

Influence of CRT Glass Quantity on the Properties of Red Mud-CRT Glass Ceramics Fired at Different Temperatures

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Abstract: This paper reports the production of ceramics using CRT (cathode ray tube) glass and red mud by varying the amount of waste glass and firing from 700-950 °C. The raw materials were characterized using chemical composition, IR (infra-red spectroscopy), XRD (X-ray diffraction), DSC-TGA (differential scanning calorimetry-thermo gravimetric analyses) and SEM (scanning electron microscopy) analysis. The ceramic specimen was produced by mixing a constant quantity of red mud and 0%-40% of waste glass, then firing from 700-950 °C. The linear shrinkage, water absorption, apparent porosity, bulk density, weight loss on ignition, flexural strength and chemical resistance test were used to evaluate the produced ceramics. Raw materials had good fluxing properties which were improved with firing. All the specimens produced had water absorption values below the maximum value of 20% specified by the norm for roofing tiles while specimens with 30%-40% CRT glass and fired at 850 °C, and specimens with 20%-40% CRT glass and fired at 950 °C had water absorption values below 10%, which is the norm for ceramic tiles. For specimens 0%-40% CRT glass fired at 950 °C, specimens 5%-40% CRT glass fired at 850 °C, specimens 10%-40% CRT glass fired at 750 °C and specimen with 20% CRT glass fired at 700 °C, water absorption values were all lower than 17% which is ASTM C62 specifications for bricks. The norm of flexural strength for floor tiles > 25 MPa but all values were less than 25 MPa. The ceramic shows a maximum loss of 0.10% in HNO₃ against 0.06% for NaOH. The two waste materials can effectively be used to produce ceramic materials with good properties.

Key words: CRT glass, red mud, ceramics, firing, roofing tiles, floor tiles.

1. Introduction

One of the greatest environmental challenges of the 21st is the management of waste particularly solid waste. Integrated waste management, prioritizing the reduction of waste at source or recycling of the generated waste is currently being advocated worldwide as a means of protecting the environment from the effect of dumping of industrial, domestic, electronic etc. solid wastes. However, most wastes are viewed today as valuable resources for the production of other useful materials. For example, red mud as well as glass has been added to clay to produce bricks and ceramics or for partial replacement of concrete

[1-5].

Red mud is the solid waste by-product resulting from the production of alumina from the Bayer Process. On average globally, between 1 and 1.5 tons of red mud generated per ton of alumina produced and over 150 million tons of the residue are produced [6]. Unfortunately, it is estimated that only about 2-4.5 tons are used annually in some way (Cement: 500,000 to 1,500,000 tons; Raw material/additive in iron and steel production: 200,000-1,500,000 tons; Roads/landfill capping/soil amelioration: 200,000-500,000 tons; Construction materials (bricks (tiles, ceramics, etc.): 100,000-300,000 tons; other (refractory, adsorbent, acid mine drainage, catalyst, etc.): 300,000 tons) [6]. This means over 145 million tons are being dumped in different parts of the world

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with the risk of causing another disaster like the Ajka, Hungary red mud spill that killed ten people, injured 200, and eliminated all aquatic life in a 71 km stretch [7]. Currently, most red mud produced from alumina plants is disposed in landfills or dumped at sea. The cost of disposal is very high, constituting for approximately 5% of alumina production [5]. Hence the question arises what to do with these large volumes of red mud with high polluting character. Because it contains compounds of Na, Ca, Si and K, it can act as fluxing agent and so could be of particular importance for use in the production of bricks, roofing tiles, rustic floor and wall tiles [1].

One other waste material that can be used as a brick or ceramic additive is waste glass. It is not biodegradable, thus creating disposal problems as it is disposed principally in landfills with significant environmental impacts. According to the United Nations estimates, 200 million tons of solid wastes are disposed annually worldwide with glass constituting 7% [2]. In some developing countries like Palestine, about 1.2 million tons of solid wastes are generated annually with glass constituting 4% [2]. But there are no existing recycling activities for this glass. The present recycling of CRT (cathode ray tube) glass is mainly for the manufacture of new CRT cement or ceramic [8]. In most developing countries CRT glass presence is still very dominant as most people lack the resources to acquire modern LCD (liquid-crystal-display) televisions. Our recent study on the assessment of e-waste management in Maroua showed that televisions with CRT screens were the most used by the population [9]. Many researchers around the world have studied the use of waste glass as an additive in fired clay bricks, tiles and stoneware to enhance the properties [2]. This is due to its silica, CaO, Na₂O, K₂O and MgO content which are fluxing agents and can thus replace common fluxes such as feldspar or other mineral fluxes to save energy in the ceramic manufacturing process [10]. Most of these studies used glass from 5% to 45% by weight and

