

# Effect of Temperature on Mechanical and Chemical Properties of Fired Ceramic Produced from Cathode-Ray Tube (CRT) Waste Glass and Red Mud Bauxite from Ngaoundal (Cameroon)

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**Abstract:** This paper report the characterisation and study of physico-chemical stability of ceramic floor tile. Ceramics obtained at different temperature (750, 850 and 950°C) were produced using a bauxite red mud of Ngaoundal and CRT waste glass mixture. Six samples were prepared by varying the mass of glass powder according to a percentage weight ranging from 0 to 40 %. The experiment carried out consisted of characterizing both the raw materials and the ceramics produced by chemical analysis, mineralogical analysis (X-ray diffraction, infrared spectroscopy, scanning electron microscopy, distribution size, thermal analysis, linear shrinkage, percentage of water absorbed and flexural strength. The results obtained showed that linear shrinkage slightly increases (0,7 to 1,5 %) as the mass of the glass powder added increases while the percentage of the absorbed water decreases (18,5 to 10,8 %) leading to a decrease in porosity. Flexural strength increases from 4 to 10,8 MPa when the mass percentage of the additive and temperature increases, this being related to the density of the product due to the formation of a vitreous phase brought by the additive. As a result, addition of glass powder into the red mud bauxite studied allowed the production of ceramics with ameliorated properties.

**Key words:** Red mud bauxite, CRT waste glass, firing temperature, mechanical and chemical properties.

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## 1. Introduction

Red mud is the largest amount of industrial waste generated during the production of alumina. Depending on the amount of bauxite and the type of process, during the production of each 1 tons alumina 1.2–1.3 tons dry red mud is generated [1]. Valorization of industrial wastes and by-products has emerged as an important environmental issue since industrial development generates tonnes of hazardous wastes which have high content of toxic elements, including heavy metals and metalloids. Today, the need for the development of efficient management approaches focusing on reduction of the volume of wastes, immobilization of hazardous elements and energy savings is of high priority [2]. The enormous quantity of 'red mud' discharged by industries producing alumina from bauxite represents an environmental and economical problem. Numerous reports proposing reuses of 'red mud' have been advanced, especially for the production of

ceramic bodies or cements [3], [4]. With the development of technology in the electronics sector the demand for consumer electrical and electronic equipment (EEE) has been on the increase since 1980s and countless number of EEEs, especially computers and televisions have been sold to consumers around the world [5]. The cathode ray tube of TV or PC monitor is composed of 85% CRT glass, which is possible to be separate and cleaned by using different suitable techniques. Recycling techniques for metals, plastics, and the other electronic components already exist, while the utilization of end of life (EOL) CRT glass is quite problematic [6], [7]. However, CRT waste glass is abundant and available especially in urban zones as consequence of discharged E-waste in the environment. Although these E-waste are available, in many African countries and particularly in Cameroon, there are not always specialized industries in charge of collecting, storing and reusing waste materials, despite the fact that it can provide added economic benefits if CRT waste glass are able to be converted to useful materials and recycled [8], [9]. Thus, this can make CRT waste glass potential secondary input for traditional ceramics production. Wasted glasses are recycled into new glass items to reduce the release of atmospheric carbon dioxide. Powders of wasted glasses can replace a ceramic component, acting as a flux in silicate compositions instead of feldspar or other mineral fluxes, to save energy in manufacturing processes [6], [10]. Red mud and waste glass generated have mostly the same element and oxide present in clay. Oxides contain in red mud and waste glass require low temperature to melt and act as binders which link particles during the sintering process. The role of such addition was extensively experimented in porcelain, in stoneware, in earthenware, and in clay ceramics [11], [12]. The present study focused on red mud characterization in term of chemical composition using X-ray fluorescence spectrometry, structure using X-ray diffraction (XRD), Infra-red spectroscopy (IR), microstructure or morphology were analyzed using scanning electron microscopy coupled to (SEM/EDS). In addition, some technological properties such as the linear shrinkage, flexural strength, water absorption and chemical analysis were performed in fired product.

## 2. Materials and Experimental Methods

### 2.1. Bauxite Potentials of Study Area

It is estimated that Cameroon has the 6<sup>th</sup> world bauxite reserves. With approximately 1.8 billion tons from which 1 billion tons are estimated for the two groups of deposits situated in the Minim- Martap and Ngaoundal. These 1.8 billion tons seems to be underestimated, since there are many non-explored bauxite indices in Cameroon as indicated by the SABAP exploration license [13]-[15] and the recent geological map of Cameroon.



Fig. 1. Bauxite and sampling area.

### 2.2. Raw Materials Preparation

#### 2.2.1. Preparation of Sample

Fig. 1 shows the sample area where bauxite has been obtained. Red mud used for this study was produced in our laboratory from bauxite collected at Mount Ngaoundal (6° 27' 55" Nord, 13° 16' 16" Est). The method used to extract red mud from bauxite was that employed by Benhamou *et al.*, 2008 [13], [16] adapted from the

Bayer process. Where in 20 g of finely crushed and grind bauxite (powder particles of diameter < 75 $\mu$ m) were mix with 40 ml solution of 3M NaOH in an Erlenmeyer flask and stirred (300tr/min) for 10 minutes. The reaction mixture was then, heated at 80°C for 20 minutes, allowed to cool down and filtered. Red mud was collected on the filter paper while the filtrate was collected in a Becker. Red mud obtained was dried at 110°C overnight and used for characterization without further treatment.

### **2.2.2. Characterization Techniques**

The chemical composition of the red mud was analyzed using a wavelength dispersive X-ray fluorescence apparatus (Shimadzu, XRF-1800). In addition, X-ray diffraction (DRX) analysis was performed to evidence the mineralogical category of the clay using a Philips X'Pert PRO diffractometer equipped with a Ni-filtered Cu-K $\alpha$  ( $\lambda=1.542$  Å) radiation. The apparatus operated at 40 kV and 100 mA in a step scan mode. Diffraction patterns were collected at a scanning speed of 0.01671s<sup>-1</sup>. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analyses were performed using a 2960 TA Instrument under argon from room temperature up to 1000°C at a heating rate of 5°C/min. The microstructures of the red mud were analyzed by scanning electron microscopy (SEM) (Hitachi S-4500). Infrared spectroscopy (IR) and Ramann Spectra (Horiba Jobin-Yvon LabRam HR spectrometer coupled) was used to identify some surface groups on red mud.

