-1750°C for fireclays according to ASTM classification of refractories by temperature ranges [5]. The SEM micrographs were taken on X1500 magnification. Both clay samples showed a remarkable improvement on densification. As sintering temperatures increases, there was progressive improvement on the surface morphology of Kpata and Qua'an Pan fireclay bricks as presented in Fig. 1 and Fig. 2. Percentage porosity reduced as sintering temperatures were increased.

TABLE 1. Chemical composition (%)

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	P ₂ O ₅	Mn	SO ₃	Cr ₂ O ₃	Cl	ZnO
Kpata	74.876	22.599	0.609	0.907	0.091	0.217	0.156	0.222	0.029	0.296	0.296	0.176	-
Qua'an Pan	63.673	32.673	1.401	0.928	0.073	0.324	0.557	0.268	0.035	0.317	0.134	0.044	0.014

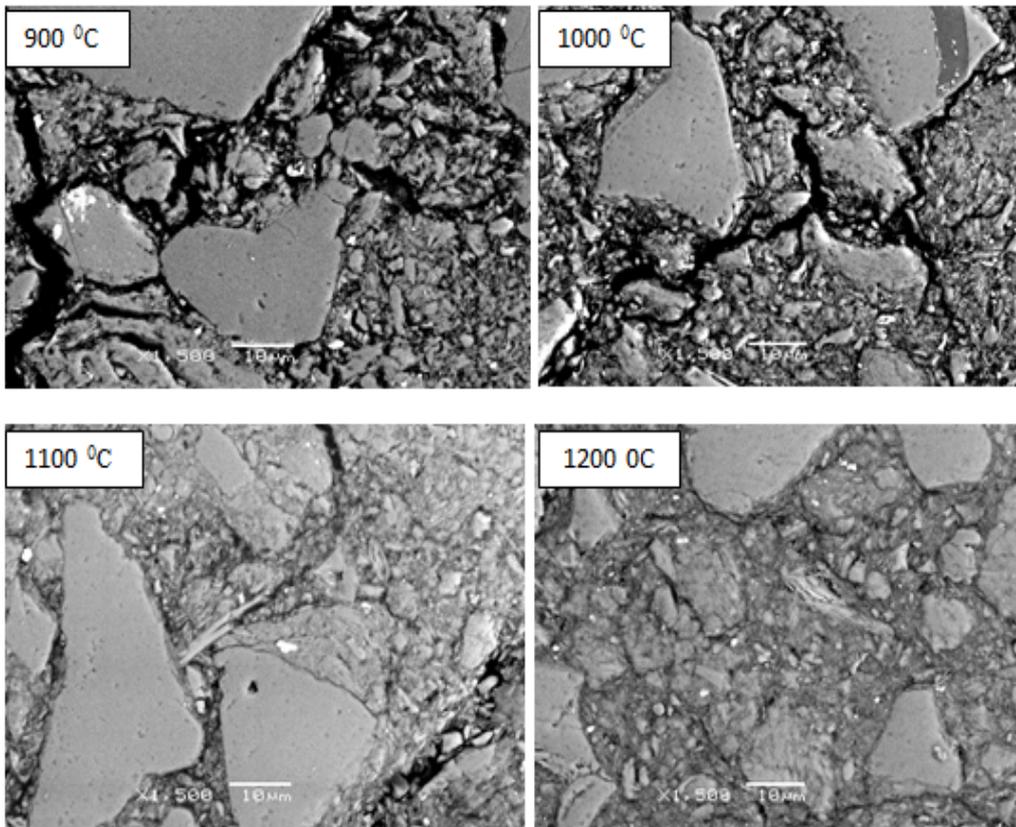


Fig.1: SEM micrographs (X1500) of Kpata fireclay brick and surface morphology at varied sintering temperatures.