firing from 750 to 1,100 °C [2, 10] and using clay as the main material while Vieira et al. [1] prepared clay-red mud ceramics by using red mud as additive (0, 20 and 40 weight %) and firing at 950 °C and 1,050 °C, published studies on the production of red mud glass ceramics, involving the use of glass and particularly CRT glass as additive is very scarce. In this study, red mud-CRT glass ceramics were prepared by varying the quantity of glass from 0% to 40 % and firing at 700, 750, 850 and 950 °C. XRF (X-ray fluorescence), XRD (X-Ray diffraction), IR (Infra-red spectroscopy), TGA-DSC (thermo gravimetric analyses-differential scanning calorimetry) and SEM (scanning electron microscopy) were used to characterize red mud and waste glass while the properties of the produced ceramics were determined using weight loss on ignition, flexural strength, bulk density, porosity, water absorption, linear shrinkage and weight loss in nitric acid and soda solutions. The results of this study will help in the massive utilization of bauxite residue as well as glass, hence protecting the environment and improving the economics of the bauxite industry.

2. Materials and Methods

2.1 Raw Materials Preparation and Characterization

The CRT glass used was collected from local electronic repair workshops in the town of Ngaoundere. The glass shards from the recovered TV screens were washed with tap water to remove impurities and dried at room temperature. They were then crushed and sieved to obtain particle size of 65 µm which were stored and used for characterization studies and ceramic production without further processing.

Due to the fact that Cameroon is not yet processing its bauxite, the bauxite collected at Ngaoundal (6°27'55" N, 13°16'16" E) was used in producing the red mud in the laboratory as described in our previous studies [11, 12]. Sampled bauxite was washed with distilled water, crushed in porcelain mortar and dried at

105 °C for 24 h using a HERAEUS type VT 5042 EK oven. The dried particles were then sieved to obtain particle size of 75 µm used for characterization studies and ceramic production.

The chemical composition of red mud and waste glass was determined using the XRF (AXIOS PANalytical, Dy 1680) while the surface functional groups were determined by FTIR (Fourier transform infrared spectroscopy Spectrophotometer, (Bruker Make), Model: ALPHA-P). The presence of mineral compounds in waste glass and red mud, was determined by XRD using a Philips X'Pert PRO diffractometer. The different diffraction peaks recorded were compared with those of similar samples reported in literature for identification. The DSC-TGA technique (SDT Q600 V20.9 Build 20) was used to identify the different phase transformations involved in waste glass and red mud samples with heating while SEM was used to determine the surface morphology of the solid materials.

2.2 Production and Characterization of the Red Mud-CRT Glass Ceramic Specimens

Ceramic samples were obtained by firstly placing the powder forms of red mud and CRT glass in a porcelain mortar, and then moistening it with demineralised water (Table 1 shows different mixtures). The mixture (75 g, Table 1), was well homogenized and then compacted in a mold (dimensions 82 mm × 42 mm × 9 mm). The pressure of 12 MPa was applied to the mold containing the paste using a hydraulic press (EURO LABO, model 25.011). Obtained ceramic samples were kept in the open air (laboratory conditions) for 48-72 h and then

dried at 110 °C in a muffle furnace (NABERTHERM, model LH 60/40) for 24 h. These samples were then fired at 700 °C, 750 °C, 850 °C and 950 °C for 2 h using this same furnace with a heating rate of 5 °C/min, then allowed to cool down overnight. Tiffo et al. [10] reported that glass starts to melt at about 680 °C, so the choice of firing from 700 °C.

The properties of the specimens obtained from sintering at the four temperatures with varying composition of glass were evaluated.

The loss of mass due to drying at 110 °C and firing at different temperatures was determined using ASTM-C326 standard [13] whereas the determination of the linear shrinkage was done by studying the variation of the mean length of spots recorded on the parallelepipeds between drying at 110 °C and firing at different temperatures as described in ASTM-C326 standard [13].

Water absorption was carried out on cylindrical fired test specimens according to ASTM-C373 standard [14] where ceramic samples were weighed and immersed in a beaker containing distilled water and the ensemble boiled for 2 h, and then allowed to cool for 24 h at room temperature. The specimens were then wiped with a paper towel and weighed.

The flexural strength, determination was done according to ASTM-C 674 standard [15] in which, the parallelepiped ceramic was placed on two cylindrical supports and horizontal portion fixed on a basement of vertical movement. A third motionless cylinder was placed above, parallel and symmetrical to the previous one. The basement that carries both cylinders has an average ascent of 3 mm/min to reach breaking point. The dynamometer then indicates the force exerted to

Table 1 CRT glass-red mud mixtures used for ceramic production.