### **2.2.3. Formulation, Shaping and Mechanical Properties**

Six mixtures of raw materials denoted respectively RM<sub>100</sub>WG<sub>0</sub>, RM<sub>95</sub>WG<sub>5</sub>, RM<sub>90</sub>WG<sub>10</sub>, RM<sub>80</sub>WG<sub>20</sub>, RM<sub>70</sub>WG<sub>30</sub>, and RM<sub>60</sub>WG<sub>40</sub>, labelled RM<sub>x</sub>WG<sub>y</sub>, (with x the % of red mud and y % of waste glass) were prepared. The mixture was completely oven-dried at 110°C for 24h then crushed and sieved using a 75  $\mu$ m sieve. For each type of mixture, samples were produced by extrusion in parallelepiped (82 mm  $\times$  42 mm  $\times$  9 mm) using a uniaxial pressing techniques at 10bars. [8], [17] After forming the ceramic body specimens were dried for 48 h at ambient temperature of laboratory, oven-dried at 110°C for 24h then fired in a kiln (Nabertherm, model LH 60/40) at different temperature 750°C, 850°C and 950°C. For firing temperature up to 500°C the heating rate was set to 2.5°C/min. Once the desired temperature was reached, the sample was left at constant temperature for 2hours and then heated at a rate of 5°C/min up to the selected firing temperature where it was left for 2hours before cooling down to room temperature. The firing temperature of 750°C was chosen as a result of preliminary test which showed that the used CRT waste glass powder started to melt around 680°C. Linear shrinkage was determined on parallelepiped fired test specimens thanks to a caliper (ROCH France, Patented S.G.D.G.) and water absorption was carried out on cylindrical fired test specimens using NF-P-18-554 standard [18]. Flexural strength was performed according to EN-100 standard [19] on parallelepiped fired test specimens using an electro-hydraulic press (M & O, type 11.50, and No 21) operating at an average rate of 3 mm/min. The flexural strength was calculated from the breaking load using the relation  $FS = (3PL) / (2bd^2)$  where P is maximum load, L is the distance between the supports of the bean, b is the fired average width and d is the fired average depth.

### **2.2.4. Chemical Corrosion Studies**

Acidic HNO<sub>3</sub> leaching solution is more corrosive than water alkaline NaOH solution. The increase of additional quantities of waste glass generally better the chemical durability of the products. The sintered crucible was transferred to an air oven at 110°C for 24 hour. The total weight loss % from the grains was calculated. This procedure of corrosion study was recommended by ASTM specification No. C-22573, ISO.719-1985 and by different authors [20], [21].

## **3. Results and Discussion**

### **3.1. Characterization of Red Mud and CRT Waste Glass**

#### **3.1.1. Chemical Composition of the Powders**

CRT glass used in this study was collected from crushing and grinding process in a CRT monitor treatment facility in Ngaounderé (Cameroon), where the particle size was under 75 $\mu$ m. The chemical composition of glass determined by X-Ray Fluorescent (XRF) analysis is shown in Table 1. We noticed that CRT glass contains about 18 wt% PbO. The powder contains a high amount of silica (56.11%) and a little quantity of CaO (2.30%) along with Na<sub>2</sub>O (5.46%). Considerable amount of Na<sub>2</sub>O + K<sub>2</sub>O (15.47%) and CaO + MgO (4.42%) could act as energetic fluxing agent when added with the clay powder to get mixtures enabling to make associated products [8], [9], [17], [23]. According to this, waste glass material can constitute a potential fluxing agent that can substitute feldspar for manufacturing ceramics. The main reason for using waste glass here is to reduce the consumption of energy for economic purposes [8], [9], [17]. In addition, waste glass replaces completely feldspar since the latter is expensive and not available everywhere for effective economic consumption while the former is easily found free especially in certain urban zones [7], [22]. Dried red mud was substantially inert up to 900°C, the loss of H<sub>2</sub>O from aluminium hydroxides and of CO<sub>2</sub> from silico-alumino-carbonates being the only detectable effects [3]. In red mud the Fe<sub>2</sub>O<sub>3</sub> mass percentage of 29.21 is high and this is not favorable to allow for ceramics with high mechanical values [7], [8], [23], [24]. Conversely, this amount of Fe<sub>2</sub>O<sub>3</sub> in presence of uncolored waste glass is beneficial to get red colored ceramics [7], [8], [25], [26]. At higher temperatures major components such as Fe<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> reacted to give Fe<sub>2</sub>TiO<sub>4</sub> with O<sub>2</sub> evolution. This reaction was responsible for the intensification of the brown colour above 1200°C [3], [27], [28]. This technological aspect is of great importance because it makes possible the use of red mud so as to produce ceramics with red tonality, mostly for roofing and rustic floor tiles. Iron oxide accounts for the bodies red pigmentation after firing [8]. Due to the great amount of Fe<sub>2</sub>O<sub>3</sub> in the red mud and presence of low content of Na<sub>2</sub>O + K<sub>2</sub>O (4.77% mass) and CaO+ MgO (2.83% mass), oxides which act as fluxes at temperatures greater than 1000°C, the sintering of this red mud could require high temperature to get reliable ceramics [17]. Hence using the red mud for ceramic production might need adjustment of its chemical composition [9]. This can be possible through addition of energetic fluxing agents such as sodium or potassium feldspars or waste glass [9]. Loss on ignition is 20.21% and this is not different from values commonly met for red mud finding by [29] (17.62%). 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 18.34% PbO content constitute the major challenge of CRT glass usage.

Table 1. Chemical Composition of the CRT Waste Glass and Red Mud

sample	LOI	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	PbO	TiO <sub>2</sub>
<b>Waste glass</b>	11.23	56.11	3.02	0.09	2.56	1.86	0.28	5.46	10.01	0.04	18.34	0,09
<b>Red mud</b>	20.26	7.68	29.6	27.21	2.75	0.08	0.08	4.71	0.06	0.12	-	1,89

