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Synthesis of a Geopolymer with the Blend of Clay from Share Deposit and Cullet of Cathode Ray Tube

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ABSTRACT— The potentiality of Share clay as an ingredient for synthesis of metakaolin based geopolymer and the effect of partially substituting the clay with the cullet of cathode ray tube (CRT) was investigated. Calcined clay in partial substitution with powdered CRT in the mix proportion of 0%, 25%, 50% and 75% by weight was reacted with sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). The resulting geopolymer samples were used to produce test specimens and the effect of different percentage addition of CRT on the physicomechanical properties were evaluated. The starting materials and the products were characterized using Scanning Electron Microscopy (SEM) to evaluate morphology, X-Ray Fluorescence (XRF) for oxides composition, X-Ray Diffractometer (XRD) for compositional phases, Atomic Absorption Spectroscopy (AAS) for elemental components and Fourier Transform Infra-Red microscopy (FTIR) for characteristic bonding. Results shows that the alumina and silica content of Share clay are 30% and 92% respectively while that of the CRT were 3% alumina and 29% silica. The water absorption of the geopolymer increases with increasing CRT addition. The Compressive strength in 24 hours ranged from 13 to 17 N/mm^2. The drying shrinkage ranged from 0.5mm to 1.25 mm. CRT is as usable as other pozzolanic material to be used with kaolinite in the synthesis of geopolymer. This offers the opportunity for resource recovery from and beneficial use of CRT, which is considered a toxic e-waste.

KEYWORDS: Binder material, Kaolinite, Alumino-silicate, Cathode-ray tube.

1. INTRODUCTION

In the 21st century, the desire for low heat energy process and demand for cleaner and sustainable environment compel not only the development of an alternative binding material to Ordinary Portland Cement (OPC) but also electronic waste (e-waste) recovery and recycling programs. The process of OPC manufacture involves enormous quantity of heat energy and concomitant environmental pollution. In addition, the procurement of the raw feed leads to environmental degradation. Consequently, geopolymer was proposed as a probable substitute binder to the commonly used OPC. The manufacture of OPC requires high energy. Consumption and it releases CO_2 into the environment. Other greenhouse gases are also liberated into the atmosphere during OPC production processes [1], [2]. Beside the environmental pollution caused by the production of OPC, is the potential hazards constituted by the solid electronic wastes (e-wastes) which are non-biodegradable. Prominent among these classes of wastes is the CRT [3]. The CRT is an example of components in some electronic devices with hazardous characteristics which if improperly handled, may pose a risk to public health or the environment. Lead glass mainly comes from waste CRT monitors [4]. Typically, the CRT represents 42% of the TV or the computer monitor's weight and the glasses are a complex mixture of lead, barium, and silica. Studies have demonstrated that the neck and the funnel glasses of the CRT are hazardous waste [5]. Older colour and monochrome CRT glass may contain toxic substances such as cadmium and lead which can be trapped in the geopolymer structure to prevent their release into the environment thereby minimizing potential hazard, which the material could pose if not properly disposed [6]. In order to reduce the emission in the cement processes, many efforts have been made to finding other cementation materials to replace OPC [7]. The production of OPC binder has been recognized as source of between 5-8% of anthropogenic CO_2 emissions worldwide, which causes significant environmental damage. However, geopolymer concretes has

the benefit of significantly lower CO_2 emissions– up to~90% than conventional OPC concrete [8]. Geopolymer starting materials consist of specified amount of silica and alumina which is dissolved in alkaline medium [9]. They contain no dangerous solvents, are incombustible and do not release toxic fumes or gases. The production of geopolymer requires relatively low energy and relatively cheap materials that are available in large quantities. Kaolinite (a type of clay) is the most often used raw material because of its high content of alumina [10]. Yet its continuous mining due to its many industrial applications may lead to environmental degradation and also portends danger to future industrial sustainability.