% CRT glass	Mass (g) of glass used	% Red mud	Mass of red mud (g) used
0	0	100	75.00
5	3.75	95	71.25
10	7.50	90	67.50
20	15.00	80	60.00
30	22.50	70	52.50
40	30.00	60	45.00

break the specimen which is then used to calculate the mechanical resistance to bending.

Apparent porosity, bulk density and porosity were determined by Archimedes' Principle using the ASTM-C373 standard [14].

For the specimen's resistance to acid and base corrosion test, 0.1 N HNO₃, and 0.1 N NaOH were used (NEN-7375 standard) [16]. Samples were dried again for 24 h at 105 °C, and then weighted masses of the respective samples immersed in different 500 mL beakers containing acid or base solutions for 1, 3, 5, 7 and 9 days, after which the samples were withdrawn and carefully wiped with dry tissue, dried again for 24 h at 105 °C in the oven and weighed to get the final mass. The difference between this final mass and the initial mass of sample before treatment with acid or base gives the mass loss due to acid or base corrosion.

3. Results

3.1 CRT Glass and Red Mud Characteristics

The chemical composition of the red mud and waste CRT glass is shown in Table 2 where it is observed that Fe₂O₃, Al₂O₃, SiO₂, Na₂O, CaO and TiO₂ are major components of red mud. The high amount of Na₂O + K₂O (4.77%) and CaO + MgO (2.83%), oxides which act as fluxes can easily facilitate the liquid phase formation during the firing stage of the ceramic [1], hence, ceramic production at lower temperatures. From Table 2, it is equally observed that the waste CRT glass is mainly composed of SiO₂ (56.115), K₂O (10.01%), Na₂O (5.46%) Al₂O₃ (3.02%), CaO (2.56%) and MgO (1.86%) with higher content of fluxing agents. Red mud and CRT glass composition can be grouped into three groups [3]. The

first group called the glassy phase forms the framework and surfaces of ceramics and is composed of SiO₂ and Al₂O₃ content. The second group is the gaseous components constituted by carbon and Fe₂O₃ content, which can generate gases and bloat the ceramic bodies in the firing process. Finally the third group is fluxes constituted of alkali metal oxide and alkaline earth metal such as CaO, Na₂O, K₂O and MgO, which help in lowering down the melting point. From Table 2, it can be deduced that the glassy phases in CRT glass are 59.13% higher than in red mud with (37.28%). The fluxes in waste glass are 19.89% higher than the 7.60% in red mud. Conversely, the gaseous components are higher in red mud (47.47%) compared to 11.32% in waste CRT glass. Thus, the bloating properties of the ceramic will be greatly defined by red mud while the CRT glass will be the major contributor in lowering down sintering conditions and main phases to form the framework inside and outside the ceramics.

The IR curve of CRT glass (Fig. 1a) shows the dominance of quartz material with strong peaks from 967.24 to 417 cm⁻¹ while the weak peak at 1,489.01 cm⁻¹ is assigned to O-C-O [10]. Also the very weak IR band 2,032.08 cm⁻¹ indicates the presence of some organic impurities. The IR spectra of red mud are given in Fig. 1b, where the strong peaks from 3,053.50-3,525.95 cm⁻¹ attributed to the hydroxyl groups while the strong peaks at 1,558.78 and 1,397.68 cm⁻¹ represent the C=O and S-O groups respectively [17, 18]. The peaks at 1,069.93 and 982.90 cm⁻¹ are Si-O groups while the peak at 732.20 cm⁻¹ is the Si(Al)-O group [17, 18]. The bands from 465.04-406.25 cm⁻¹ are the Fe-O groups [17, 18].

Table 2 Chemical composition of the CRT waste glass and red mud.

Sample	LOI	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	P ₂ O ₅	PbO	TiO ₂
Waste glass	11.23	56.11	3.02	0.09	2.56	1.86	0.28	5.46	10.01	0.04	18.34	0.09
Red mud	20.26	7.68	29.6	27.21	2.75	0.08	0.08	4.71	0.06	0.12	-	1.89