### 3.1.2. Infra-Red Spectra

Fig. 2 shows the IR spectra of red mud. The absorption bands at 3653 ; 3735 ; 3648 ; 3335 cm<sup>-1</sup> vibrations of express the stretching OH groups gibbsite base mineral of kaolinite network [8], [30]. This result can be confirmed by XRD and ATG/DSC spectra. The bands located at 1060cm<sup>-1</sup>correspond respectively to stretching vibrations of OH of water molecules while those at 966 and 913 cm<sup>-1</sup> express the vibration of Si-O-Al group of the network. The bands at 973 and at 795 cm<sup>-1</sup> indicate the stretching vibration of Al-OH with Al in VI coordination. The band at 600cm<sup>-1</sup> indicates the vibration of Si-O-Si and Si-O-Al groups of the network [8], [31], [32]. This play an important role in the formation of aluminosilicate network formed with silicon and aluminum atoms, tetrahedrally surrounded by oxygen atoms [34]. The absorption bands around 1558cm<sup>-1</sup>,

is due to the stretching vibrations of C=O, indicating the presence of carbone dioxide and S-O, showing the presence of SO<sub>3</sub>. Here the change in the dipole moment of the water hydroxyl-stretching plays an important role and so the intensity of the infrared spectrum is more. Stretching vibrations of C=O is found at 1457cm<sup>-1</sup> confirms the presence of carbonate groups .The main reason being the presence of chemisorbed CO<sub>2</sub> in Red Mud. The weak Peaks around 850 cm<sup>-1</sup> respectively are Si-O vibrations [29], while peaks at 799 cm<sup>-1</sup> is due to Fe-O-Se stretching. This IR data confirms the results of XRD and XRF. This is in agreement with the values found in the literature [29], Fig. 3 shows the IR spectra of waste glass. The bands respectively at 910 cm<sup>-1</sup> is fingerprint of quartz [8], [31], [33] while the band at 1400-1500cm<sup>-1</sup> is assigned to vibration of O-C-O. Also the band at 732-798 cm<sup>-1</sup> expresses the absorption of quartz inferring marks of crystalline silica within the great amount of amorphous phase of the soda-lime waste glass [8]. This is evidence of high amount content of silica as shown in the chemical composition of waste glass. The XRD pattern of waste glass is shown in Fig. 4 and exhibits mainly a large dome between 20° and 40° (2θ) which expresses great amorphous silica content [21] of waste glass. The wide absorption band around 1295cm<sup>-1</sup> is characteristic of amorphous silica in this material [30]. Absorption bands ranging from 732 to 798cm<sup>-1</sup> indicate the presence of quartz traces in the glass powder [30]. Absorption band around 3648; 3735; 3938 are attributed to Si-O-Si and O-Si-O bond deformation vibrations [31]. The band around 2359 cm<sup>-1</sup> is attributed to the deformation vibration of the O-C-O bond indicating the presence of sodiun and potassium in the glass powder as bicarbonate. Indeed, sodiun oxide and potassium oxide are contained in the glass powder. Indeed the sodiun oxide contained in the glass in large quantity, reacts with the CO<sub>2</sub> of the air to give sodiun bicarbonate [32].

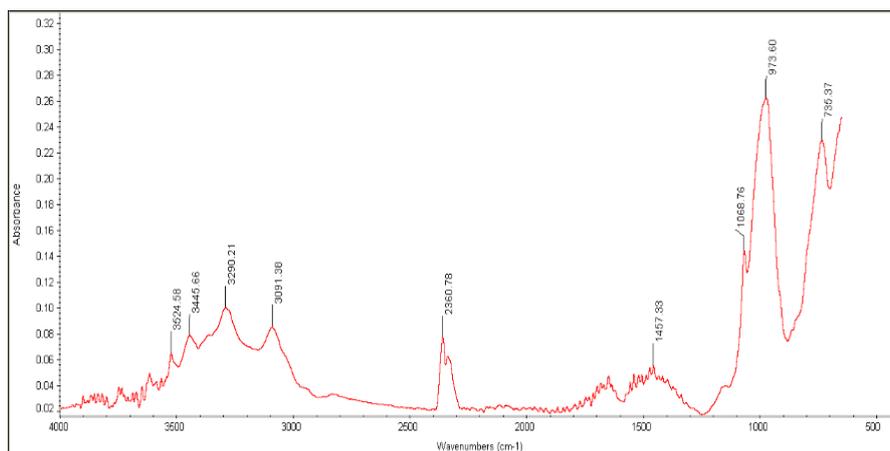


Fig. 2. IR curve of Ngaoundal red mud.

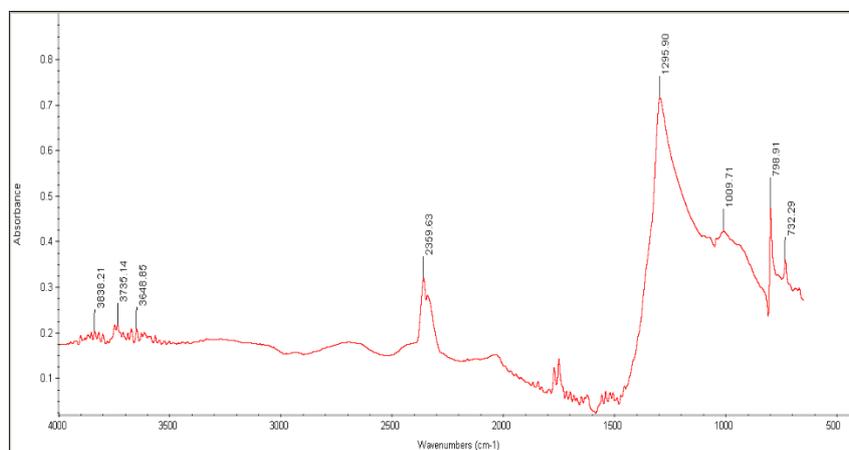


Fig. 3. IR curve of CRT waste glass.



The TGA (Thermal Gravimetric Analysis) diagram of the red mud sample shows three steps for the weight loss. The 1<sup>st</sup> one occurred in the range of 25–225°C (weight loss about 0.99% of the total weight equivalent to 0.212mg), corresponding to the evaporation of physically adsorbed water [3], [35]. The 2<sup>nd</sup> and 3<sup>rd</sup> ones occurred in the ranges of 225–380°C (weight loss about 10.70% of the total weight equivalent to 2.3mg), and 380–680°C respectively corresponding to total weight loss of about 5.9% equivalent of 1.253mg, attributed to the loss of chemically adsorbed water [36], [37]. The organic matter composing the paste is totally degraded, without. There are only two endothermic peaks, at 282.99°C and 490.67°C, corresponds to the loss of structural hydroxyl groups and is due to the transformation of kaolinite to metakaolinite according to the following reaction. On the DSC (Differential scanning calorimetry) curve. This is associated with the dehydration of gibbsite to form  $\alpha$ -alumina [38]. These results confirm that red mud is hermally stable up to about 680°C.

