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# X-ray Fluorescence analysis of geopolymer produced from spent fluid catalytic cracking catalyst and Kaolin

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# Abstract

Spent Fluid catalytic cracking catalyst, raw and calcined Kaolin were used to produce Geopolymer which was analysed using X-ray Fluorescence spectroscopy. The influence of curing conditions on the properties and microstructures of the geopolymers was further investigated. The compressive strength of the various geopolymer formulations were determine after curing to ascertain its utilization for the production of low-cost energy-saving geopolymer products. Results of the compressive strength of the 100% kaolin formulation (B<sub>100</sub>) was 22.88 N/mm<sup>2</sup> at 14 days and 32.73 N/mm<sup>2</sup> 28 days respectively which showed that the strength of the produced Geopolymer improved as the curing day increases. Result of X-ray Fluorescence spectroscopy showed the following elements Si, Al, Fe, Ti and heavy metals such as Co, Zr, Pd, Pb, Y, Nb, Au. However, Mg, K and Ca were not detected as reported by previous studies. The result of the amount of the reactive elements (Si and Al) gave Si as 49.55 and Al as 11.06 for the raw Kaolin; Si as 46.27 and Al as 12.13 for the calcined Kaolin and Si as 63.82, Al as 29.14 for the produced geopolymer. The Si: Al of the produced geopolymer is 2.08 which makes it a polysialate geopolymer with excellent geopolymeric properties, which can be considered as an important material for building, pottery and major components in material and technological industries.

Keywords: Kaolin, elemental analysis, XRF, Metakaolin, Geopolymer, polysialate.

# Introduction

Geopolymers are three-dimensional alumina-silicate materials which have ceramic-like characteristics such as chemical resistance, electrical and thermal insulation properties & brittle at a reduced temperature<sup>1</sup>. Generally, the blending of polymers is a suitable and effective method used to expand modern research and obtain new materials with improved characteristics<sup>2</sup>. Geopolymers with excellent and improved characteristics can be improved from such methods. Blending a fine pure metakaolin (Na<sub>2</sub>SiO<sub>3</sub>) solution, sodium hydroxide pellets and water can be used in making a simple geopolymer. Geopolymerization materials are numerous and cover materials that are rich in Si (such as fly ash, slag and rice husk) and high in Al (clays such as kaolin, bentonites). Kaolin is an industrial clay mineral of great importance chemically composed of alumina, silica, oxygen and hydrogen with chemical formula Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>. The raw materials (raw kaolin and metakaolin) in this study are Class F materials according to ASTM-C618, 2004, as they have no calcium content amidst others (N and C). The key discrepancies between the classes are the Si + Al + Fecontent, for Class N and F must have > 70 wt% and > 50 wt% Class C. The geopolymer properties vary with Si/Al, Na/Al and the water content<sup>3,4</sup>. Geopolymer's physical, chemical and microstructural qualities depend considerably on the formulation. Nigeria has abundant Kaolin deposit which can be sourced as wastes from quarry sites. Important occurrences have

been reported in southern and western Nigeria with few in the eastern part of the country<sup>5</sup>. At the temperature range of 550-600°C endothermic dryness of kaolinite starts to produce disordered metakaolin, but constant hydroxyl loss is perceived at 900° $C^6$ . Therefore, dehydroxyllation of clay (kaolinite) results in metakaoline with alumina structures of 4, 5 and 6 coordinated aluminum ions<sup>7</sup>. The dehydroxylation process is an endothermic process because of the energy needed to remove the hydroxyl ions that are in a chemical bond. The hydroxyl ions are closely linked to the alumino-silicate frame thus they may be removed only at temperatures above 550°C. Different analytical methods have been developed for the characterisation and polymers. synthesized Such methods include: Gas chromatography-flame photometric detection, gas chromatography-mass spectrometry, electrochemical methods, polarography, X-ray fluorescence spectrometry (XRF), Field emission scanning electron microscopy, X-ray diffraction, differential scanning calorimetry and FTIR, spectroscopy<sup>5</sup>.