When natural resource is used faster than it can replenish itself, it becomes rapidly depleted [11]. In order to reduce the rate of depletion of natural resources, spent materials and industrial wastes such as fly ash, blast furnace slag and waste glasses are used as additive in partial replacement for clay during preparation of geopolymer. In the claim of [12], a geopolymer mortar may be formed with the mixture of pozzolanic material, silicon oxide, and alkaline activator solution. The pozzolanic material may be fly ash or metakaolin. The silicon oxide source may be sand. The alkaline activator solution may be composed of a liquid sodium silicate and a sodium hydroxide solution. Davidovits [13] reported that incorporating clay and glass waste forms geopolymer material by the reaction between aluminosilicate powder and an alkali solution. [14] investigated alkaline reactivity potentials of calcined clay and ground waste window glass with NaOH/Na2SiO3 solution to form geopolymer. The study found that waste window glass enhanced both early and late strength gain of the geopolymers and improved the physical properties. [15] mixed cathode ray tube (CRT) glass waste with OPC to produce high-strength mortars and found that sample pieces produced with the mortars showed low elution of hazardous elements when subjected to leaching tests. In allusion to these facts, it is conjectured that the use of CRT cullet as a precursor for geopolymer formulation will be more beneficial. Apart from improved chemical inertness, enhanced physical properties and increased strength that can be achieved by the mixture shown in Figure 1, there is the added advantage of immobilizing the toxic substances found in the CRT. Hazardous wastes can be rendered innocuous through chemical waste stabilization or physical waste encapsulation. The foregoing research efforts have proved that geopolymers exhibit excellent compressive strength (up to 120 MPa) and rapid strength gain, with 95% of their ultimate strength achieved in as little as three days under proper curing conditions [21]. Geopolymers also exhibit low vulnerability to chemical attacks, and are practically inert to attack by sulfate salts because they are not based on calcium silicate. Geopolymers are also inert to alkali- aggregate reaction, which is a common concern with OPC because they are composed of an alkaline silicate network. The inorganic binder is resistant to many detrimental effects of high temperature and can serve as protection for wooden structures and adhesives for binding aggregates. These superior thermal, chemical and mechanical properties make geopolymers alternative construction materials. The geopolymer mortar may be applied to concrete or brick surfaces, and may serve as a corrosion resistant barrier. Therefore, the uniqueness of geopolymer, an inorganic binder, to achieve rapid high strength and the ability to immobilize chemical toxins under different leaching conditions necessitated this research.

2. Materials and Methods

Kaolin was sourced from natural deposits in Share, North central Nigeria. Samples were collected by grab method using random composite sampling technique. The clay samples from Share deposit is essentially siliceous aluminosilicates that belongs to the kaolinite group of clays [16]. The sampled clay was oven dried at 105^{o}C to remove moisture content and subsequently crushed in a rolling rod to reduce the particle sizes to 250µm. The Endicott Ltd, London stainless sieve was used to remove larger particles. The powder was then sieved with a 250µm mesh size; a portion of the clay was heated using a muffle furnace at 700^{o}C for 5 hours to obtain calcined clay in accordance to [17]. The CRT samples were drawn from electronic waste dump at Ilorin Kwara State Nigeria. The tubes were broken with a sledge hammer into fragments of 100-150mm maximum diameter and the broken pieces were washed with water to remove extraneous materials, and then

air dried. The sizes of the pieces were reduced to 10 -15mm equivalent spherical diameter using a laboratory jaw crusher. A toothed roll grinder was used to further reduce the particle size to ≈2-0.4mm and finally pulverized using a ball mill to75% <65µm particle size.

2.1 Particle Size Analysis

The particle size analysis of both calcined and raw share clay was done using Auto sieve shaker, the weight of desired sieve sizes was weighed and recorded, 100g of each sample of raw and calcined clay was weighed on a weighing balance. Stack of sieve set comprised of 500, 250, 125, 63 and < 63µm. The stack was arranged such that the sieve with larger opening was placed at the top, followed by the sieve with smaller opening. A bottom pan was placed last to collect particles less than 63um in size. The clay sample was charged into the stack of sieve from the top with a large sieve size of (500µm) at the top and covered. The stack of sieve runs in the magnetic sieve shaker for 20 minutes. The amount of clay retained on each sieve and the amount that passed through the sieve were weighed and recorded.