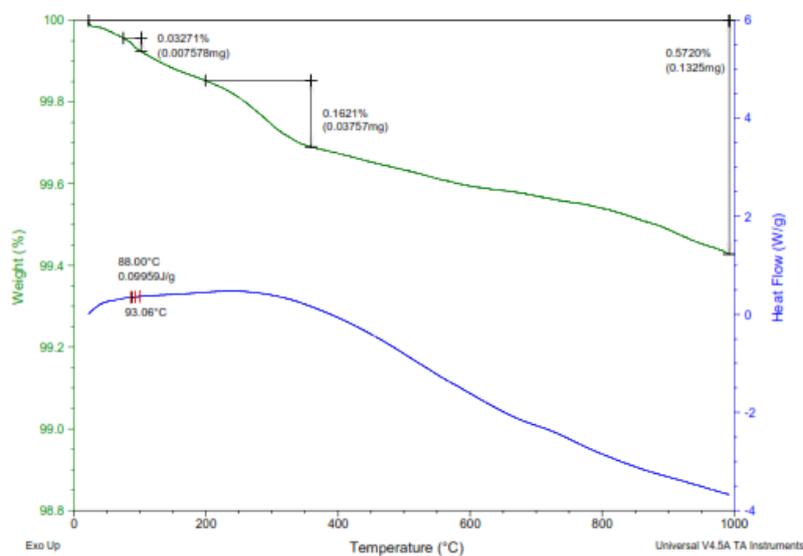


Fig. 7. DSC-TGA curves of CRT Waste glass (blue is DSC, green is TGA).

An important endothermic peak, the maximum of which is around 283°C, corresponds to the dehydroxylation of gibbsite [35] according to equation 1:



According to the study conducted by [38], the slight endothermic hook observed at 380°C corresponds to the second stage of dehydroxylation of gibbsite to boehmite. Note also that this dehydroxylation ends before 400°C as observed [38]. According to Kloprogge *et al.*, the maximum dehydroxylation temperature of gibbsite is around 300-310°C but for well crystallized gibbsites this range is 350-380°C. Thus the gibbsite contained in our material is well crystallized.

An endothermic peak around 491°C that corresponds to the dehydroxylation of boehmite [36]-[38] according to Equation 2:



The thermogravimetric curve of red mud has two thermal accidents:

The first starts around 200°C, the maximum is at 283°C and ends at 390°C. The phenomenon revealed

corresponds to the dehydroxylation of gibbsite in boehmite [33], [34]. This accident is accompanied by a mass loss of about 17.52%;

The second, very low accident is at 491°C as in the DSC curve. It corresponds to the dehydroxylation of boehmite [35], [36]. This accident is accompanied by a loss of mass of about 5.82%.

The TGA diagram shows a continuous weight loss distributed in the range of 50–1000°C. The figure shows three main portions of mass loss as the rise of temperature. The first one is during the heating temperature interval of 50–1000°C when the physically absorbed water and chemically bound water is off. Before the firing temperature is up to 400°C, the sample loses 0.04507 mg who is equivalent of 0.19% total of its weight. At the end of the process, we have 0.57% of weight loss equivalent of 0.1325mg. this result show that we have poorly loss of material du ring firing temperature. For the DSC thermogram, there is a peak at 93°C corresponding to the loss of water in the glass powder. A large exothermic peak has been observed at 93°C, which can be associated with crystallization of SiO<sub>2</sub>. These thermal analyses were originally of the sintering program applied in this study. The effect of K<sub>2</sub>O, Na<sub>2</sub>O, MgO and CaO can be seen when the raw material started to have a reaction at around 700–900°C.

### 3.1.5. Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX)

Scanning Electron Microscopy and Energy Dispersive X-Ray of sample red mud is shown in Fig. 8. It is observed that the arrangement of the particles is relatively loose with high porosity and small particle size. The main elements observed are Fe, O, Ti, Al, Si and Na. The sample show more basic nature (whiter colouration) indicating higher surface area of contact with the base during extraction. This highly white colouration is expected as bauxite is digested in concentrated sodium hydroxide. The EDX analysis of the Red Med sample ensures the presence of particular elements in it and is shown in Fig. 8. This element is confirmed by XRF, XRD and IRTF.

The SEM/EDX images of waste glass sample is shown in Fig. 8. It is observed that the arrangement of the particles is relatively loose with high porosity and small particle size. The EDX analysis of the waste glass sample ensures the presence of particular elements in it and is shown in Fig. 8. The main elements observed are Fe, O, Ti, Al and Si.

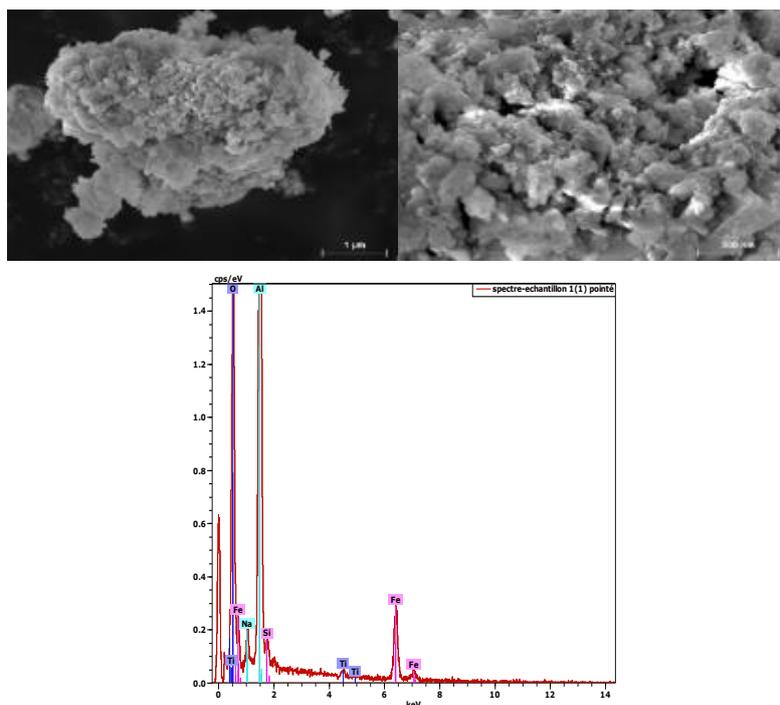


Fig. 8. Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) of red mud.

