

BIBECHANA

A Multidisciplinary Journal of Science, Technology and Mathematics

ISSN 2091-0762 (Print), 2382-5340 (Online)

Journal homepage: <http://nepjol.info/index.php/BIBECHANA>

Publisher: Research Council of Science and Technology, Biratnagar, Nepal

Investigation of the firing temperature effects on clay bricks of Kathmandu valley (Nepal); Part-I: Mineralogical phase characterization

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Article history: Received 25 August, 2018; Accepted 9 October, 2018

DOI: <http://dx.doi.org/10.3126/bibechana.v16i0.21319>

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Abstract

Present research work was focused to investigate the firing temperature effects on mineralogical phase composition of nine clay bricks collected from the brick factory of Kathmandu valley using X-ray diffraction (XRD) patterns and Fourier transform infrared (FTIR) spectra analyses. Main mineralogical phases of quartz, feldspars, spinel, mullite and hematite in the brick specimens fired at different firing temperatures including muscovite type of mica mineral in the sun-dried brick specimen are identified from XRD and FTIR analyses. Disappearance of the muscovite type of mica clay mineral with feldspars enhanced to form alumina rich spinel phase at firing 900° to 1000° C, and finally the primary mullite phase in the fired clay brick samples is clearly observed at 1100° C firing temperature.

Keywords: Brick; Firing temperature; Phase composition; Clay minerals; XRD; FTIR.

1. Introduction

The clay brick is one of oldest building materials yet known and its widespread use is mainly due to the availability of clay minerals used for the production and durability in both load bearing and non-load bearing structures. It was a fundamental building material in the Mesopotamian, Egyptian and Roman periods and the use of clay brick was increased and became specialized in order to maximize its benefits during the Roman period [1]. The durability of the bricks is its ability to withstand an applied load without failures [2]. The clay brick is used extensively in the construction industry all over the world. The global brick production is estimated about 1.5 trillion annually [3] and Asian countries only accounts about 89-90 % of the global production, i. e., about 1.35 trillion bricks [4]. The clay brick production in Kathmandu valley is estimated more than 3.3 billion units and with increasing demands of the clay bricks for construction industry, bricks quality and cost become more important nowadays [5].

There are different types of clay bricks and are divided into various groups based on their mineralogical compositions, uses [6] and quality and appearance [7]. The main factor to manufacture these bricks is the clay raw material types used and the firing temperature during their production, because both of these affect the quality and durability of bricks [8-11]. The firing of clay bricks at high temperature is generally practiced to obtain coherent clay bricks with controlled microstructures, because the presence of pore in the fired bricks at low firing temperature is generally one of the causes for showing their low mechanical strength [12]. Depending on the degree of firing, the porosity and water absorption capacity of the clay brick samples may vary. The fired clay brick is extremely durable and is perhaps one of the man-made building materials so far. It is said that the durability (i. e., mechanical) properties of the clay bricks depend mainly on their mineralogical and physico-sintering properties. Furthermore, the physico-sintering properties of the clay brick depend on the mineralogical composition of the clay minerals used to manufacture it, the manufacturing process and firing temperature [13].

The mineralogical, physico-sintering and mechanical properties of clay bricks are varied depending on the degree of firing at high temperature and these properties are interrelated to each other. The physico-sintering and mechanical properties of different clay bricks were investigated at different firing temperatures and the brick sample fired at 1100 °C showed the best mechanical properties [12, 14-17]. It was reported that the firing temperature was the key factor to modulate the physico-sintering and mechanical properties of clay bricks, not so much affected by firing time [18]. Similarly, it was reported that the apparent porosity and water absorption capacity of the fired clay bodies were decreased with increasing their firing temperature, while the bulk density and Young's modulus of elasticity of the bodies were increased with increasing the temperature [14, 17].

The quality assessment of different types of clay bricks is based on their mineralogical, physico-sintering and mechanical properties. However, such technical properties of the clay bricks [19, 20] and ceramic tiles [21, 22] of Nepal were studied by very few research groups in Nepal. Considering these facts, the present research work is focused to carry out an investigation of the firing temperature effect on the mineralogical phases of the fired clay brick samples by XRD and FTIR analysis.

