Structural and Mechanical Properties of Non-Glazed Ceramic Tiles Developed from Selected Mineral Deposits in Uganda

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Abstract

Uganda is well endowed with clay resources; however, comprehensive knowledge about the composition, structure and suitability of these clays for ceramic tile production is lacking. In this study, we provide a comprehensive characterisation of locally sourced clays in Uganda and their suitability for ceramic tile production. In the study, we developed ceramic tiles using feldspar, kaolin, ball clay and sand from four different sites in Uganda. We focused on analysing the surface morphology, crystallographic structure and mineralogical composition of the raw materials. In addition, we examined the mechanical properties of the developed tiles with the different mixture ratios of the clay types. The surface morphology of the raw materials was analysed by using a scanning electron microscope. The structural analysis of the raw clay materials was done using X-ray diffraction. The mineralogical composition of the raw materials was investigated using energy dispersive X-ray spectroscopy and Xray fluorescence spectroscopy. The results indicated that the strength and rapture modulus are influenced by the composition of kaolin and feldspar. We concluded that the selected mineral deposits can be used in the production of ceramic tiles in Uganda.

Keywords: Clay minerals, ceramic tile, XRD, SEM, EDX, XRF, mechanical properties

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Introduction

Since the dawn of time, construction has been a crucial component of human activity to cope with harsh environments and climate [1]. Natural materials that are resilient and long-lasting were used, and included stone, wood, clay, concrete, bricks, metals, sand, lime bricks, concrete bricks, concrete blocks, non-clay bricks, plastics and tiles. The first materials used were those that were found naturally, such as clay, stone and wood.

One material that has been used to produce ceramics is the naturally occurring clay. The word ceramics is derived from the Greek word *keramos* which means potter's clay or products obtained through the action of fire upon materials [2]. Ceramic materials are compounds of alumina and silica bonded by strong ionic and covalent bonds thus making them strong and useful in nature [3]. Around 2600 BC, Egyptians constructed the step pyramids using ceramic tiles because of the durability of ceramic-made goods. Ceramics originated in the field of pottery and were used to decorate tombs [4]. Later, ceramics were used for making fire bricks, insulators, sanitary and kitchen ware, resistors, magnetic memory systems, optical communication devices, and tiles, among other things.

The tiles produced can be of different types and given different names, such as ceramic, porcelain, mosaics, encaustic, cement and natural stone tiles [5]. Mosaics consist of small pieces of tiles arranged on a sheet. When laid and grouted, these tiles give the impression of thousands of tiny tiles. Mosaics are used in bathrooms. Encaustic tiles are made using coloured clays, while porcelain tiles are produced using finer, dense clays. Ceramic tiles are preferred for their beauty and durability, and can be used on almost any wall or floor at home and also busy commercial floors [6]. They are one of the hardest and durable kinds of tile on the market and can last for 10 to 20 years with proper maintenance. They are resistant to corrosion and have a high strength, making them ideal for homes in warmer climates [5]. Owing to the above distinct characteristics of ceramic tiles, there is an increasing need for the installation of tiles on floors and walls of structures in both developed and developing countries.

Uganda is well endowed with mineral resources such as clay and gold which offer a wide range of industrial investments and business opportunities [7]. Various researchers have characterised different minerals from different clay deposits in Uganda and others have investigated the mechanical properties of ceramic tiles. Olupot [8] characterised feldspar and quartz raw materials from Mutaka (Bushenyi), Lunya (Kayunga) and Lido Beach (Wakiso) with the aim of assessing their potential as raw materials in the manufacture of electrical porcelain insulators. The results of their studies indicated that Mutaka feldspar was purely microcline and suitable for use with minimal beneficiation and was therefore suitable for use in the manufacture of electrical porcelain insulators. Olupot *et al.* [9] characterised feldspar and quartz from Mutaka, Lunya and Lido Beach but did not develop tiles from these sites [8].

Moses and Ben [10] studied the effect of holding time on the compressive strength of fired slabs produced from Ntawo clay deposits in Mukono. The results indicated that the compressive strength of ball clay beyond 800 °C did not need longer holding time since the compressive strength was not affected beyond this temperature [10]. The structural characterisation of structural, surface morphology and chemical composition were not studied. Kirabira *et al.* [11] characterised raw mineral powders from Mutaka, Mutundwe and Ntawo clay deposits with the aim of ascertaining their suitability for developing ceramic products with emphasis of fireclay refractories. The bulk density obtained was 1 938 kgm⁻³ (standard value between 1 900 and 2 000 kgm⁻³) and the cold crushing strength was 44 MPa (standard value must be greater than 22 MPa). These results indicated that kaolin from Mutaka and ball clay from Mukono were the deposits recommended as viable and reliable supplies of raw materials for the manufacture of fireclay refractory bricks [11].