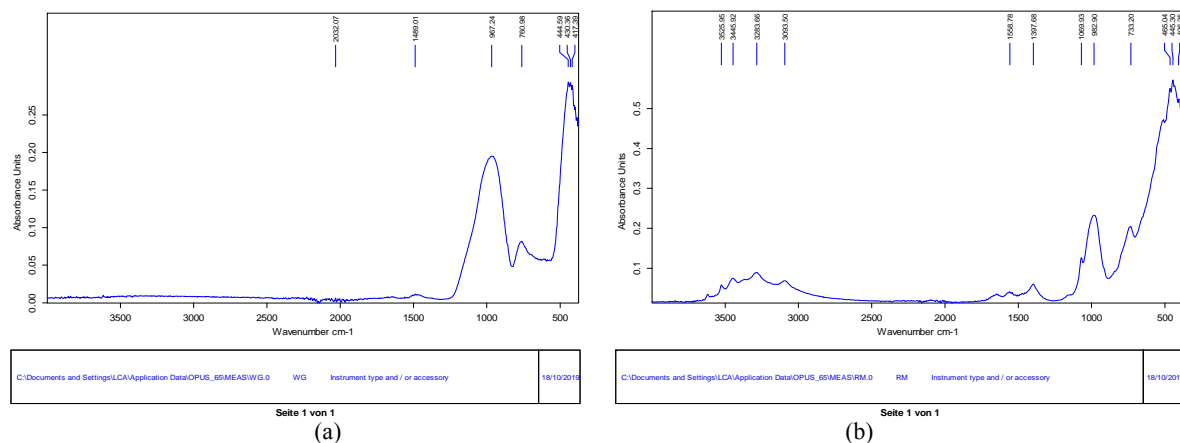


Fig. 1 IR curve of (a) CRT waste glass and (b) red mud.

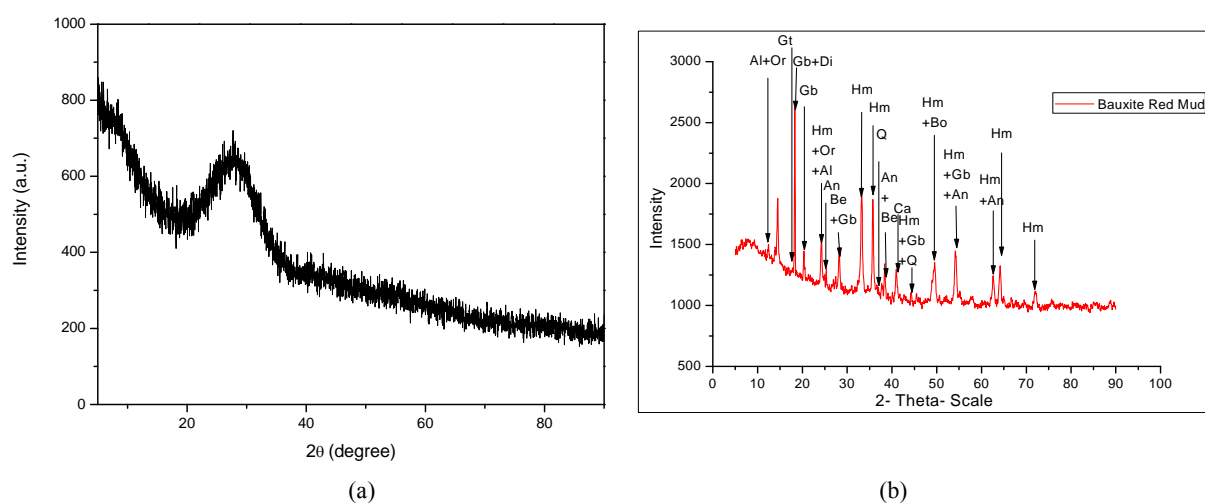


Fig. 2 XRD pattern of (a) CRT waste glass and (b) red mud.

An: Anatase (TiO_2); Hm: Hematite (Fe_2O_3); Di: Diaspore ($\text{AlO}(\text{OH})$); Gb: Gibbsite ($\text{Al}(\text{OH})_3$); Q: Quartz (SiO_2); Ca: Calcite (CaCO_3); Go: Goethite ($\text{FeO}(\text{OH})$); Al: Albite ($\text{NaAlSi}_3\text{O}_8$); Or: Orthose (KAlSi_3O_8); Bo: Bohemite.

The mineral phases in waste CRT glass and red mud were determined using XRD and the curves are shown in Figs. 2a and 2b. From Fig. 2a, a halo shape diffraction peak is observed between 2θ value of 20° and 40° indicative of the presence of amorphous silica [10, 19]. The XRD curve of CRT glass shows the presence of background noise, indicating the presence of impurities in this material. The XRD pattern of red mud is given by Fig. 2b and it is seen to contain the minerals: Anatase (TiO_2); Hematite (Fe_2O_3); Diaspore ($\text{AlO}(\text{OH})$); Gibbsite ($\text{Al}(\text{OH})_3$); quartz (SiO_2); calcite (CaCO_3); Goethite ($\text{FeO}(\text{OH})$); Albite ($\text{NaAlSi}_3\text{O}_8$); Orthose (KAlSi_3O_8); and Bohemite. As reported in literature, it can be seen that this red mud is dominated

by iron minerals [11]. The presence of quartz, orthose and albite silica minerals and alumina minerals confirm the XRF results. The XRD results show the variable contribution of red mud in the glassy phase, the gas components and fluxes of fired ceramics.