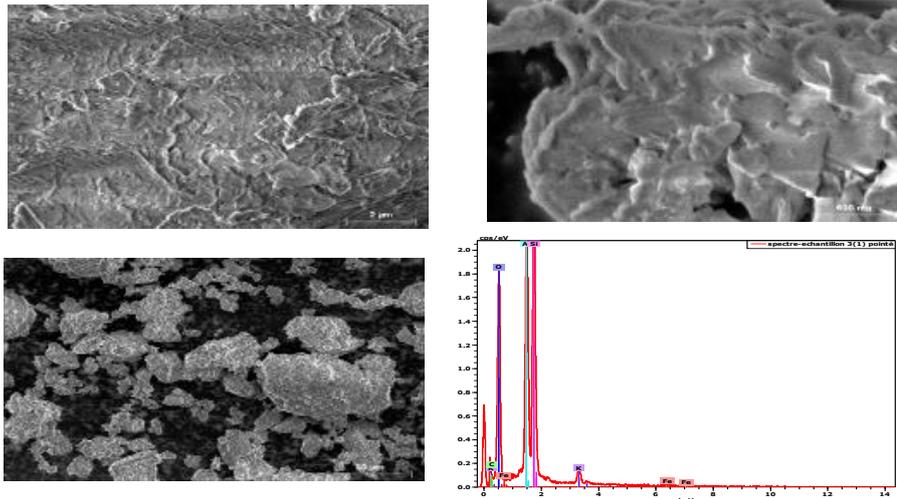


Fig. 9. Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) of CRT waste glass.

The SEM/EDX images of waste glass sample is shown in Fig. 8. It is observed that the arrangement of the particles is relatively loose with high porosity and small particle size. The EDX analysis of the waste glass sample ensures the presence of particular elements in it and is shown in Fig. 8. The main elements observed are Fe, O, Ti, Al and Si. Microscopy (SEM) of waste CRT glass and red mud 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.

1) Particle size Crt waste glass

Table 2. Particle Size Distribution of Crt Waste Glass

Specific surface area (m <sup>2</sup> /g)	D <sub>10</sub> (μm)	D <sub>50</sub> (μm)	D <sub>90</sub> (μm)
1.3	1.724	12.012	62.136

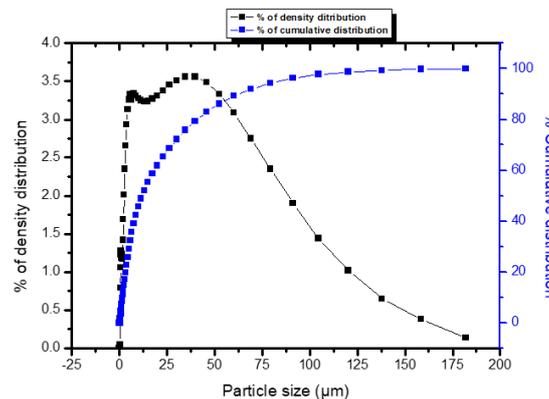


Fig. 10. Particle size of CRT Waste glass.

2) Particle size of Red mud

The particle size distribution also has a great importance on the compactness and the porosity of the dry product. If the dry product is very porous, water exchange is facilitated as is the passage of gas during cooking Ramadan *et al.*, (2001). A tight granulometry does not allow to obtain a high compactness. The computerized processing of the results made it possible to obtain the distribution of volcanic slag particles according to their occupied volumes. The curves obtained (Fig. 9, Fig. 10) show respectively the volumes occupied by each class of particles (black curve) and the cumulative volumes (curve in blue) of glass powder, red mud and

andosol. Using the blue curve, we evaluated the maximum diameter corresponding to 10% (d10), 50% (d50) and 90% (d90) of the cumulative particle volume.

Table 3. Particle Size Distribution of Bauxite Red Mud

Specific surface area (m <sup>2</sup> /g)	D <sub>10</sub> (μm)	D <sub>50</sub> (μm)	D <sub>90</sub> (μm)
2.02	0.775	19.250	84.226

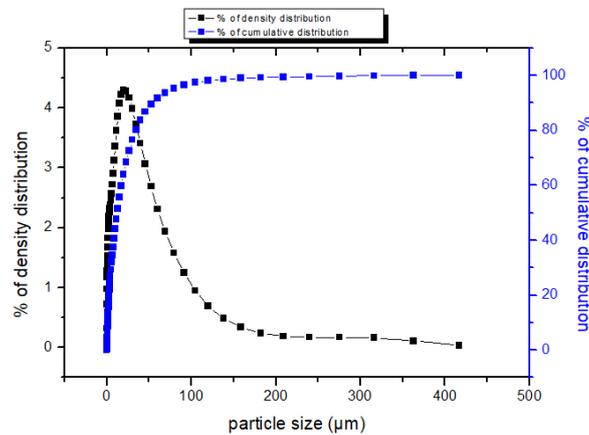


Fig. 11. Particle size of bauxite red mud.

### 3.2. Physical and Mechanical Properties of the fired Specimens

Water absorption, density and firing shrinkage are important properties of ceramic products. They have a great important and they are under very close control in the industry. Water absorption level could be regarded as a significant indication of the level of vitrification/densification and the lower the water absorption the lower the product open porosity. Density also is important to have information about the extent of sintering/densification. In the production of standard size ceramic articles, it is not desired to have a high firing shrinkage which sometimes may depend on the loss on ignition (LOI) of the unfired green structure [17], [39]-[41].

#### 3.2.1. Water Absorption

Water absorption was measured by Archimedes Method for all sintered pellets. The water absorption and bulk density of the fired pellets can be determined by this method. The lowest water absorption was determined for the 10% waste glass added pellets which were fired at different temperature. The water absorption decreases with increased waste glass content and firing temperature in the 750-950°C range as shown in Fig. 11. This behavior may be related with the calcination of the increasing amounts of calcite with the increase in the sludge addition percent. Water absorption and porosity of fired product decrease with increasing temperature and waste glass percentage. This is a natural behavior of a ceramic body [42]-[44]. With increase in firing temperature, values for water absorption tend to decrease since a greater densification of the sample occurred. The water absorption of the samples ranged from 9.4% to 18%. The water absorption of sample fired at 950°C is below 10%, which is compatible with the range specified for ceramic tiles this is confirmed by Shanjida S. *et al.*, [42].