Ochen [12] produced porcelain stoneware tiles using kaolin and feldspar from Mutaka in Bushenyi, ball clay from Ntawo in Mukono and sand from Lido Beach in Wakiso. The chemical analysis carried out by the X-ray fluorescence method revealed 24.9% alumina content in Mutaka kaolin and 95.0% silica content in Lido sand and 3.2% iron oxide content in ball clay from Mukono [12]. Ochen developed tiles and determined the shrinkage, strength and water absorption. The tiles exhibited strength of 34 MPa, 0.0% water absorption and shrinkage of 9% (weight) [12]. All these values were in accordance with those given in the South African National Standards for standard tiles.

Uganda has many various clay deposits but limited studies had been carried out on clay from some of the sites with regard to their characterisation and suitability for production of tiles. Recently, there has been a steady increase in the usage of tiles in Uganda and interest to produce tiles locally. There are plenty of clay materials from different parts of Uganda, but there is a lack of dependable knowledge about the suitability of the local clays regarding their composition, structure and potential or suitability to produce ceramic tiles. In this study, we therefore developed tiles using clays from four clay sites and investigated the surface morphology, crystallographic structure and mineralogical composition of the raw materials used. We then determined the variation of mechanical properties of the developed tiles with the different mixture ratios of the clay types. The specific objectives of this study were to determine the surface morphology, crystallographic structure and mineralogical composition of kaolin, feldspar, ball clay rich clays and sand from the four sites and to investigate the compressive strength and modulus of rapture of the tiles produced using different mixture ratios of the four clay types.

Materials and Methods

Mineral Collection and Preparation

Four clay types were collected from four locations, namely, Ntawo in the Mukono District, which is rich in ball clay, Lunya in the Kayunga District, which is rich in feldspar, Buwambo in the Luwero District, which is rich in kaolin and Lido Beach in Entebbe, where sand was collected. Figure 1 illustrates the four locations. Each mineral was excavated 60 cm below the surface and weighed a total of 300 kg. The minerals were collected and then placed in polyethene bags for storage.



Figure 1: Map showing the locations of Ntawo, Lunya, Buwambo and Lido Beach deposits

The ball clay was left to mix in 50 litres of water for a week to separate the heavy sand particles, pebbles and rock pieces from light vegetative impurities such as roots, grass and leaves. The clay paste was taken out of the plastic container after removing the plant material, leaving the heavy pebbles and stones to settle at the bottom. The ball clay paste was then poured thinly over a flat metal surface and allowed to dry in open air inside a

room for a week. The dry clay was ground in a ball mill. The milled clay was then sieved (75–80 μ m sieve).

The kaolin-rich clay was dried in the sun for five days and grounded into a powder and then sieved (75–80 μ m sieve). The sieved kaolin powder was placed in buckets and then covered. The feldspar rich clay was dry milled for 50 hours using a ball mill running at a speed of 50 rev/min and thereafter sieved (75–80 μ m sieve). The sand was wet milled using a ball mill for 40 hours running at a speed of 50 rev/min using porcelain milling stones and then sieved (75–80 μ m sieve). The wet sand was then poured onto a flat surface, left for five days, dried in the sun and sieved (75–80 μ m sieve). The composition of ball clay, kaolin, feldspar and sand was measured and mixed as presented in table 1.

Raw material	Composition ratio (wt %)					
	Α	В	С	D		
Ball clay	25	25	25	25		
Kaolin	40	30	25	20		
Feldspar	30	40	45	50		
Sand	5	5	5	5		
Total	100	100	100	100		

Mineralogical Characterisation

The surface morphology of the raw materials was investigated by a scanning electron microscope (SEM). Structural analysis of the raw clay materials was done using X-ray diffraction (XRD) at a wavelength of 0.15418 nm. The mineralogical composition of the raw materials was investigated using energy dispersive X-ray spectroscopy (EDX) and X-ray fluorescence spectroscopy (XRF).