2. Materials and Methods

Nine pieces of sun-dried clay brick sample specimens and one piece of fired clay brick sample from factory of Shiddi Vinayak Chimney Vatta Udhyog of Lalitpur (Nepal) were collected in this study. Among these nine pieces of the sun-dried clay brick samples, eight pieces were fired at 700°, 750°, 800°, 850°, 900°, 950°, 1000° and 1100° C using muffle furnace maintaining two hours as a soaking period and remaining one piece of the sun-dried clay brick sample was used to identify their mineralogical compositions using XRD and FTIR spectroscopic techniques. Among these nine brick samples, only four samples of NB-0 (sun-dried), NB-900 (fired at 900° C), NB-1000 (fired at 1000° C) and NB-1100 (fired at 1100° C) were selected to carry out present research work.

A small broken piece of each brick sample specimens was thoroughly hand grounded in agate mortarto make a fine powder for XRD and FTIR analyses. Mineralogical phase characterization of the clay brick samples was carried out at Nepal Academy of Science and Technology (NAST), Khumaltar, Lalitpur using XRD analysis on a D8 Advanced Diffractometer (Bruker, Germany) with CuK α radiation ($\lambda = 0.15418$ nm) at a scanning rate of 2°/minute in 2 θ mode between 10° and 70°. Major and minor phases present in all the brick powder samples used to record for the XRD measurement were identified from their basal spacing (d-spacing) in accordance with corresponding JCPDS database files [23]. The brick powder sample was loaded in an XRD sample holder in a randomly orientated way to minimize preferred orientations of clay minerals. Furthermore, identification of the mineralogical phase present in three fired clay brick samples at 900°, 1000° and 1100° C and one sun-

dried sample used in this study were qualitatively analyzed with the help of their recorded FTIR spectra using IR-Tracer-100 (Shimadzu, Japan).

3. Results and Discussion

Mineralogical phases present in the powder form of the sun-dried brick collected from the local factory of Kathmandu valley and its fired clay brick samples at three different firing temperatures of 900°, 1000° and 1100 °C raw materials were identified with the help of the XRD patterns as shown in Figs 1-4. The mineralogical phases identified in the powder sample of the sun-dried clay brick (i.e., NB-0) are mainly of muscovite type of mica mineral with feldspars, quartz and hematite as shown in Fig. 1. However, the mineralogical phases existed in other three fired brick samples of NB-900, NB-1000 and NB-1100 fired at the temperature of 900°, 1000° and 1100 °C, respectively, are found to be almost same phases, i.e., quartz, feldspars, alumina rich spinel and hematite including mullite as shown in Figs 2-4. In particular, the fired clay brick sample of NB-1100 shows the XRD peaks of mullite including quartz, feldspars, spinel and hematite as shown in Fig. 4.

This result revealed that the muscovite type of mica mineral present in the sun-dried brick sample is found to be disappeared in the range of 900° -1100° C firing temperature. On the other hand, the mullite phase is not observed for the sun-dried and fired clay brick samples of NB-0, NB-900 and NB-1000 as shown in Figs 1, 2 and 3, respectively. Consequently, it can be said that the mineralogical phases developed in the fired clay brick samples produced by firing at different temperatures are different and hence their physico-sintering and mechanical properties should be different. These results show that the disappearance of the muscovite type of mica clay mineral with decreasing the intensity of feldspars diffraction peak and appearance of spinel phase and finally the mullite peaks in the fired clay brick samples is clearly observed with increasing the firing temperature from 900° to 1100 °C.