The TGA-DSC curves of red mud and waste glass are shown in Figs. 3 and 4 respectively. From Fig. 3, the decomposition of red mud shows three weight loss portions. The first occurs from about $30\text{--}200^\circ\text{C}$ (0.9854%), resulting from the evaporation of physically adsorbed water [3, 11]. The second weight loss occurs from about $200\text{--}375^\circ\text{C}$ (10.70%) and is attributed to the loss of chemically adsorbed water resulting from the decomposition of gibbsite ($\text{Al}(\text{OH})_3$)

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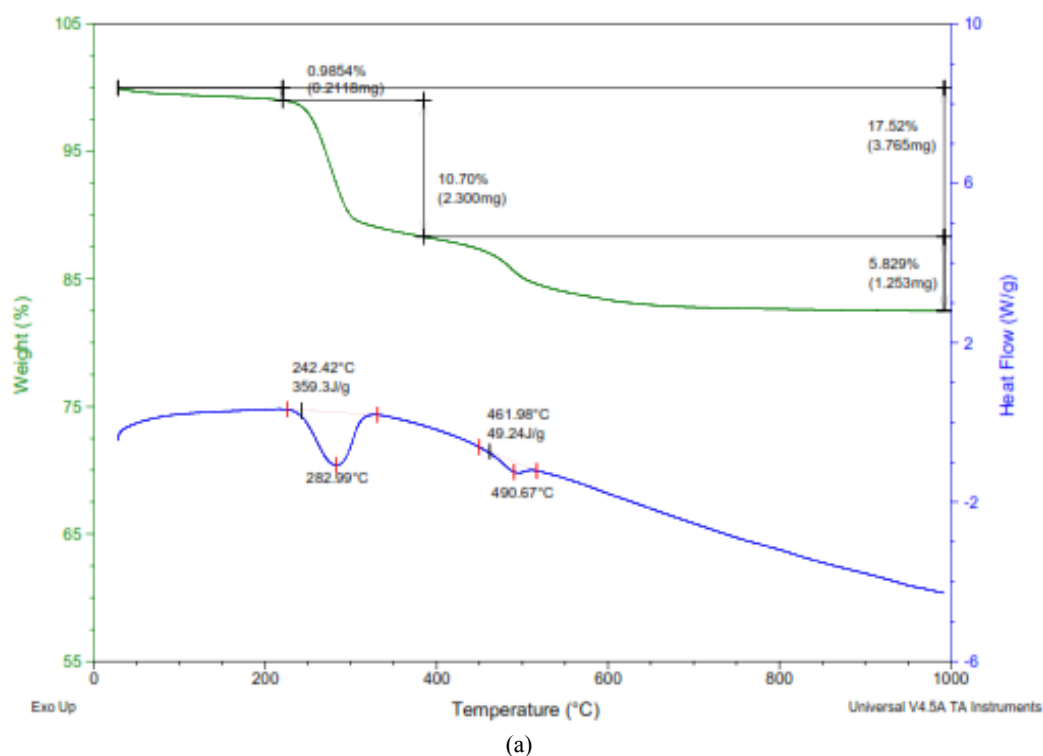


Fig. 3 DSC-TGA curves of red mud (blue is DSC, green is TGA).