Tile Production

A total of 25 samples for each tile composition were made. Tile composition A had a mixture of 25 g of ball clay, 40 g of kaolin, 30 g of feldspar and 5 g of sand to form a mixture of 100 g. Next, 5 cm^3 of water was added to the powder and mixed for 15 minutes. The powder mixture was semi-dried and poured into a rectangular steel mould of inner dimensions $110 \text{ mm} \times 50 \text{ mm} \times 10 \text{ mm}$. The mould was then placed in a jigger table for two minutes to level the content and then transferred to a compact compression machine where a force of 170 kN was applied to the mixture. The sample was then carefully removed from the mould and left to dry in the shade for one week. The same procedure was repeated for all the samples. Tile compositions B, C, and D

were formulated as presented in Table 1 after following the same procedure for tile composition A. The dried samples of tile compositions A, B, C, and D were annealed at 1 100 °C for 50 minutes and left to cool to room temperature. The samples were then kept in buckets for further analysis.

Results and Discussion

Crystal Structural Properties of Raw Minerals

The crystal structural properties for kaolin, ball clay, sand and feldspar were determined by the X-ray diffraction technique. The diffraction data were compared with standard data from files of the Inorganic Crystal Structure Database (ICSD). Figure 2(a–d) shows the diffraction pattern for all the minerals and their corresponding ICSD data.

In Figure 2(a), which shows the XRD pattern for kaolin, the diffraction peaks are located at $2\theta/^{\circ} = 12.29$, 20.30, 24.80, 26.70, 27.40, 35.89, 38.39, 41.73, 46.89 and 66.31° and compared with ICSD data. The major peaks are located between 12.29 and 24.80° and are similar to the major peaks for kaolin located between 12.26 to 24.81° in the literature [13], [14]. This confirms that the mineral used is kaolin. In Figure 2(b), which shows the XRD pattern for ball clay, the scattering peaks were located at 2θ (°) = 12.27, 20.82, 25.03, 26.53, 27.35, 36.84, 42.41, 49.88, 60.07 and 68.08° and compared with ICSD data. The major peaks are located at 20.82, 26.53 and 27.35°. As indicated in the literature, standard ball clay has kaolinite as the main mineral [15]. This confirms that the mineral used is kaolin clay has kaolinite as the main mineral [15]. This confirms that the mineral used is kaolin. As indicated in the literature, standard ball clay has kaolinite as the main mineral [15]. Figure 2(c) shows the XRD pattern obtained for sand (silica) with a broad peak at $2\theta = 22.86^{\circ}$. The broad peak shows the amorphous nature of the silica nanoparticles. In Figure 2(d), which shows the XRD pattern for feldspar, the scattering peaks are located at 2θ (°) = 20.69, 26.40, 29.52, 39.43, 50.28, 54.77, 60.21 and 67.93° and compare well with ICSD data.



Figure 2: XRD pattern for raw (a) kaolin, (b) ball clay, (c) sand, and (d) feldspar

Surface Morphology of the Raw Minerals

The surface morphology and histograms of variation in particle size distribution of kaolin, ball clay, sand and feldspar minerals are shown in Figure 3.

Figure 3(a) and (b) shows the surface morphology and a histogram of variation in particle size distribution of kaolin. The particles are irregular in shape with a porous texture. In addition, they are agglomerated with average particle size of approximately $0.202 \pm 0.131 \,\mu$ m. Figure 3(c) and (d) shows the surface morphology and a histogram of variation in particle size distribution of feldspar. The samples show an irregular surface morphology of particles with average particle size of approximately $0.153 \pm 0.105 \,\mu$ m. The surface morphology and histogram of variation in particle size of ball clay are shown in Figure 3(e) and (f). The ball clay particles are agglomerated, fine grained and form uniformly rounded crystals in pockets between kaolinite fragments with an average size of approximately $0.095 \pm 0.055 \,\mu$ m. Figure 3(g) and (h) shows the surface morphology and a histogram of variation in particle size distribution of sand. The SEM image of silica displays the silica particles in an agglomerated form. It also displays fine silica particles with a porous agglomerated texture with average particle size of approximately $0.168 \pm 0.198 \,\mu$ m.