2.2 Preparation of samples for elemental analysis

Samples were digested in aqua regia by weighing 0.2g of each of raw clay and cathode ray tube powder into separate conical flasks, add an amount of 10ml of concentrated Nitric acid (HNO_3) to each of the samples and placed on the digestion apparatus and heated to near dryness. Subsequently, 10 ml of concentrated hydrochloric acid (HCL) was added to the drying samples in the flask. Addition of 10ml Hydrofluoric acid and heating to near dryness was repeated, but this time not to complete dryness so as to prevent explosion. Lastly 10ml of concentrated HCL was added to it and cooled for few minutes. The digested samples were filtered and the filtrate transferred into a clean 50ml standard flask and made up to 50ml with distilled water [18].

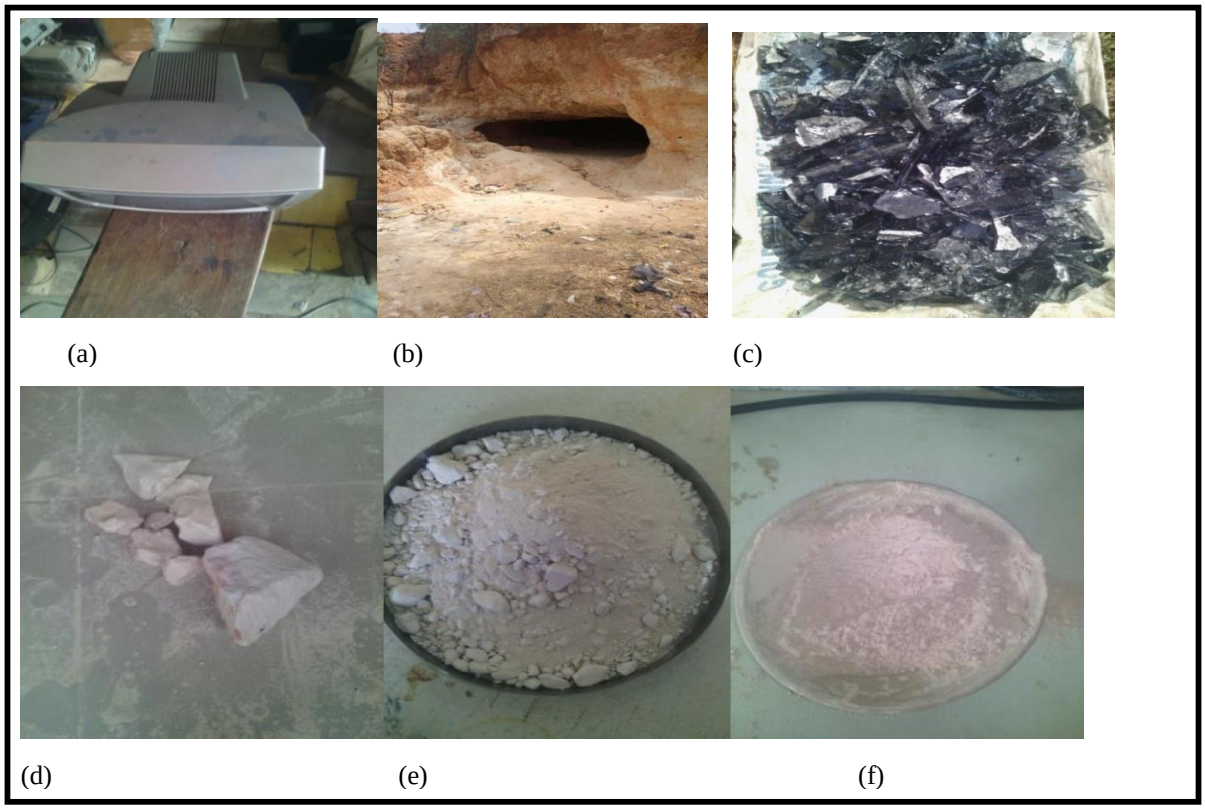


Figure 1. Material samples: (a) Cathode ray tube, (b) Clay deposit at share, (c) Broken cathode ray tube, (d) Raw clay, (e) crushed raw clay, (f) crushed, sieved and calcined clay.