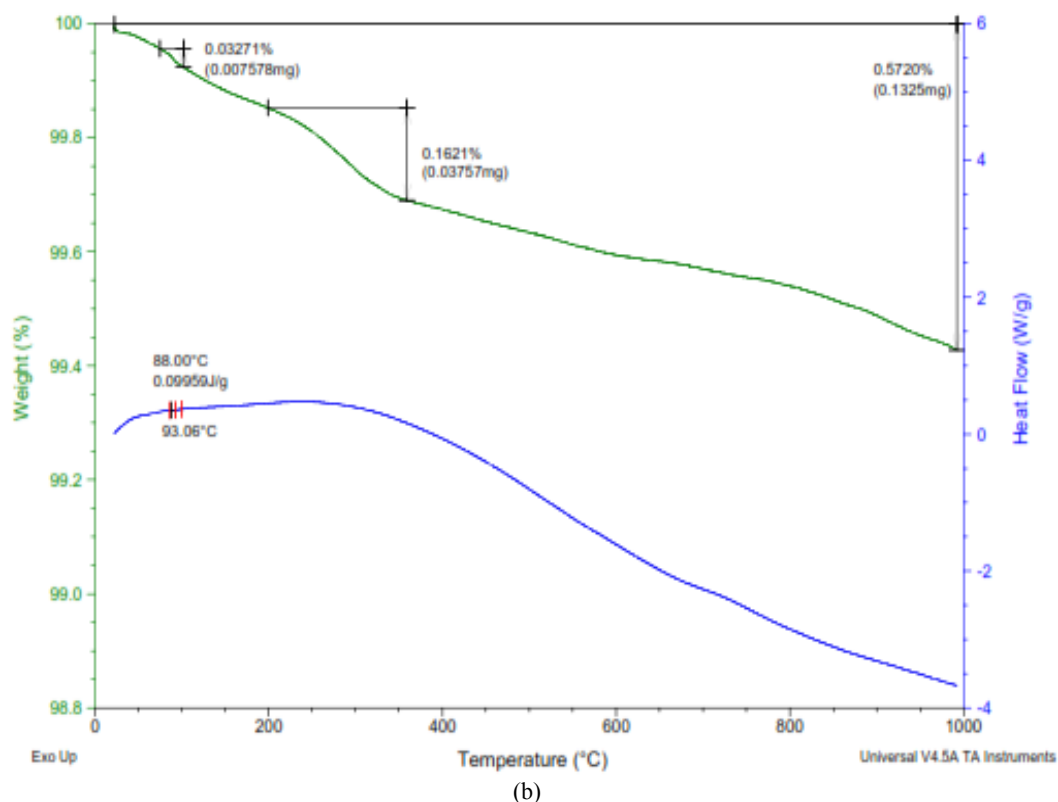


Fig. 4 DSC-TGA curves of CRT waste glass (blue is DSC, green is TGA).

to alumina and H₂O. The third weight loss occurs from about 375-650 °C (0.829%) and is attributed to the decomposition of CaCO₃ to CaO with the release of CO₂. Two endothermic peaks are observed on the DSC curve at 282.99 °C and 490.67 °C, resulting from the release of surface water and chemically bound water [3, 11]. The contribution to the amount of fluxing materials and glassy phase by red mud will increase as from temperature above 650 °C. From the TGA-DSC curves of waste glass presented in Fig. 4, two weight loss portions at 60-100 °C (0.03271%) and 200-300 °C (0.1621%) are observed. The total loss of 0.5720% is insignificant. This small loss may be due to volatilisation of absorbed water and other contaminants [20], confirming the XRD analysis. CRT glass will probably show very little material loss during firing.

The results of the SEM of waste CRT glass and red mud of particle size, 2 µm are shown in Figs. 5a and 5b. The SEM image of glass (Fig. 5a) and that of red mud (Fig. 5b) are seen to have very low porosity and very crystalline structure, an indication that mixing the two raw materials will produce a very homogenized material with low porosity.

3.2 Properties of Produced Red Mud-CRT Glass Ceramics

The properties of the produced red mud-CRT glass

ceramics are presented in Table 3.

The linear shrinkage is seen to increase with increase in CRT glass content at all the firing temperatures (Table 3). However, these values increase with increase of temperature. With increase in temperature there is an increase production of the liquid phase due to the alkaline and earth-alkaline oxides (fluxing oxides) that red mud contains, hence also contributes to the increased shrinkage of the ceramic [1]. The fluxes in waste glass used are 19.89% against 7.60% for red mud. Thus, an increase in CRT glass quantity increases the amount of fluxing agents while increasing firing temperature produces more fluxes from red mud (due to decomposition of mineral phase) and causing water to evaporate out of the system. This process causes the ceramic particles to fuse together with higher proximity, thus enhancing linear shrinkage [2]. These results however are in accordance with those of Vieira et al. [1] who used heavy clay mixed with red mud to produce ceramics but slightly contradicted the study of Abdeen and Shihada [2] who produced fired clay bricks using clay and waste glass, particularly at 40% waste glass addition where a negative shrinkage was obtained. The difference may be due to variation in the glassy phase, gaseous and fluxing components of red mud and clay.