This research adopted the X-ray fluorescence spectrometry (XRF) to characterise the raw kaolin and metakaolin which are starting materials and the geopolymer produced. Knowing the quantity and composition of the reactive elements will allow a realistic and usable formulation, however, cannot account for the extent of reaction of the responsive components<sup>8</sup>. Determination of the elements is a key step in determining the nature of minerals like kaolin. Elements like Si, Al, Fe, Mg, Ti,

Mn, P, Ca, K and Na are essential in the study of clay minerals in their structural formulas<sup>9</sup>. X-ray fluorescence (XRF) is an analysis of the chemical composition of many types of materials (solid, liquid, powder, filtered and other forms) as well as the concentrations of large, small or, in some cases, trace elements. XRF is rapid, accurate and also non-destructive in some circumstances<sup>10</sup>. The scope of application for XRF analytics includes a very wide range of applications including metals, concrete, oil, polymer, plastics and food sectors as well as mining, mineralogy and geology. Although XRF is a common instrumental method for analysing various materials and numerous standard processes have been established, the main problem associated with its direct use to characterize newly developed materials is the unavailable reference materials with certified contents of specific elements. X-ray fluorescence spectroscopy (XRF) is commonly used in conjunction with Xray diffraction (XRD) because XRF can only detect elemental sample composition, but cannot be utilized to extract phase or crystallographic information, hence it is vital to integrate XRD and XRF information to achieve a complete understanding of the sample composition<sup>8</sup>.

The most popular XRF configuration is the XRF energy dispersion (ED). A high-energy x-ray beam impacts the sample surface during an ED-XRF experiment. This in turn emits lower-energy x-rays, a behaviour called 'fluorescence.' Each chemical component creates luminous x-rays with energy unique to that component. The emitted X-rays are shown as a spectrum with peaks at different levels at different energy. The energy of a peak identifies the chemical element that makes it and the height of a peak typically may be connected to the sample's abundance. The abundance of discovered items is then measured and quantified using a software. Materials of reference are always used to calibrate the process or to check for the outcomes. Depending on the equipment used, the analysis is given in terms of the various elements or of their oxides.

This research led to the production of a geopolymer from spent fluid catalytic cracking catalyst, metakaolin by the calcination of Kaolin which iscost effective and energy saving. The properties of metakaolin-based geopolymer can be productively utilized in the manufacture of fire resistant/fireproof materials, cement, refractories, ceramics, decorative pieces, and thermal insulators.

The research is aimed at determining the likelihood of utilizing Spent Fluid Catalytic Cracking Catalyst and Kaolin to produce a low cost energy saving geopolymer for various applications.

# Methodology

Raw kaolin clay was obtained from Somak industries, Auchi, Edo State, Nigeria. The physical form is off-white, it was pulverized and sieved before chemical analyses. A commercialgrade  $Na_2SiO_3$  was purchased from Redal Chemicals Nigeria, in Port Harcourt, Rivers State. The pure metakaolin ( $Na_2SiO_3$ ) has the following chemical constituents:  $SiO_2$  29.4%,  $Na_2O$  14.7% and  $H_2O$  55.9% while the sodium hydroxide (NaOH) used pellet used has 99.9% purity.

The spent fluid catalytic cracking catalyst (SFCC) was obtained from Port Harcourt Refining Company (PHRC), a subsidiary of the Nigerian National Petroleum Corporation.

Loss on ignition (LOI) at 1000°C was determined on the raw kaolin (RK), using the standard operating procedure (SOP). Handheld XRF spectrophotometer (Oxford Instrument X-Met 7500) was used to analyse the chemical composition content of the raw kaolin (RK), calcined kaolin at 650°C (CK<sub>650</sub>) and the produced geopolymer sample. The concentration of the various elements present in the samples were displayed on the screen and the results were immediately printedout<sup>1</sup>.