#### 3.2.2. Linear Shrinkage

Highly porous ceramic tiles samples are mechanically weak and less porosity has the good flexural strength. The linear shrinkage is a translation of the reactivity of the material [9], [17], [37], [39]. The use of waste glass content gave way to abundant glassy phase development. The quantitative presence of this phase played a key role in the sintering process. As a matter of fact, as the viscous liquid became abundant, the firing

shrinkage increased and the water absorption decreased. The increase of waste glass content resulted in the consumption of cristobalite, a partial dissolution of quartz and development of mullite. The increase of waste glass from 0 to 40% increased firing shrinkage from 0.72 to 1.28% at 750°C, 0.88 to 1.41% at 850°C and 0.93 to 1.53% at 950°C. Linear Shrinkage of fired product was lowest in the control samples (0%) at the given temperatures. According to water absorption and linear shrinkage, we notice that the lower shrinkage is due to poor densification caused by poor vitrification in accordance with the lower values of flexural strength (Fig. 13) for the flux free samples. High shrinkage cause destruction of ceramic and low linear shrinkage is factor that may contribute to reduce the risk of appearance of cracks and dimensional defects in ceramic samples [38]. Normally a good quality exhibits shrinkage below 8%. Result show that there is linear increase in the linear shrinkage as the amount of waste glass and temperature increases [38]. The water absorption (Fig. 11) is in accordance with the firing shrinkage through the overall decrease of the water absorption with the increase of the waste glass content. In the formulations with high flux content, increased vitrification is noticeable with lower water absorption due to the coalescence and the formation of closed porosity [39]. The increase of flux content from 0 to 40% decreased water absorption from 18.28 to 11.07% at 750°C, 17.94 to 11.03% at 850°C and from 17.52 to 10.26% at 950°C. Firing shrinkage is one of the important criteria representing the quality of the specimen and a lower value of firing shrinkage is always desired to the researchers. All this result from the graph shows that mostly shrinkage increased with increasing temperature and waste glass percentages. This result is in accordance with Tiffo *et al.*, [8], Djangang *et al.*, [9]. It may be suggested that a high content of fluxing oxides in the system helps the formation of glassy phases which fill in the pores, thus leading to a shrinkage gain that decreases on firing [42], [43].

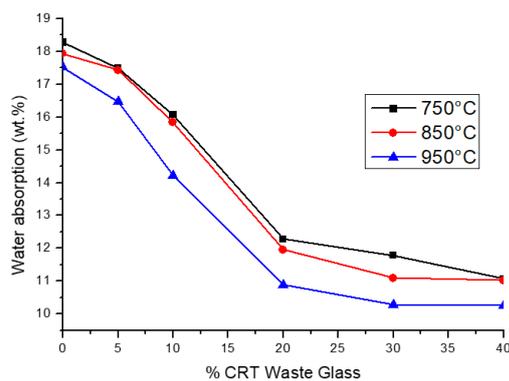


Fig. 12. Water absorption versus waste glass content (750°C, 850°C and 950°C).

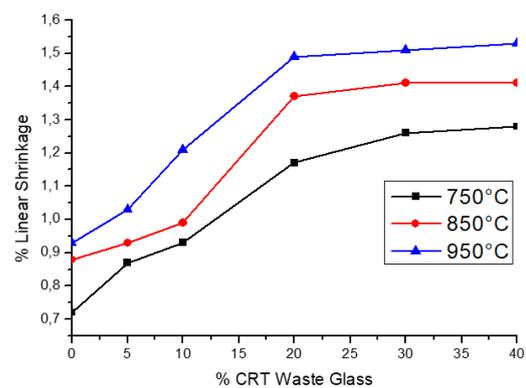


Fig. 13. Linear shrinkage versus waste glass content (750°C, 850°C and 950°C).

### 3.2.3. Flexural Strength (MPa) and Apparent Porosity

The flexural strength of fired product was lowest in the control samples (0%) at the given temperatures. The flexural strength (Fig. 12) is greater with the increase of waste glass content and this observation is in accordance with the increased densification of the samples through the formation of a glassy phase. The increase of flexural strength is also related to the presence of minerals such as mullite and cristobalite. The increase of waste glass content from 0 to 40% increased flexural strength from 4.2 to 8.40% at 750°C, 4.58 to 8.89% at 850°C and from 4.73 to 9.02% at 950°C. Therefore water absorption is directly related to apparent porosity, its value decreases in the overall temperature range with increase amount waste glass.

Generally as the porosity increases, the strength properties decrease in traditional ceramic systems. According to Flexural strength and apparent density, we notice that the higher flexural strength is due to better densification caused by good vitrification in accordance with the lower values of water absorption Fig. 11 and linear shrinkage Fig. 12 for the flux content. The apparent density of the samples fired at 750°C, 850

and 950°C decrease with the increase in temperature and the amount of waste glass adding. Increase the waste glass percentage up to 0-20% increase considerably the value of apparent porosity. When the waste glass is between 30-40%, we have an optimum in the percentage of apparent porosity in fired product. We can conclude that the best percentage of incorporation of waste glass particle is 20% and temperature 950°C is better for sintering product. Furthermore, this result is in accordance with work of Shanjida S. *et al.*, [42]. The apparent porosity versus temperature curves shows a similar trend to that of water absorption. With increase in firing temperature, values for water absorption and apparent porosity tended to decrease since greater densification of the sample occurred. No significant variation is observed in bulk density values.

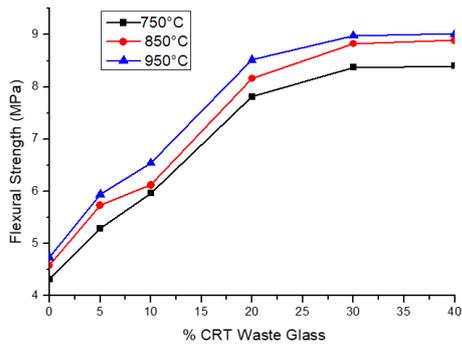


Fig. 14. Flexural strength versus waste glass content (750°C, 850°C and 950°C).

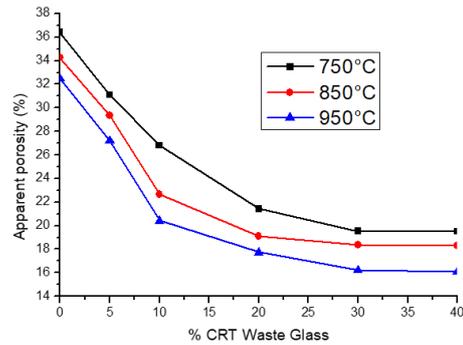


Fig. 15. Apparent porosity versus waste glass content (750°C, 850°C and 950°C).

### 3.2.4. Bulk Density (g/cm<sup>3</sup>) and Apparent Density (g/cm<sup>3</sup>)

The bulk density of all fired product had no significant influence on either the amount of waste glass added to the mixture and the chosen firing temperatures (Fig. 15). The apparent density of the samples fired at 850 and 950°C was lower than that of samples fired at 750°C. This was because the samples fired at 850 and 950°C had much more glassy phase than that product at 750°C, since this is intrinsically related to the total closed pore volume of the samples (Fig. 16) [40], [41]. However, B. Touogam *et al.*, [45] show that the increase in density is closely correlated to the linear shrinkage. An increase in density generally results in an increase of linear shrinkage and a decrease in the water absorption [9]. This shows that the vitrification of the sample is higher RM<sub>70</sub>WG<sub>30</sub> than others. However, these values are in the range of ceramic tile. They not exceed 15%. In this temperature range, the melt process ability of crystalline substances is not sufficient [46], [47].