2.3 Preparation of Caustic Activator

An empty beaker was weigh on the weighing balance and 1 liters of distilled water was added to it which makes (1, 238g) the beaker containing 1 liter of distilled water was put inside a bowl half filled with ice and the 320g of sodium hydroxide pellet was pour inside the beaker steadily and stirred with glass rod and left for 30 minutes to cool. The weight of the 8 Molar of sodium hydroxide prepared was weighed as (1, 557g). The same gram of sodium silicate was added to the prepared sodium hydroxide to prepare the activator in weight per weight ($1:1^{w}/_{w}$) and the solution was kept for 24 hours before casting into the mold to cool the solution to room temperature.

Table 1. Mix proportion of the materials used

% ratio of CC : CRT	CC (g)	CRT (g)	Activator (ml)
100:0	1410	0	740
50:50	705	705	600
75:25	1057.5	352.5	600
25:75	352.5	1057.5	600

2.4 Preparation of Geopolymer mortar Samples

Geopolymer mortar with percentage ratio of 100:0, 75:25, 50:50, 25:75 was prepared. The calculated ratio for each raw material in its solid form was mixed thoroughly to homogenize mixture and a calculated volume of activator was added to an agitated homogeneous solution and quickly cast into the mold of (length of 22.2 cm, height of 5.4 cm and width of 13.9 cm) and shaken for 5 minutes with the side of the mold gently tapped to expelled air from the cast. It was found that the raw clay geopolymer mortar was viscous and brown in color. It was covered with aluminum foils and kept for 3 days; the geopolymer was removed from the mold after 3 days and kept in sealed bags to cure at ambient temperature.

2.5 Water Absorption Test

The water absorption test was performed in Kwara State University civil engineering workshop using sensitive weighing balance and mould. Each of the samples was labeled with respective containers. Each container was weighed on a sensitive weighing balance and recorded. The samples were submerged in water and left to become thoroughly saturated. After 24 hours, the samples were removed and dried with towel and air dried for 30minutes. Each of the samples was weighed and recorded as saturated samples. The samples were oven dried at 100^{o}C for 24 hours, each dried sample was weighed and the mass recorded.

3. Results and Discussions

3.1 Particle Size Analysis

The particle sizes of the clays were compared as shown in Tables 2 & 3 to validate the effect of calcination of the clays. The calcined clay showed an increased surface area which enhances the reactivity of the clay and the chemical activator at maximum solid-liquid ratio of the mixture. The percentage of particles that passed through the sieve decreases as the sieve openings narrows in the raw clay while reversed situation was observed with the calcined clay

Table 2. Particle size analysis of raw clay

Sieve Sizes	Mass Of Sieve	Mass Of Sieve + Sample	% Retained	Cumulative % retained	% Passing
250 µm	409.0	433.1	24.1	24.1	75.9
125 µm	409.7	458.2	48.5	72.6	27.4
63 µm	412.1	434.4	22.3	94.9	5.1
<63 µm	381.4	385.5	4.1	99	

Table 3. Particle size analysis of calcined clay

Sieve Sizes	Mass of Sieve(G)	Mass of Sieve + Sample (G)	% Retained	%Passing
250 µm	409.0	427.0	18	18
125 µm	409.7	445.7	36	54
63 µm	412.1	446.5	34.4	88.4
<63 µm	381.4	392.6	11.2	99.6

3.2 Metal Concentrations

Comparatively, metal concentrations of the calcined clay and cathode ray tube were reported in Table 4. Calcined clay presents no lead and cadmium content but Crt showed lead concentration at g/kg and other metals. Cathode ray tube constitutes hazardous waste when spent therefore, immobilizing this class of waste is mandatory.