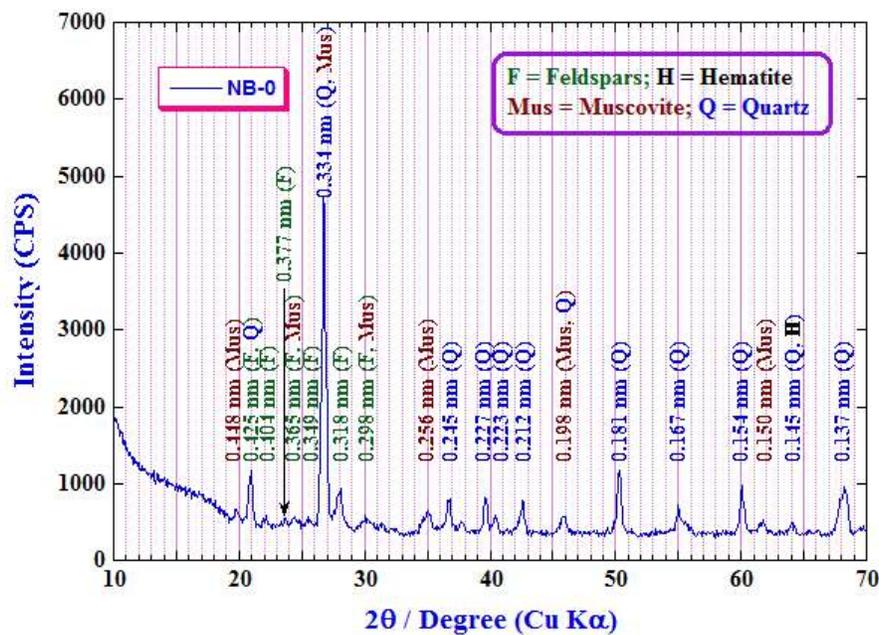


Fig. 1: XRD patterns of the sun-dried brick sample of NB-0.

It was reported that alumina-rich spinel phase was appeared in the ceramic bodies after fired at above 900° C and the spinel phase was lost and mullite crystals begin to develop at about 1050°-1200° C and the spinel phase completely disappeared with increasing firing at 1200 °C and the mullite phase becomes very pronounced [24]. This type of research results of the effect of firing temperature on the development of phases in the ceramic bodies could be helpful to identify the firing temperature of the locally available clay brick samples. It is generally assumed that the physico-sintering and mechanical properties of the fired ceramic bodies depend on the mineralogical composition, defects or faults developed during the firing of the ceramic bodies at high sintering temperatures and impurities [25-28]. For example, the presence of hematite, calcite and dolomite, as impurities of the ceramics raw materials was reported to be one of the causes for low mechanical strength of the fired ceramic bricks at high temperature [29]. In this study, the fired brick samples show the presence of hematite phases, although no indication of the presence of calcite and dolomite phase as impurity.

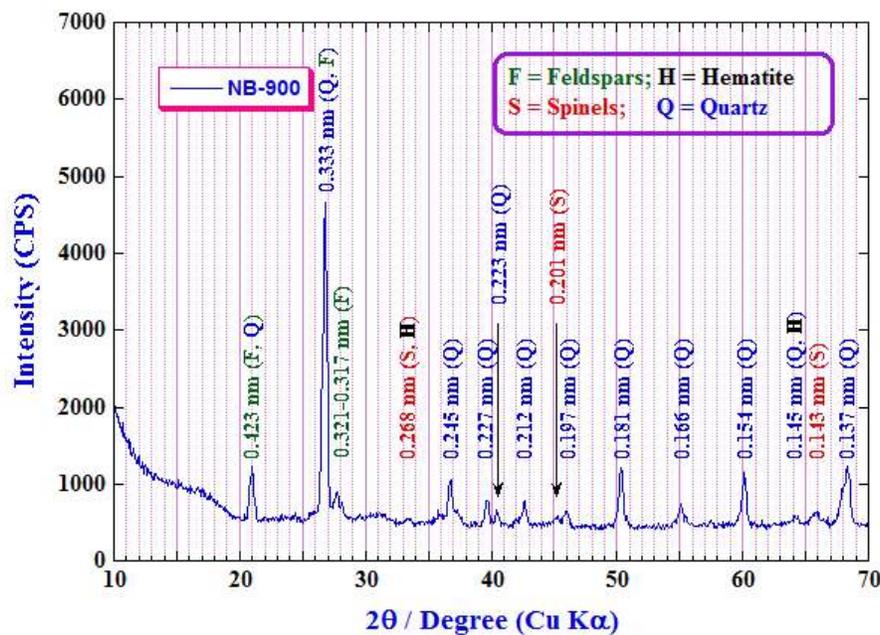


Fig. 2: XRD patterns of the brick sample of NB-900 fired at 900° C.