Figure 3: (a) SEM image of kaolin, (b) Histogram for particle size distribution of kaolin particles, (c) SEM image of feldspar, (d) Histogram for particle size distribution of feldspar particles, (e) SEM of ball clay, (f) Histogram for particle size distribution of ball clay particles, (g) SEM image of sand (h) Histogram for particle size distribution of sand particles

EDX/XRF Results of the Raw Minerals

The sampling areas and EDX spectra showing the mineralogical composition of kaolin, ball clay and feldspar are shown in Figure 4.

Figure 4(a) and (b) shows the sampling areas and EDX spectra of kaolin respectively. The observed peaks for kaolin included potassium, oxygen, aluminium and silicon. We observed that the intensity for oxygen is higher than that of aluminium. The aluminium peak is slightly higher than that of silicon. The average percentages by mass of kaolin were obtained through quantitative analysis and based on five measurements, namely, 51.61% for oxygen, 11.15% for aluminium, 10.83% for silicon and 0.43% for potassium. The ratio of silicon to aluminium in the kaolin was ~ 1:1, thus confirming the presence of kaolin as the major mineral.

Figure 4(c) and (d) shows the sampling areas and EDX spectra of ball clay respectively. The elemental analysis by EDX on ball clay showed the presence of aluminium and silicon, which is in agreement with the XRD results for kaolin. The EDX spectra on ball

clay also show peaks of iron, potassium, calcium and titanium. The results indicated that the ball clay is characterised by oxygen content of 45.53%, silicon (Si) content of 12.91% and aluminium (Al) content of 4.52%. The clay showed contents of sodium (Na) of 0.39% and iron (Fe) of 0.86%. The percentage weights of titanium (Ti), calcium (Ca) and potassium (K) in the ball clay were 0.30%, 0.41% and 0.28% respectively. the EDX results for experimental ball clay is in agreement with its XRD results.

Figure 4(e) and (f) shows the sampling areas and EDX spectra of feldspar respectively. The observed peaks contains mainly metallic elements of sodium, calcium, aluminium and magnesium with non-metallic elements of silicon and oxygen. These kinds of EDX spectra belong to feldspar whose molecular formula is (Na,Ca)AlSi₃O₈) [16].



Figure 4: (a) Sampling areas of kaolin, (b) EDX spectra of kaolin, (c) Sampling areas of ball clay, (d) EDX spectra of ball clay, (e) Sampling areas of feldspar, (f) EDX spectra of feldspar

Elemental Composition of Sand

The mineralogical composition of sand was characterised by XRF. The results indicate that sand is mainly composed of silica with trace amounts of aluminium oxide (Al₂O₃) and iron (III) oxide (Fe₂O₃). The major oxide of interest was silicon dioxide (SiO₂). The percentage weight of SiO₂, Al₂O₃ and Fe₂O₃ were 99.5%, 0.2% and 0.2% respectively. The XRF results were in agreement with the XRD results for sand.

XRD Results of Tile Compositions after Firing

The phases present in the fired tile compositions A, B, C and D were identified by comparing them with the standard ICSD data of the expected phases after firing. Figure 5 shows all the phases present in the tiles after firing and the phases are

summarised in Table 2. The XRD peaks shows the highly crystalline tiles. It can be observed that in all the tile samples, the dominant kaolinite mineral constituent phase is present with other phases that have evolved as a result of firing.



Figure 5: XRD pattern for the tile compositions A, B, C and D after firing

Sample No	Scattering angle, 20 (°)					ICSD No
	Tile A	Tile B	Tile C	Tile D	Phase	
1	12.3	12.3	12.3	12.3	Kaolinite	63316
2	16.4	16.4	16.4	-	Mullite	66263
3	17.5	17.5	17.5	-	Illite	90144
4	20.8	20.8	20.8	20.8	Quartz	16331
5	-	-	-	21.81	Illite	90144
6	22.4	22.4	-	-	Calcite	10264
7	23.0	-	23.0	-	Calcite	10264
8	26.6	26.6	26.6	26.6	Quartz	16331
9	27.48	27.48	-	-	Kaolinite	63316
10	29.44	29.44	29.44	-	Calcite	10264
11	34.89	34.89	34.89	-	Illite, kaolinite	90144, 63316
12	35.95	35.95	35.95	-	Kaolinite	63316
13	36.51	36.51	36.51	36.51	Quartz	16316
14	36.97	36.97	36.97	36.97	Mullite	66263
15	38.36	38.36	38.36	-	Kaolinite	63316
16	39.41	39.41	39.41	39.41	Quartz	16316
17	42.36	42.36	42.36	42.36	Quartz	16316
18	45.56	45.56	45.56	45.56	Illite	90144
19	47.31	47.31	47.31	47.31	Mullite	66263
20	50.00	50.00	50.00	50.00	Quartz	16316
21	55.05	55.05	55.05	55.05	Kaolinite, illite	63316, 90144