Table 4: Percentage metal concentrations in calcined clay and cullet of CRT

S/Code	Cd(g/kg)	Fe	Pb	Mg	Mn	Ca	Na	K	N
Calcined Clay	0.00	2.1	0.00	0.35	0.0325	0.725	0.00	0.00	0.00
CRT	0.00	0.00	2.925	0.00	0.00	0.05	0.0625	0.0625	1.99

3.3 Fourier Transform Infrared Spectroscopy (FTIR)

The Fourier transform infra-red analysis was conducted in Chemistry department, Kwara State Nigeria using Schimadzu Japan. The FTIR Spectra of Share Raw Clay and calcined clay are shown in figure 4 & 5. In the IR Spectrum of raw clay, the bands at 3619, 3651, and 3695cm^{-1} shows frequency peaks with medium absorbance strength of OH stretching, at strong bands at 3448cm^{-1} of OH stretching vibrations were observed which are due to the hydroxyl groups attached to the Al octahedron sheet, there are also band associated with OH bending vibration at AL-OH detected at about 912cm-1 while band associated with Si-O-Si stretching vibration were observed at 1032 and 1007cm^{-1}. All these bands established the presence of kaolinite in Share clay. The band observed at 1628 was associated with stretching of OH of adsorbed water. The band at 789 and 754 cm^{-1} are indicative of Al-OH-Si inner surface vibration, the band at 557cm^{-1} are hydroxyl stretching of quartz. In calcined clay the band at 1628cm^{-1} and the absorption bands at 3619 and 3651, 3695cm^{-1} expresses the stretching vibration of –OH groups of kaolinite network in raw clay and absent or has disappeared in calcined clay [19] implies raw clay lost some amount of water when calcined at 700oC. The Si-O-Si stretching and vibration of metal-kaolinite was observed at 1033. The band at 771cm^{-1} was due to 4-coordinated Si-O-Al stretching vibration as reported by [14].

3.4 X-Ray Fluorescence

X- Ray Fluorescence Analysis Result of raw, calcined and cathode ray tube conducted using SKYRAY INSTRUMENT, (EDX3600B), elements between magnesium (Mg, Z=12) and Uranium (U, Z=92) with high resolution and fast analysis. The XRF results in Tables 5 & 6 show that the main component of this clay is silica of (92%) and alumina of (30%) for calcined clay which confirmed that some materials have been lost when it was heated for 700^{o}C. The CRT has silica content (29%) followed by K_2O (4%) also alumina of (3%) while other minerals are present in minimal amount.