FTIR spectra analysis of four clay brick samples of NB-0, NB-900, NB-1000 and NB-1100 prepared by sun-dried, fired at 900°, 1000° and 1100° C, respectively, was carried out for the mineralogical phase identification and the results are depicted in Fig. 5. The two absorption peaks at 3725 and 3600 cm^{-1} are almost diminished for all brick samples used in this study which is attributed to OH stretching vibration of hydroxide in the brick samples. This absorption band region should be accompanied by an absorption band in the region of 1620-1650 cm^{-1} assigned to H-O-H bending of adsorbed water molecules [30, 31], although there is not clearly observed the FTIR peak is in this region for all four different clay bricks. The FTIR absorption peak at 2975 cm^{-1} particularly for the brick samples of NB-900 and NB-1000 as depicted in Figs 5(b) and 5(c), respectively, indicated that these brick samples should contained small amount of organic matters which is diminished by firing at about 1100 °C for NB-1100 as shown in Fig. 5(d). The similar observation was reported in previous researchers also [32].

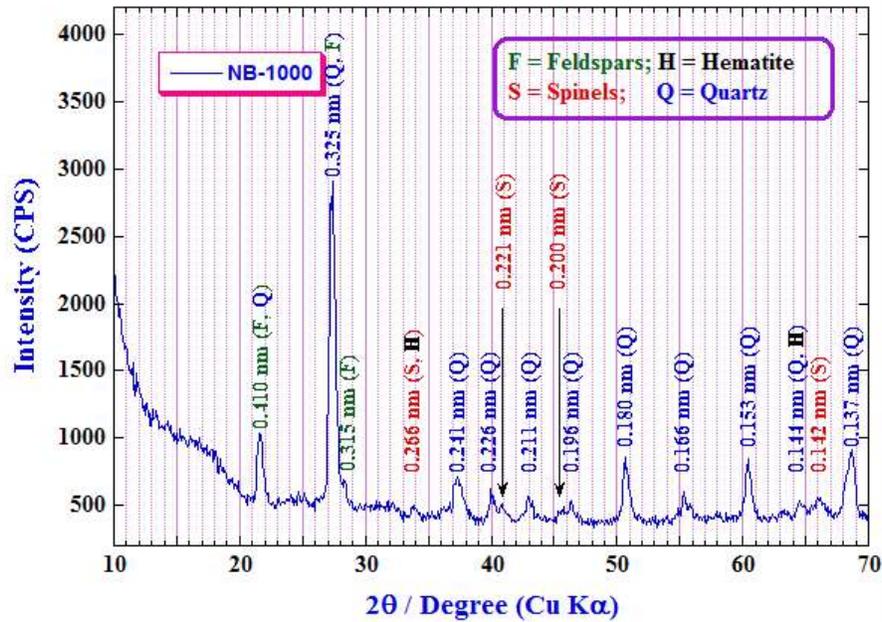


Fig. 3: XRD patterns of the brick sample of NB-1000 fired at 1000° C.