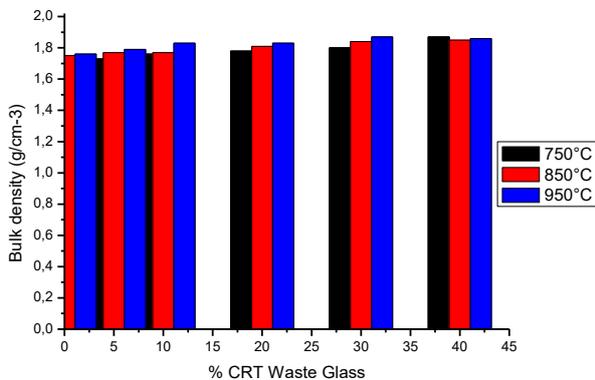


Fig. 16. bulk density versus waste glass content (750°C, 850°C and 950°C).

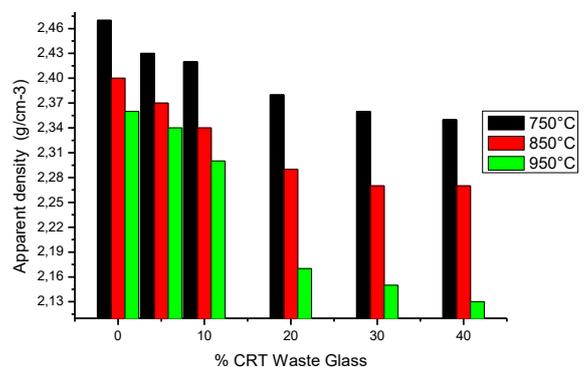


Fig. 17. Apparent density versus waste glass content (750°C, 850°C and 950°C).

The increase in density is closely correlated to the linear shrinkage. An increase in density generally results in an increase of linear shrinkage and a decrease in the water absorption [43].

### 3.2.5. Chemical Corrosion Studies

The chemical resistance test reveal the following characteristics: We notice that adding 20% of waste glass particle is the best percentage of incorporation. The chemical durabilities of the incorporated waste glass derivatives are generally better than their original samples. In order to determine its corrosion resistance, the support sintered at 950°C , was soaked at 50 °C using nitric acid (pH = 3) and soda solutions (pH = 12). And the weight of the support was measured each day during 7 day. The results reported in Fig. 17 show that formulation (red mud fired+20% waste glass) fired at 950°C supports presents a good chemical resistance in acidic medium and in basic medium. For 7 days of treatment, the weight loss does not exceed 0.1 wt. % in two solutions. This is in the range of Bélibi *et al.* [47], [48].

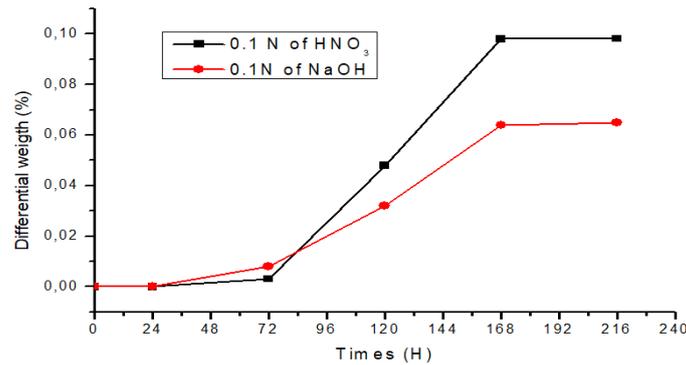
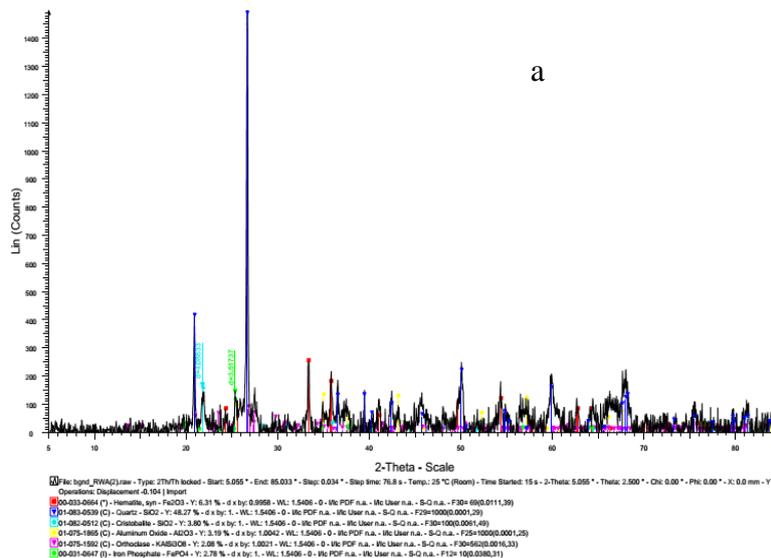


Fig. 18. Weight loss of support in nitric acid and soda solutions as a function of time.

### 3.2.6. Mineralogical Composition and Micrograph of Fired Body

The XRD pattern shown in Fig. 18(a) and Fig. 18(b) establishes that the reference tiles fired at 950°C contains six predominant crystalline phases, quartz (SiO<sub>2</sub>) 48.27%, orthoclase (KAlSi<sub>3</sub>O<sub>8</sub>) 2.08%, hematite (Fe<sub>2</sub>O<sub>3</sub>) 6.38%, and cristobalite (SiO<sub>2</sub>) 3.82%, Aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) 3.19% and iron phosphate FePO<sub>4</sub> (2.78%). Meanwhile, the XRD pattern of the formulation contains predominant crystalline phases, quartz (SiO<sub>2</sub>) 77.23%, orthoclase (KAlSi<sub>3</sub>O<sub>8</sub>) 6.28%, hematite (Fe<sub>2</sub>O<sub>3</sub>) 28.86%, and cristobalite (SiO<sub>2</sub>) 6.52%, Aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) 4.78% and iron phosphate FePO<sub>4</sub> (8.23%) as the major constituents included in the amorphous phase. The XRD of patterns of these tiles differ slightly from each other. The aluminium oxide, cristobalite, orthoclase, and quartz crystallines are the factors that contribute to the increase of the flexural strength and low porosity of the fired floor tile.