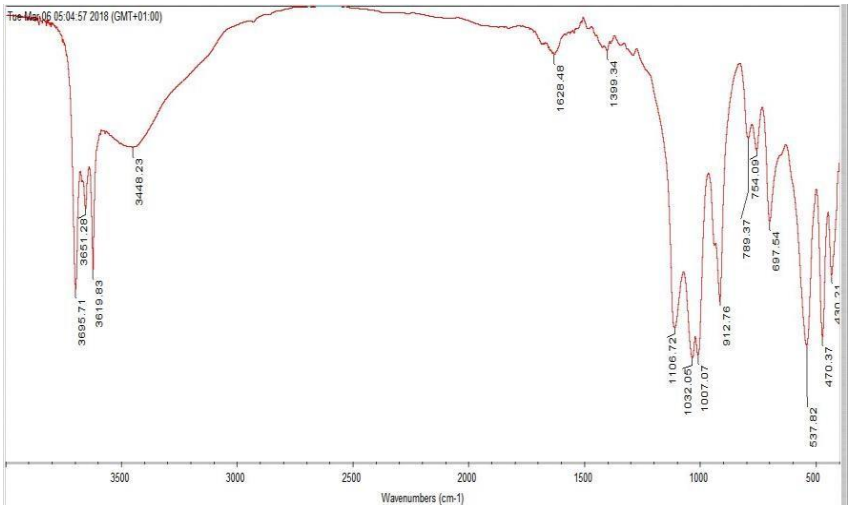


Figure 4: Raw Clay

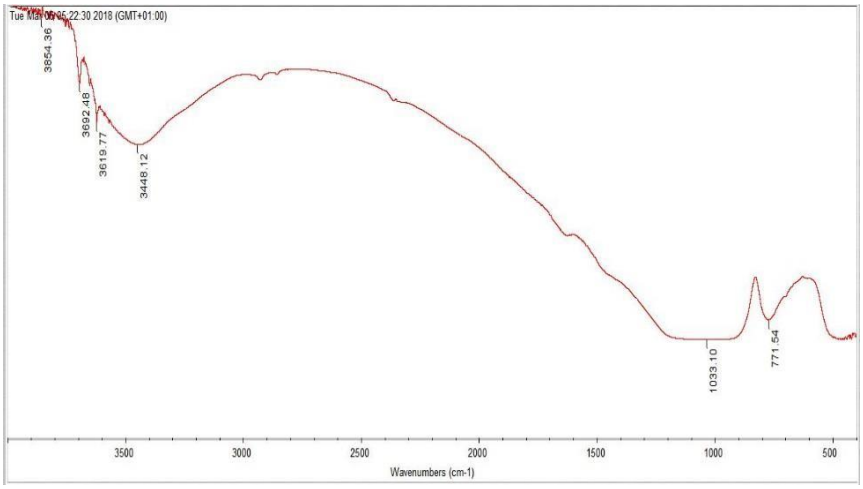


Figure 5: Calcined Clay

Table 5. X-Ray Fluorescence Results

Elemental Composition	Raw Clay	Calcined Clay	Cathode Ray Tube
Al	17.8559	16.1180	1.6132
Si	48.9436	43.2715	13.8560
P	0.1583	0.1301	0.2818

V	-	-	0.1688
K	0.2972	0.2806	3.4611
Ca	0.0512	0.0323	0.0546
Ti	0.0819	0.00	0.1752
Co	0.0104	0.0036	-
Fe	1.4387	1.0166	0.1604
Ni	0.0337	0.0419	-
Mn	-	-	0.0203
Cu	0.0227	0.0398	-
Zn	0.0609	0.0807	0.2649
Sn	1.0827	1.4137	0.0746

Table 6: X-Ray Fluorescence of the clay and the CRT powder

Chemical Composition	Raw Clay	Calcined Clay	Cathode Ray Tube
Al_2O_3	33.7387	30.4549	3.04814
SiO_2	104.7050	92.5707	29.6421
P_2O_5	0.3627	0.2981	0.645716
V_2O_5	-	-	0.30134
K_2O	0.3580	0.3380	4.16924
CaO	0.0716	0.04519	0.076396
TiO_2	0.136617	-	0.292286
CoO	0.01321	0.0045756	-
FeO	1.85089	1.30786	0.20635
NiO	0.04288	0.053317	-
MnO	-	-	0.02621
CuO	0.0284	0.04982	-
ZnO	0.07581	0.100455	0.329747
SnO2	1.37459	1.79483	0.09471

3.5 Scanning Electron Microscopy

Scanning Electron Microscopy shows the morphological features of the raw clay and metakaolin. It shows the microstructure with no heat treatment and after heat treatment at 700^{o}C. The raw clay has a large grain size and the boundary is big, after heating at 700^{o}C the clay develops a clearer, lesser grain size and boundary. The amorphous of some of the element are almost transformed to the crystalline phase and the surface becomes

smoother as shown in Figure 3.