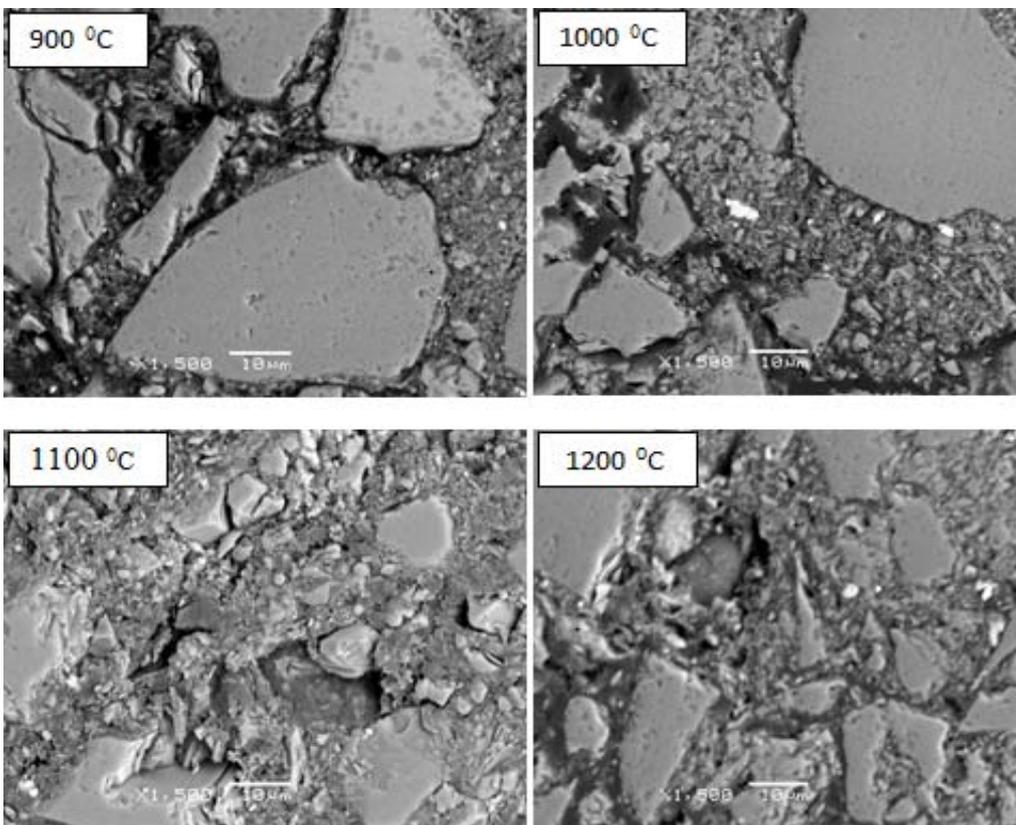


Fig.2: SEM micrographs (X1500) of Qua'an Panfireclay brick and surface morphology at varied sintering temperatures.

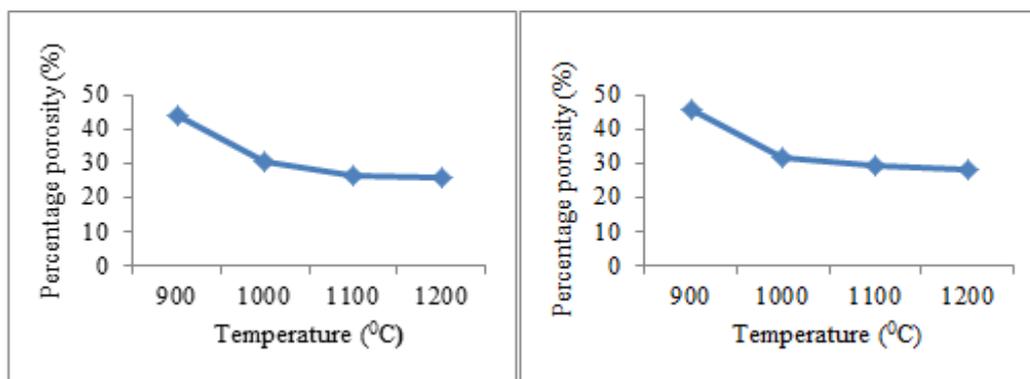


Fig. 3: Percentage porosity against varied sintering temperatures of Kpata and Qua'anPan fireclay bricks.