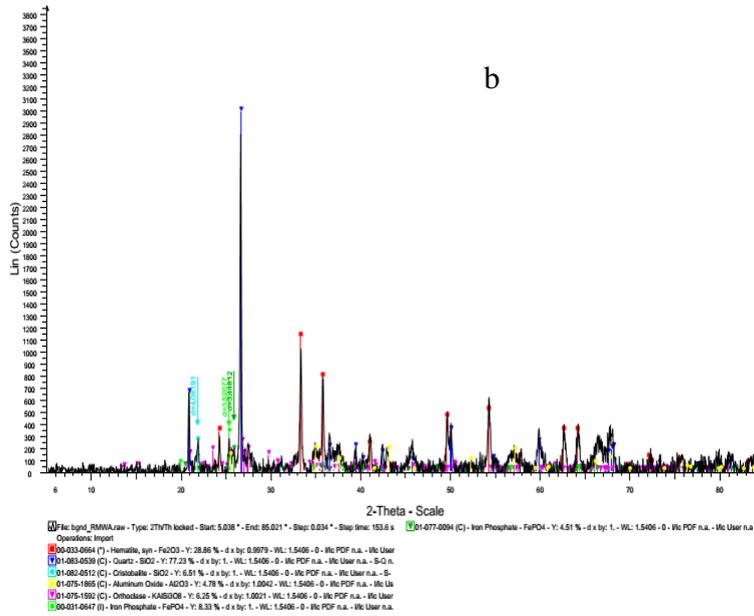


Fig. 19. XRD patterns of the fired product: (a) RM<sub>80</sub>WG<sub>20</sub>, tile formulation b) the formulation RM<sub>70</sub>WG<sub>30</sub> tile.

The microstructure of fired product shows a closely dense structure relating to the low water absorption, low porosity and the high flexural strength. The chemical composition of fired product is given by the EDS composition in table show that the PbO<sub>2</sub> present in waste glass are not available in fired product. This is very interesting because the leaching of this heavy metal is not good for the environment.

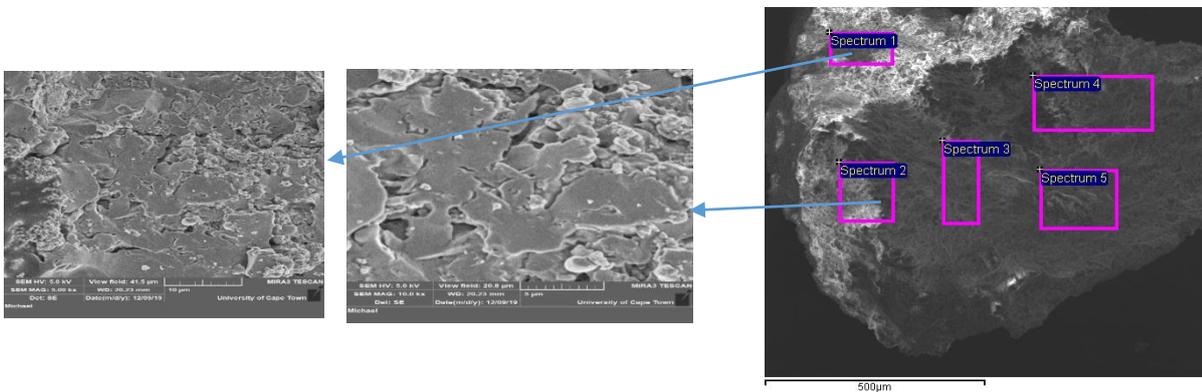


Fig. 20. SEM micrograph of specimens containing.

Table 4 gives the elementary composition of the spectrum of the formulation having the best mechanical properties.

Table 4. Elementary of EDS Analysis

Spectrum	C	O	Na	Al	Si	K	Ti	Fe	Total
Spectrum 1	18.31	55.53	1.16	8.81	12.94	1.12	0.52	1.6	100
Spectrum 2	8.97	55.84	1.21	11.5	16.63	1.85	0.84	3.18	100
Spectrum 3	9.16	57.88	1.47	10.15	17.2	1.42	0.54	2.18	100
Spectrum 4	5.28	56.64	1.45	11.91	19.04	1.77	0.7	3.21	100
Spectrum 5	5.83	56.15	1.49	15.18	15.88	1.51	0.8	3.16	100
<b>Mean</b>	<b>9.51</b>	<b>56.41</b>	<b>1.36</b>	<b>11.51</b>	<b>16.34</b>	<b>1.53</b>	<b>0.68</b>	<b>2.67</b>	<b>100</b>

## 4. Conclusion

The chemical and mineralogical analysis of the raw material revealed the presence of alkaline oxide and rich amount of ferrous oxide. Waste glass can be considered as potential fluxing additive that can substitute feldspars in wall tile for low heating temperature production. 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. The present results shows that it is possible to use bauxite red mud and CRT waste glass a raw material for roof tile. Physical and mechanical tests were performed to investigate the CRT waste glass addition on the physical and mechanical characteristics of roof tile sample. Mechanical properties and corrosive of fired products improve with the increase of temperature. This growth comes from the vitrification of the material after firing and the formation of the vitreous phase. The present investigation revealed that ceramic tiles can be manufactured using amount of waste glass as binder. This can be explained by the fact that fusible or melting products such as alkaline oxides, alkaline earths and oxides ( $F_2O_3$ ,  $TiO_2$ ) melt and will form a liquid phase. This liquid phase reacts with the solid phases. On cooling, densified products are obtained whose elements are linked by a more or less important glassy phase. More specifically the sample with red mud presented the following characteristics: Linear shrinkage values of samples decreased with increasing waste glass content. Water absorption of samples decreased with increasing flux content and firing temperature. These results showed that the use of this waste material in roof tile could be of help to the environment. From the discussion so far it is concluded that: the mechanical properties of the roof tile varied with varying firing temperature due to phase changes/chemical reaction between the phases in the red mud. The optimum mechanical and chemical property for roof tile within the temperature range considered is obtained at 950°C with 20% waste glass.

## Conflict of Interest

The authors declare no conflict of interest.

## Author Contributions

Djomou paul carried out laboratory tests and physico-mechanical and chemical characterization. Tsamo Cornelius contributed to the interpretation, discussion of the results, the manuscript writing plan and the writing of the document. Harouna Massai contributed to the reading and the formatting of the manuscript. All authors had approved the final version.

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