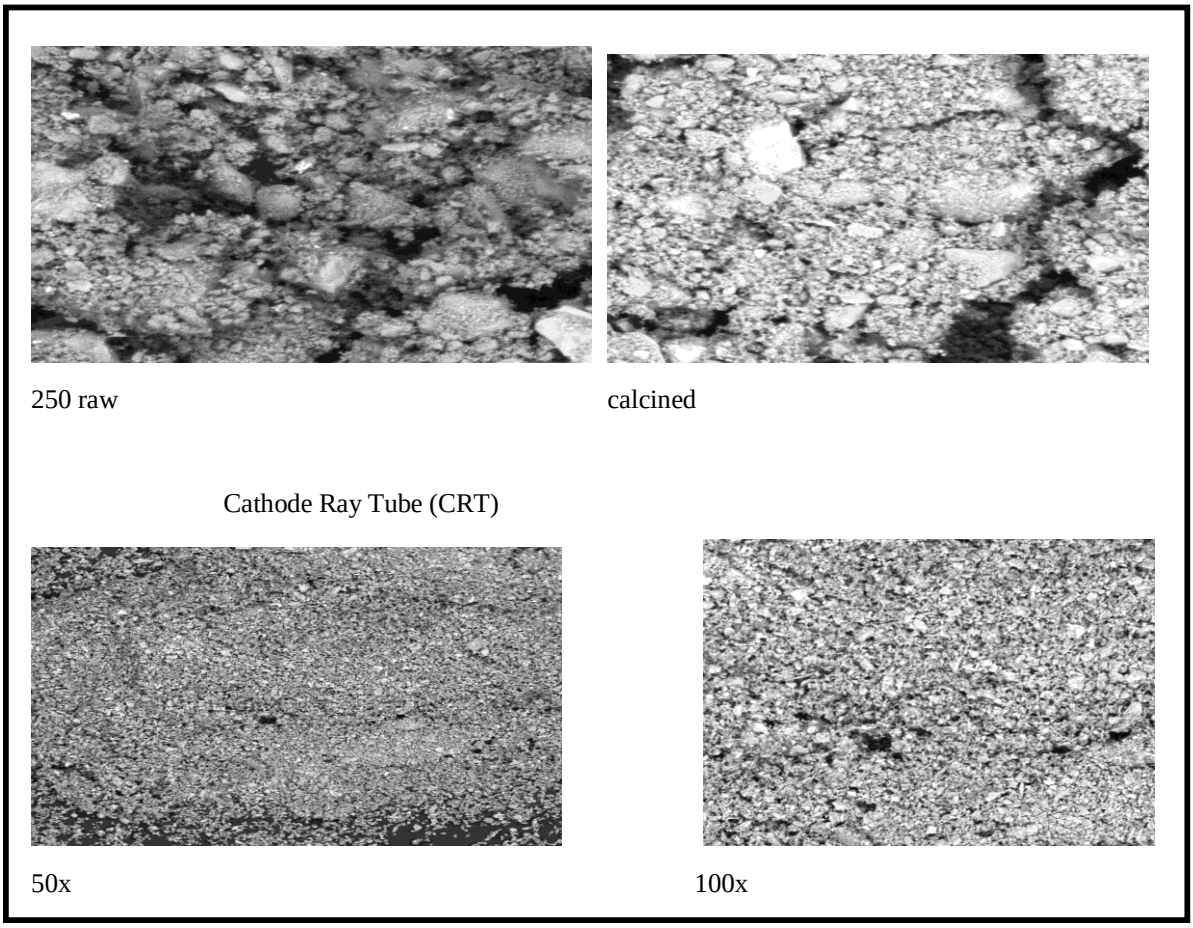


Figure 3: Scanning Electron Microscopy (SEM)

3.6 Slurring of the Clay/Crt ratio

During the formation of the geopolymers of different ratios as presented in Table 7, ratio (100:0) release bubbles and after shaking for few minutes the mould become very hot, at ratio (50:50) it produce bubbles but not as much as that of 100% clay and the mould has less heat, ratio (75:25) the bubbles and hotness is also reduced and (25:75) has the least bubbles and hotness and is very slow to solidify after 24hours with a jellylike surface compared to others. The heat liberated during the formation shows it is an exothermic reaction.

Table 7: Mix proportion of the material used to produce geopolymer

% ratio of CC : CRT	CC (g)	CRT (g)	ACTIVATOR (ml)
100:0	1410	0	740
75:25	705	705	600
50:50	1057.5	352.5	600
25:75	352.5	1057.5	600

3.7 Water absorption

Water absorption determines geopolymer durability, the result of water absorption test in Table 8 for geopolymer sample containing 75 calcined clay shows the highest water absorption capacity followed by the

one of (25%) while geopolymer with highest percentage of clay (100%) had the lowest water absorption. By replacing calcined Share clay with cathode ray tube made the geopolymer to be less porous and improved their ability to limit water penetration into the polymer structure as compared with [20].