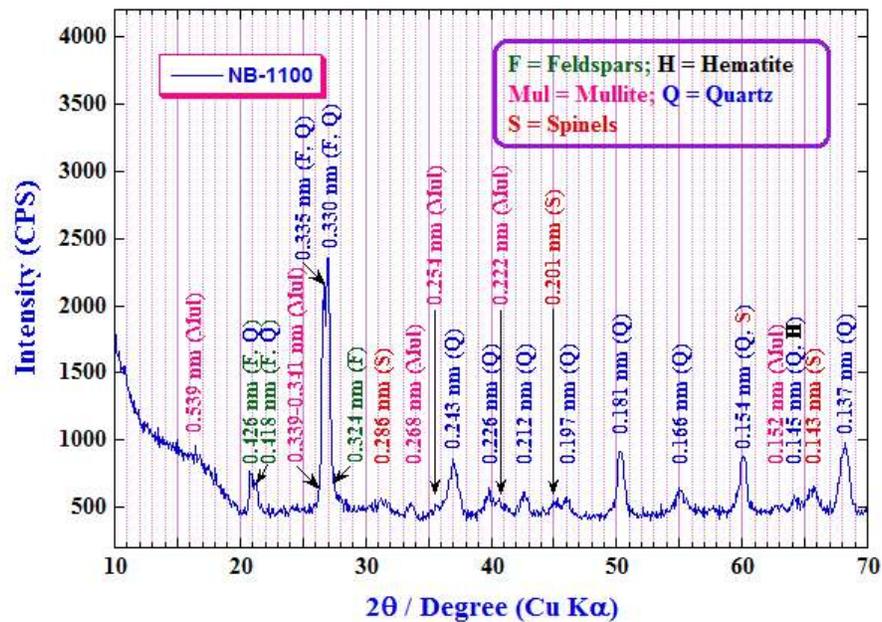


Fig. 4: XRD patterns of the brick sample of NB-1100 fired at 1100° C.

The FTIR absorption bands for clay minerals show Si–O stretching and bending as well as OH bending absorptions in 1300–400 cm^{-1} range. The Si–O stretching vibrations of clay minerals give several well resolved strong bands in the 1100–1000 cm^{-1} region [33]. The FTIR spectra of the sun-dried clay brick sample and the fired brick samples show the absorption band at 1033 cm^{-1} attributed to the Si–O stretching vibrations and the absorption bands at 515 and 445 cm^{-1} assigned to Si–O–Al

(octahedral Al) and Si–O–Si bending vibrations, respectively. A very broad absorption band in the range at 515 cm^{-1} in all the clay bricks used to analyze in this study indicated that the fired clay brick bodies available in Kathmandu valley contain a trace amounts of hematite. The FTIR absorption peak at 1033 cm^{-1} attributed to the Si–O vibrations of the tetrahedral sheet of the clay raw minerals of the fired brick sample and a new absorption peak at $1050\text{--}1040\text{ cm}^{-1}$ assigned to Si–O vibrations of amorphous silica with a three dimensional framework originated from firing treatment [34].