Mechanical Properties

The moduli of rapture and compressive strength were tested for the tiles produced for the four mixture ratios A (5:8:6:1), B (5:6:8:1), C (5:5:9:1) and D (5:4:10:1). The optical images of these tile compositions after firing are shown in Figure 6(a). Bar graphs of the modulus of rapture and compressive strength against the composition ratios were plotted to show the variation in modulus of rapture and compressive strength of the tile samples with composition ratios (see Figure 6(b) and (c)).



Figure 6: (a) Optical image of tile samples A, B, C and D after firing (b) Bar chart of modulus of rapture of tile samples against tile sample ratios (c) Bar chart of compressive strength of tile samples against tile sample ratios

Tile composition A has the highest modulus of rapture followed by tile compositions B, C and D, which had the least modulus of rapture. Based on the literature, the mechanical strength of ceramic ware depends on the mullite (3Al₂O₃.2SiO₂) content and microstructure. The higher the mullite content, the higher the strength of the ceramic ware [17]. The proportions of kaolin, feldspar, sand (quartz) and ball clay influence the phase compositions and mechanical properties of the fired ceramic tiles [18]. The modulus of rapture of the tile samples developed using composition A can be correlated with the presence of the mullite content and microstructure. Tile composition A had the highest kaolin content of 40%, whose chemical formula is identical to that of mullite when compared with tile compositions B, C and D, which had a low kaolin content.

Similarly, tile composition A has the highest compressive strength followed by tile compositions B, C and D, which had the least compressive strength. Based on the literature, the mechanical strength of ceramic ware depends on mullite (3Al₂O₃.2SiO₂) content and microstructure. The higher the mullite content, the higher the strength of the ceramic ware [17]. The proportions of kaolin, feldspar, sand (quartz) and ball clay influence the phase compositions and mechanical properties of the fired ceramic tiles [17] [18] [19] [20], [21]. The compressive strength of the tile samples developed using tile composition A can be correlated with the presence of the mullite content and microstructure. Tile composition A had the highest kaolin content of 40%, whose chemical formula is identical to that of mullite when compared with tile compositions B, C and D, which had low kaolin content. Tile composition A in the ratio of 5:8:6:1 was therefore the recommended composition for the production of tile samples.

Summary

In this article, we characterised and used feldspar, kaolin, ball clay, and sand from mineral resources in Uganda to make ceramic tiles. We evaluated the compressive strength and modulus of rapture of the developed tiles. According to the EDX results and XRD findings, the experimental kaolin contains silicon and aluminium in amounts of 10.83% and 11.15%, respectively. The feldspar used in this study was calcium aluminium silicates, the EDX and XRD results agreed and the sand used was amorphous silica. With a kaolin–feldspar ratio of 4:3, tile composition A had the highest modulus of rapture (~ 30.836 MPa) and compressive strength (1.358 MPa), followed by tile composition B with a ratio of 3:4, tile composition C with a ratio of 5:9, and tile composition D with a ratio of 2:5, which had the lowest modulus of rapture (18.58 MPa) and compressive strength (0.818 MPa).

In this study, the null hypothesis (HO₂) was that the composition ratios of the clay mixture would not have an impact on the rapture modulus of the tiles produced. Since the modulus of rapture for the four compositions was statistically different at the two levels of significance of $\alpha = 0.01$ and $\alpha = 0.05$, this hypothesis was rejected. At the two levels of significance of $\alpha = 0.01$ and $\alpha = 0.05$, the compressive strength for the four composition ratios of the clay mixture will not affect the compressive strength of the tiles produced. We concluded that the compressive strength and rapture modulus are influenced by the composition of kaolin and feldspar.

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Author Contributions

All the authors contributed to the study conception and design. The material preparation, data collection and analysis were performed by George William Mukwaya and Emma Panzi Mukhokosi. The first draft of the manuscript was written by George William Mukwaya. All the authors read and approved the final manuscript.

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