Geopolymer Sample Preparation: Ten grams (10g) of raw kaolin (RK) was put in a crucible and placed in a pre-set muffle furnace at 650°C. The furnace was switched on and observed until the temperature rises to 650°C to activate the switch off alarm. The calcined kaolin was removed, allowed to cool and labeled CK<sub>650</sub> as the starting material. This experiment was repeated in batches until the quantity needed was obtained. 100 ml of water was added to 160g NaOH in a beaker and stirred until the NaOH dissolved. The solution was poured into a 500ml volumetric flask and deionized water was used to make up to volume. It was allowed to cool and labelled 8 M NaOH solution. 250ml Na2SiO3 was added to 100 ml 8.0M NaOH solution in a 1L beaker. The mixture was stirred and transferred to a 500ml volumetric flask and labelled as the reaction solvent and left for 24hrs before use. 1.2 ml of the reaction solvent was added to 3g premixed calcined kaolin (AC<sub>650</sub>) thoroughly mixed to produce a smooth paste. The paste was transferred to a 140  $\times$ 40×40mm mould, vibrated for 10 minutes and covered with transparent polyethylene sheet and allowed to set at room temperature. The sample was removed from the mould after 3 days, labelled as sample  $B_{28}$  and allowed to cure in a plastic bag for 28 days.

**Determination of Loss on Ignition:** The samples were ovendried at a temperature of 40°C and a weighed crucible (preignition weight) was placed in furnace for 2 hrs at 1000°C. The crucible and sample were placed in a desiccator to cool and was reweighed (post-ignition weight). Percentage loss on ignition (LOI) was calculated using the formula:



#### **Results and discussion**

The kaolin was analysed chemically to know its elemental compositions. Results shown in Table-1 are the X-ray Fluorescence of the raw kaolin (RK) and calcined kaolin at  $650^{\circ}$ C (CK<sub>650</sub>) while Table-2 shows the X-ray Fluorescence chemical composition of the Spent fluid catalytic cracking

catalyst (SFCC) and that of Spent fluid catalytic cracking catalyst calcined at 800°C (SFCC<sub>800</sub>). The results of X-ray fluorescence analysis of the produced geopolymer are shown in Table-3. The results showed that Si, Al, Fe and Ti are main elements which are higher in concentration than other elements in minimal concentrations. When the results were compared with typical clay minerals, Mg, K and Ca were not present in the studied sample and the values obtained for Si, Al, Fe and Ti were higher than the previously reported result of Ahmad et al.<sup>9</sup>. X-ray fluorescence (XRF) results showed that the total percentage of Si, Al and Fe for calcined kaolin (CK<sub>650</sub>) is 71.69% while that of raw kaolin (RK) is 73.87% with loss on ignition of 2.9%.

**Table-1:** X-ray Fluorescence Analysis of the Raw kaolin and Calcined kaolin at  $650^{\circ}$ C (CK<sub>650</sub>).

Elements (Wt %)	Raw Kaolin	CK <sub>650</sub>	
Si	49.55	46.27	
Al	11.06	12.13	
Fe	13.26	13.05	
Ti	13.61	16.35	
Со	0.28	0.00	
Ni	0.00	0.00	
Zn	9.50	9.16	
Zn	0.00	0.11	
Pd	1.53	1.33	
Pb	0.16	0.00	
Y	0.27	0.25	
Nb	0.44	0.39	
Au	0.34	0.00	
Мо	0.00	0.25	

**XRF characterization of raw kaolin (RK) and calcined kaolin (CK<sub>650</sub>):** The primary materials in the formation of a geopolymer must be rich in amorphous forms of Si and Al. The sum of silica, alumina and iron oxide of total elements should amount to a minimum value of 70 per cent (compounds) making up the pozzolanic materials with loss on ignition (LOI) of less than 10%. The higher the quantity of silica available in the combination or mix, the stronger the geopolymer<sup>11</sup>. This is because, silica influences the formation of particle interaction, yielding better alumina silicate bonds that result in better mechanical properties. The calcined kaolin has a Si:Al of 3.6 which makes it suitable in civil engineering works because of low Si:Al ratio of the range 1 to 3 results to a 3D structure that is precisely firm<sup>12</sup>.