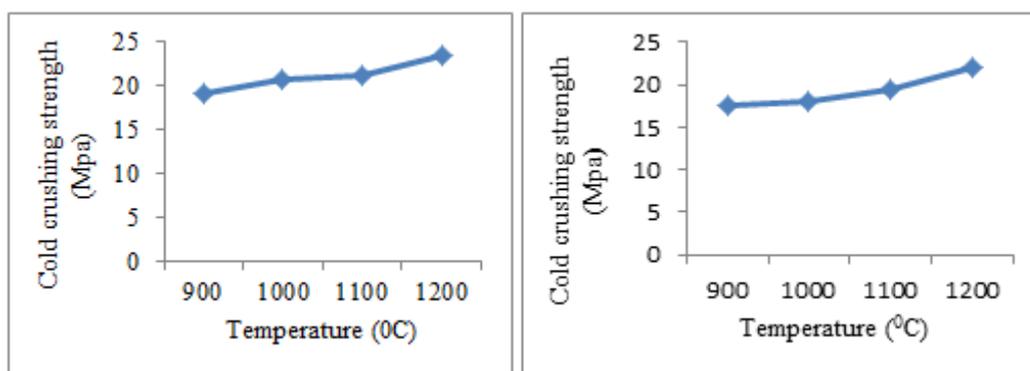


Fig. 4: Cold crushing strength against varied sintering temperatures of Kpata and Qua'an Pan fireclay bricks.

In the evaluation of physical and mechanical chattels, the evaluated results of percentage porosity and cold crushing strength in Fig. 3 and Fig. 4 of Kpata fireclay brick and of Qua'an Pan fireclay brick respectively, exhibited that Kpata fireclay brick had maximum porosity of 43.76 % at 900 °C sintering temperature, with the minimum porosity of 25.41 % attained at the best sintering temperature of 1200 °C. The Al₂O₃ was responsible for the strength in the clay material. The Qua'an Panfireclay brick had the highest porosity of 45.6 % at 900 °C sintering temperature and the lowest porosity of 27.73 % at the best sintering temperature of 1200 °C. The Qua'an Panfireclay brick had the highest Al₂O₃ content than Kpatafireclay brick as stated in Table 1. Al₂O₃ (Alumina) increases strength inthe clay material. The percentage porosity of the Kpata and Qua'an Pan fireclay bricks fell within the standard values of 20-30 % for fireclay refractory bricks [4] [13].

In terms of mechanical properties, the Kpata fireclay brick had the high cold crushing strength of 23.36 MPa at optimum sintering temperature of 1200 °C as against the lowest strength of 19.12 MPa at 900 °C sintering temperature. The Qua'an Pan fireclay brick had the highest cold crushing strength of 21.97 MPa at optimal sintered temperature of 1200 °C and the lowest strength of 17.64 MPa at 900 °C sintering temperature. The cold crushing strength also improved and increased as the sintering temperature was increased [17].The cold crushing strength (mechanical strength) of the Kpata and Qua'an Pan fireclay bricks fell within the standard values of 15-59 MPa for refractories [4].