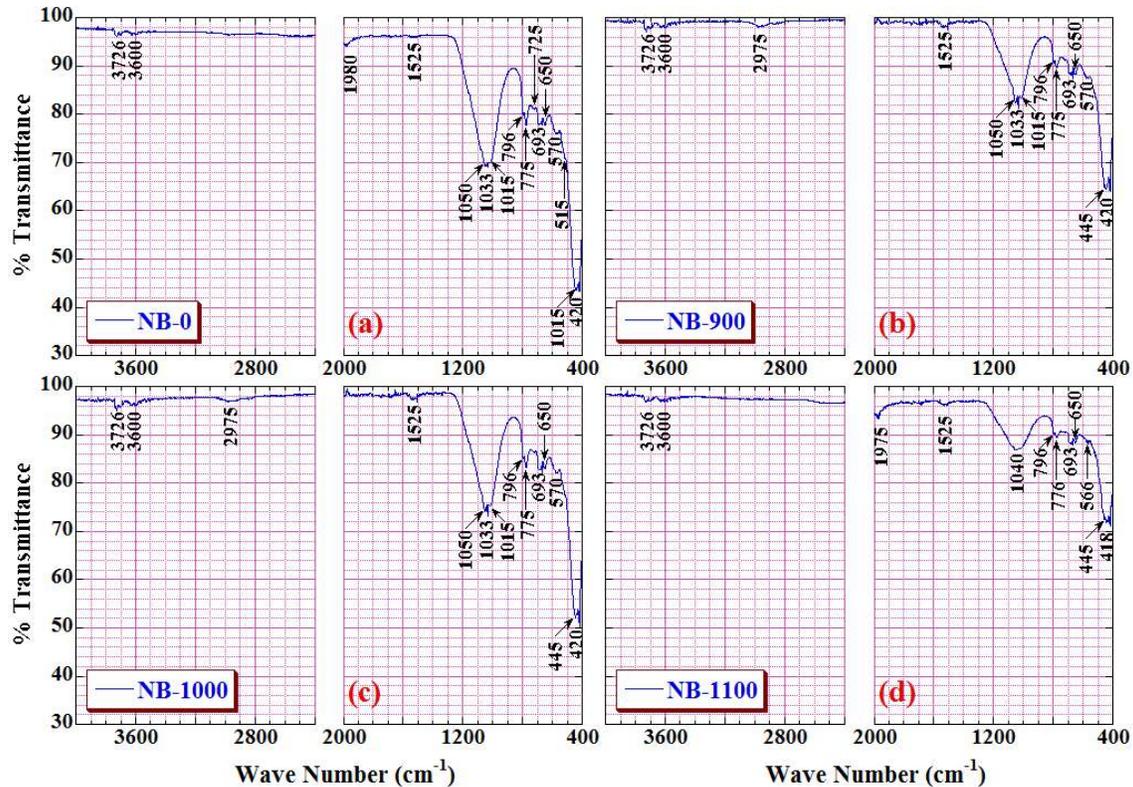


Fig. 3: FTIR spectra of four brick sample specimens of the (a) sun-dried and fired at the temperature of (c) 900° , (c) 1000° and (d) 1100° C.

The FTIR spectra of all these sun-dried and fired clay brick samples showed that they are mainly composed of quartz ($1050, 1033, 1015, 796, 725, 650$ and 445 cm^{-1}). It is meaningful for mentioning here that the previous research work was reported a FTIR absorption peak chosen at around 796 cm^{-1} to be more suitable for quantitative amounts of quartz in clay bodies [35]. On the other hand, the presence of feldspar can be explained by Si–O–Al compounded vibrations at $775\text{--}780\text{ cm}^{-1}$. These assignments are in good agreement with that previously reported for quartz and feldspar [36]. The presence of quartz in the clay brick samples can be explained by Si–O asymmetric bending vibration at 445 cm^{-1} and Si–O symmetric bending vibration at 693 cm^{-1} . On the other hand, the presence of feldspar can be explained by Si–O–Al compounded vibration at 775 cm^{-1} and symmetrical bending vibrations at $570\text{--}556\text{ cm}^{-1}$. These assignments are in good agreement with that previously reported for quartz and feldspars FTIR absorption peak value [37]. The absorption band at $775\text{--}780\text{ cm}^{-1}$, which is a complementary peak of 796 cm^{-1} band, is attributed to the presence of quartz [38]. In the

present work, quartz is identified as predominant and makes the clay self-tempered during firing of clay raw materials at high temperature to produce the clay brick samples. These FTIR analysis results are in agreement with the results obtained from XRD analysis as shown above in [Figs 1-4](#).

4. Conclusions

The investigation of the firing temperature effect on the mineralogical phase developed in the clay brick samples of Kathmandu valley was studied using XRD and FTIR techniques. Following conclusions are drawn from the above results and discussion.

- i. Mineralogy phases in the brick samples used in present study are found to be composed mainly of quartz, feldspars, spinel, hematite including muscovite type of mica mineral based on the XRD patterns and FTIR spectra analyses.
- ii. The decreased in the intensity of the diffraction peaks of feldspars and appearance of the alumina-rich spinel phase in the brick samples fired at the range of 900°-1000° C is clearly observed.
- iii. The alumina-rich spinel phase formed on the fired brick samples enhanced to form the primary mullite phase at 1100° C firing temperature.

Acknowledgement

The University Grants Commission-Nepal (UGC-Nepal) is highly appreciated for awarding the Faculty Research Grant-2072/073 to conduct this research work in Central Department of Chemistry, Tribhuvan University.

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