Table 8. Water Absorption of Geopolymer

MIX GEOPOLYMER	RESULTS (%)
100:0	32.14
50:50	37.29
75:25	57.38
25:75	53.44

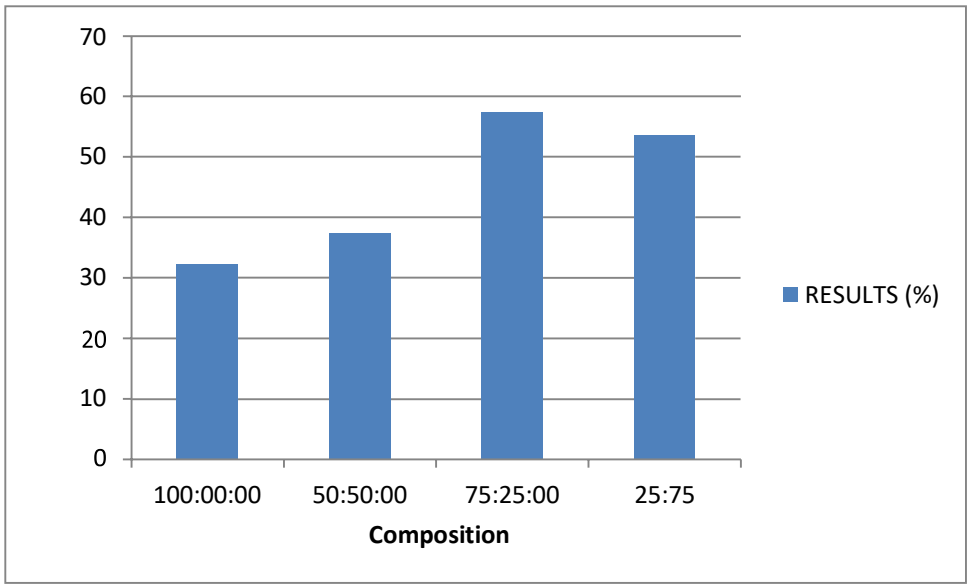


Figure 4. Water Absorptivity of different geopolymer mortals

3.8 Compressive strength

The compressive strength of all the uncured geopolymer samples range between 13.44 N/mm^2 and 17.2N/mm^2 (Table 9). [18] recommended 5 N/mm^2 as the minimum compressive strength for a load-bearing concrete masonry. The British Standard for precast concrete masonry units of 1981 recommends a compressive strength of 7N/mm^2. The strength of these geopolymers are therefore comparable with those made with concrete admixtures The measured compressive strengths are also expected to increase with curing making it stronger with time. It can also be deduced that the optimum cathode ray tube amount that yield the highest strength is 75:25.

Table 9: Compressive strength of the geopolymer samples

Sample number	Compressive strength (N/mm^2)
Sample A (50:50)	13.44
Sample B (75: 25)	17.26
Sample C (100:100)	16.37

3.9 Drying Shrinkage

The drying shrinkage of the geopolymer ranged from 0.5 to 1.25% (is it mm or % as compared to what is presented in the table) (Table 10). It is observed that the sample with 100:0 has the lowest and most desirable drying shrinkage. The maximum recommended drying shrinkage for samples of masonry units tested in accordance with the British Standard for precast concrete masonry units of 1981 is 0.06 % (21). While samples (75:25) and (100:0) are compliant, sample (50:50) drying possess drying shrinkage 100% higher than the recommended value.

Table 10: Drying shrinkage of the geopolymer samples

Sample Number	Drying shrinkage (%)
Sample A (50:50)	1.25
Sample A (75:25)	0.55
Sample A (100:0)	0.5

4. Conclusion

The use of kaolinite clay as an ingredient for synthesis of metakaolin based geopolymer and the effect of CRT was established. Admixture of Calcined clay with powdered CRT reacted with sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). The resulting geopolymer samples produced showed strong physicomechanical properties. The water absorption of the geopolymer increases with increasing CRT addition. The Compressive strength in 24 hours ranged from 13 to 17 N/mm^2. The drying shrinkage ranged from 0.5mm to 1.25 mm. CRT is as usable as other pozzolanic material to be used with kaolinite in the synthesis of geopolymer. This offers the opportunity for resource recovery from and beneficial use of CRT, which is considered a toxic e-waste.

5. Compliance with ethical standards

Conflict of interest. The authors declare that they have no conflict ofinterest.

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