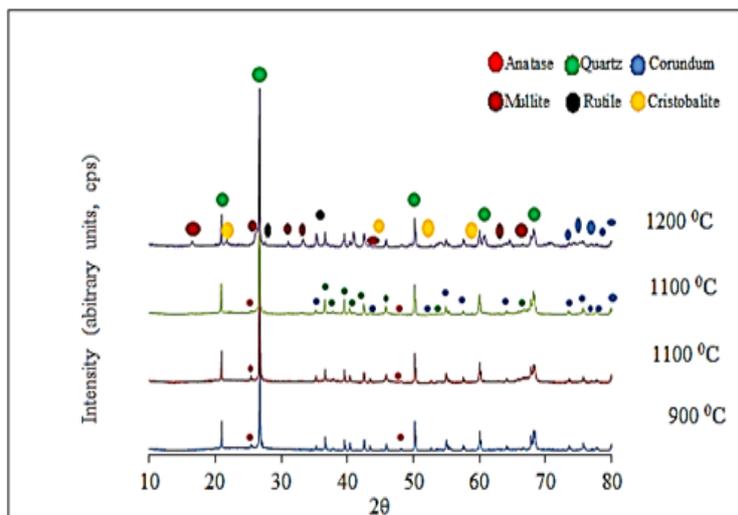


Fig 5: XRD pattern of Kpata fireclay brick.

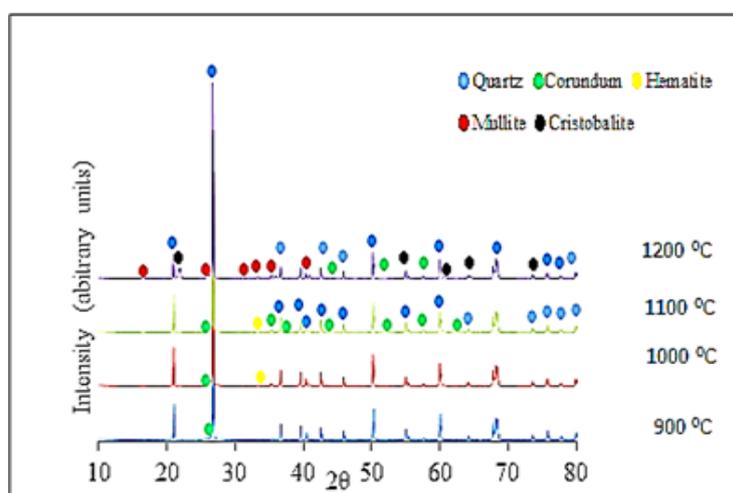


Fig 6: XRD pattern of Qua'an Pan fireclay brick.

The X-ray diffraction (XRD) results of the two clay deposits showed the crystallization and phase transformation at varied sintering temperatures. A very close observation on the Kpata clay deposit showed phase changes of quartz, corundum, anatase, rutile, mullite and cristobalite presented in Fig. 5, while Qua'an Panclay manifested phase transformation of quartz, corundum, hamatite, mullite and cristobalites shown in Fig. 6. The refractoriness of the clay samples were done one after the other by shaping the sample into a conical form. The standard pyrometric Segar cones of numbers 28, 29 and 30 of known softening temperatures were arranged on a standard circular shaped plaque with the aid of a binder. The cones at an angle of 82° to the horizontal were fixed on the plaque. It was then transferred to the refractoriness testing equipment and fired. The observation with the aid of a mirror attached to the refractoriness heating equipment, the PCE cone bent at firing temperature of 1621°C which corresponded to the equivalent of segar cone No: 29, hence, the refractoriness for Kpata fireclay bricks and this qualified the sample as high duty (Siliceous) brick. Similarly, the same PCE test was conducted for the Qua'an Pan fireclay brick using segar cones 19, 20 and 23. Its refractoriness was 1564°C which corresponded to segar cone No: 20 and qualified as low heat duty brick. Both refractory bricks qualified as fireclay bricks according to ASTM C27-98 [5]. The refractoriness of the two fireclay bricks fell within the standard refractoriness of $1500\text{-}1750^{\circ}\text{C}$ for fireclay refractories [13] [18].

IV. CONCLUSION

The stability and suitability of Kpata and the Qua'an Pan clays are based on their results of refractoriness, apparent porosity, cold crushing strength, SEM and XRD are adequate and appropriate to be used for production of refractory bricks.

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AUTHOR PROFILE

Job Ajala Amkpa PhD candidate Faculty of Mechanical and Manufacturing Engineering

Nur Azam Badarulzaman Senior Lecturer at the Department of Materials and Design Engineering

Abu Bakar Aramjat Lecturer in Ceramic Processing Technology Center, Kolej Kemahiran Tinggi MARA