From Table 3, it is also seen that water absorption

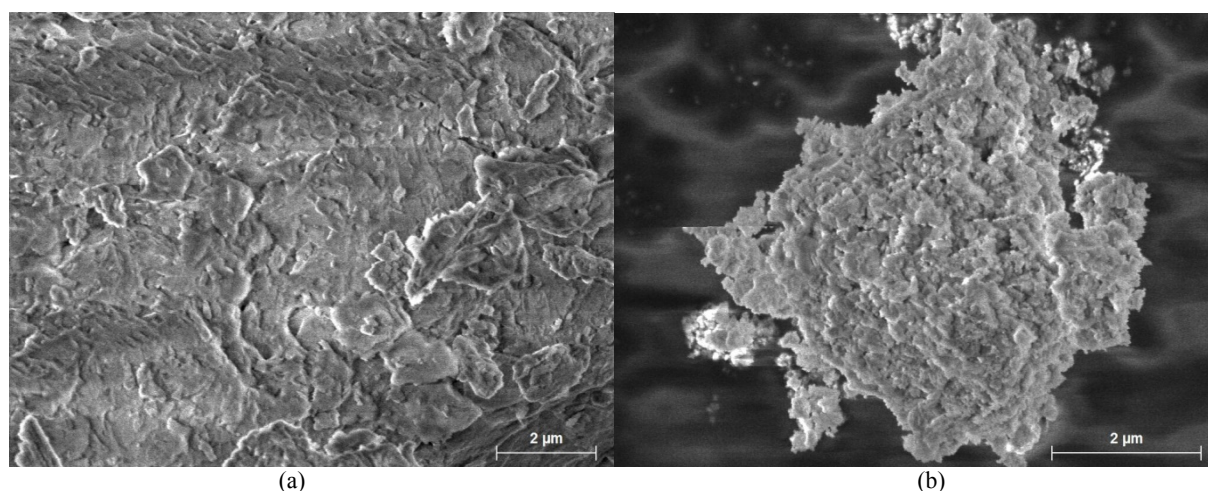


Fig. 5 SEM of (a) CRT glass (b) red mud, all of size 2 µm.

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Table 3 Properties of produced ceramics.

% CRT waste glass	Firing temperature range				
	700 °C	750 °C	850 °C	950 °C	
0	0.69	0.72	0.88	0.93	Linear shrinkage (%)
5	0.84	0.87	0.93	1.03	
10	0.89	0.93	0.99	1.21	
20	1.07	1.17	1.37	1.49	
30	1.18	1.26	1.41	1.51	
40	1.20	1.28	1.41	1.53	
0	19.01	18.28	17.94	14.52	Water absorption (%)
5	18.65	17.48	16.43	13.47	
10	17.30	16.07	15.13	13.02	
20	16.87	12.28	10.96	8.89	
30	17.09	11.78	9.83	8.28	
40	17.29	11.07	9.53	8.26	
0	37.01	36.96	35.61	34.42	Apparent porosity (%)
5	36.61	36.42	34.27	32.50	
10	35.10	33.10	30.34	27.20	
20	33.21	30.08	19.95	12.41	
30	30.47	28.49	19.42	14.44	
40	29.99	30.54	18.96	14.21	
0	1.73	1.73	1.75	1.76	Bulk density (g/cm ³)
5	1.73	1.73	1.77	1.79	
10	1.74	1.76	1.77	1.83	
20	1.78	1.78	1.81	1.83	
30	1.80	1.80	1.84	1.87	
40	1.89	1.87	1.85	1.86	
0	4.21	4.32	4.58	4.73	Flexural strength (MPa)
5	4.54	5.29	5.73	5.94	
10	4.86	5.96	6.12	6.54	
20	5.40	7.81	8.16	8.52	
30	5.68	8.37	8.82	8.98	
40	5.81	8.40	8.89	9.02	
0	4.11	4.96	5.18	5.26	Weight loss on ignition (%)
5	4.20	4.47	5.66	5.93	
10	4.58	4.69	5.92	6.23	
20	4.71	5.08	6.21	6.90	
30	4.80	5.82	6.75	7.10	
40	4.95	5.86	7.79	7.18	

and porosity decreases with increase in CRT glass content and firing temperature. According to Bordeepon et al. [21], water absorption is much related to densification and the amount of quartz influences the quantity of the amorphous phase after sintering. The increase in water absorption with increasing firing temperature is therefore, due to the greater densification of the sample resulting from the

formation of a liquid phase containing fluxing components principally from red mud and high quartz content of CRT glass. All the specimens produced had water absorption values below the maximum value of 20% specified by the norm to roofing tiles [1] while specimens with 30%-40% CRT glass and fired at 850 °C, and specimens with 20%-40% CRT glass and fired at 950 °C had water absorption values below 10%,

which is the norm for ceramic tiles [22]. For specimens 0%-40% CRT glass fired at 950 °C, specimens 5%-40% CRT glass fired at 850 °C, specimens 10%-40% CRT glass fired at 750 °C and specimens 20% CRT glass fired at 700 °C, water absorption values are all lower than 17% which is ASTM C62 specifications for bricks [2]. A greater densification was, however obtained at 20% CRT glass with the lowest porosity value of 12.41%. The durability of the ceramic material depends strongly on water absorption to avoid leaking of water [2]. Higher temperatures are thus necessary to increase density and decrease water absorption, the reason why the highest values were obtained at 950 °C.

At 0%-5% CRT glass amount, there is very little change in bulk density from 700-750 °C firing temperature, but higher values are observed for firing temperatures of 850 and 950 °C (Table 3). Bulk density increases with increasing firing temperature resulting from the consolidation or vitrification between particles. However, with 10%-40% CRT glass there is an increase in bulk density value for all the tested temperatures. Thus, the bulk density increases with increase in CRT glass quantity. The

highest value of bulk density is obtained for 40% CRT glass fired at 700 and 750 °C. According to Sultana et al. [22], the addition of more waste glass at relatively low temperature densifies the mixture, but higher temperatures, presumably bloat the mixture, decreasing the bulk density because the amount of CRT glass has increased.

The Flexural strength increases with increase in CRT glass and firing temperature, with the highest values (9.02 MPa) being observed in 40% CRT glass and 950 °C. Flexural strength is strongly dependent on porosity, the lower the porosity the higher the flexural strength values. As seen from Table 3, water absorption decreased with increase in CRT glass and firing temperature resulting in densification of particles in the samples. This results in a reduction in porosity due to vitrification hence, enhancing the mechanical strength of the samples [10]. These values of flexural strength obtained are all less than 25 MPa against values > 25 MPa required for floor tiles [23].

The weight loss on ignition of the specimens increased with increasing CRT glass and firing temperature (Table 3). However, the maximum value is obtained for 40% CRT glass specimen fired at 850 °C

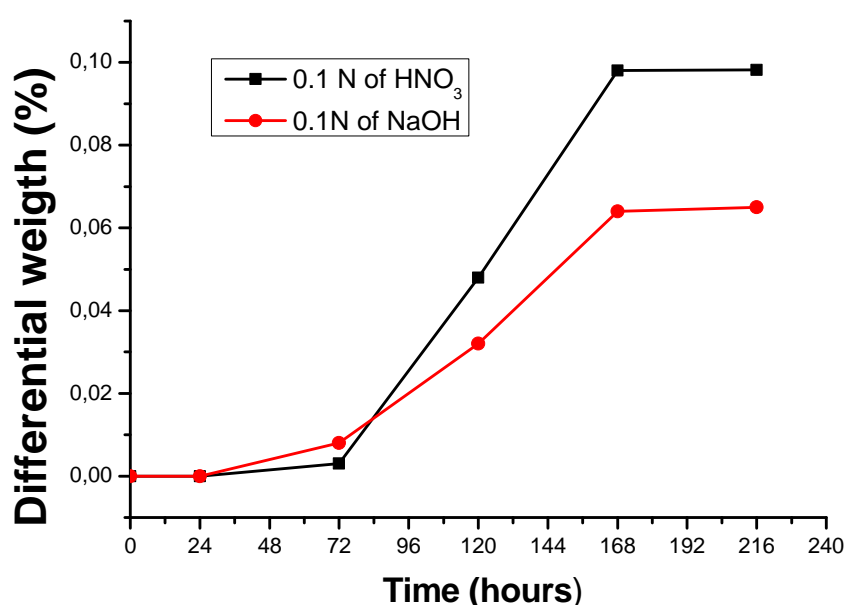


Fig. 6 Weight loss of specimen in nitric acid and sodium hydroxide solutions.

followed by a decrease at 950 °C, indicating that volatile components contributing to the loss on ignition had been removed or decomposed at temperatures below 950 °C [24].

The results of the chemical resistance test (performed for nine days) of the produced ceramics fired at 950 °C with 40% CRT glass are shown in Fig. 6. The specimen is slightly affected by both sodium hydroxide and nitric acid solutions, with HNO₃ showing a maximum loss of 0.10% against 0.06% for NaOH. However, equilibrium is established on the 7th day, probably due to the fact that few impurities on the specimens were all removed within this period. According to Kummoonin et al. [23], this loss or leaching can generally be solved by coating with either an engobe or ceramic glaze.

4. Conclusions

The preparation of ceramic materials using red mud, a waste product from the Bayer process and the waste CRT glass was investigated. The two raw materials have properties that make them good precursors for the production of ceramics with good properties as revealed from chemical composition, surface functional groups, mineral phases, different phase transitions (TGA) and morphology (SEM) determination. For example the glassy phases in CRT glass are 59.13% and 37.28% red mud while the fluxes in waste glass are 19.89% and 7.60% in red mud. The gaseous components are higher in red mud (47.47%) compared to 11.32% in waste CRT glass. Thus, the bloating properties of the ceramic were being greatly defined by red mud while the CRT glass was the major contributor in lowering down sintering conditions and main phases to form the framework inside and outside the ceramics. Increasing CRT glass content and firing temperature significantly improved all the properties of the ceramics evaluated. The produced material can be used as ceramics as well as roofing tiles but not as floor tiles. Exposure of the produced ceramic sample to nitric acid and sodium

hydroxide showed very little effect within the first seven days with acid having a greater effect than sodium hydroxide. The results of this study reveal that the industrial waste material, red mud and electronic waste material, CRT waste glass can be used to produce ceramic materials with good properties with a wide range of applications.

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