**Table-2:** X-ray Fluorescence Analysis of the SFCC and  $SFCC_{800}$ .

Elements (Wt.%)	SFCC	SFCC <sub>800</sub>
Si	47.22	45.60
Al	13.00	15.08
Fe	16.11	16.14
Ti	17.57	17.66
Со	0.57	0.00
Ni	3.18	2.92
Zr	0.72	0.79
Zn	0.00	0.00
Pd	0.92	0.97
Pb	0.00	0.00
Y	0.00	0.00
Nb	0.34	0.28
Au	0.36	0.56
Мо	0.00	0.00
LOI	4.37	

 $SFCC = Spent fluid catalytic cracking catalyst. SFCC_{800} = Spent fluid catalytic cracking catalyst calcined at 800°C$ 

XRF characterization of the produced geopolymer sample  $(B_{28})$ : The chemical composition of the produced geopolymer after 28 days cure  $(B_{28})$  in terms of its reactive components (Si, Al and Fe), is shown in Table-3. The increased percentage composition of silicon is associated with the added reagent  $(Na_2SiO_2 + NaOH)$  whereas that of Al is traced to Al inclusions. This gives way to change in microstructure of the made geopolymer<sup>13,14</sup>. The actual Si:Al obtained from the calcined kaolin or metakaolin-based geopolymer is 2.1. The targeted Si/Al ratio was 3.6 which was obtained from the Si:Al of CK<sub>650</sub>. The difference or variations of the targeted and actual ratios is 1.5. This variation could be caused by inadequate mixing time, using a non-effective shear mixer that could not breakdown the aluminosiliate and an in correct liquid-to-solid ratio, since metakaolin requires higher liquid to solid ratios to achieve adequate mixing<sup>8</sup>. Irrespective of the compositional variation, the actual Si:Al of the produced geopolymer sample is still suitable for civil engineering works and ceramics production as they require low Si: Al of 1 to  $3^{12}$ .

Comparison of X-ray fluorescence analysis of the reactive elements of the starting materials and produced geopolymers: The chemical composition of the produced geopolymers ( $A_{28}$ ,  $B_{28}$  and  $D_{14}$ ) and the starting materials ( $AC_{650}$ ) & SFCC<sub>800</sub>) as it concerns their reactive components (Si, Al and Fe), are shown in Table-4. There are discrepancies in the compositional ratios (targeted and actual Si/Al ratio). This implies that part of the starting materials reacted and there was dramatic shift in the mechanical and physical properties<sup>8</sup>. The increased percentage composition of silicon is associated to the added reagent (Na<sub>2</sub>SiO<sub>2</sub> + NaOH) whereas that of Al is traced to the aluminium inclusions that results from a band shift towards a lower wave number. This gives way to change in microstructure of the made geopolymer<sup>13,14</sup>. The actual Si:Al obtained from the calcined kaolin/metakaolin-based geopolymer is 2.1 and 2.00 for  $B_{28}$  and  $D_{14}$  products respectively. The targeted Si/Al ratio was 3.6 which was determined/obtained from the Si:Al of AC<sub>650</sub>. The difference or variations of the targeted and actual ratios are 1.5 and 1.6. These variations could be caused by inadequate mixing time, using annoneffective shear mixer that couldn't breakdown the aluminosiliate and an incorrect liquid-to-solid ratio, since matakaolin needs greater fluid to solid ratios for proper mixing<sup>8</sup>. Irrespective of the compositional variation, the actual Si:Al of the produced geopolymer samples are still suitable for civil engineering and ceramics production as they require low Si:Al of  $1-3^{13}$ .

**Table-3:** X-ray fluorescence analysis of the producedgeopolymer.

Elements	B <sub>28</sub>	
Si	63.82	
Al	29.14	
Fe	1.32	

Where:  $B_{28}$  = geopolymer produced after 28 days cured.

**Table-4:** Comparison of X-ray fluorescence analysis of the reactive elements of the starting materials and produced geopolymers.

Elements	CK <sub>650</sub>	SFCC <sub>800</sub>	A <sub>28</sub>	B <sub>28</sub>	D <sub>14</sub>
Si	46.27	45.6	63.8	63.82	64.51
Al	12.37	15.08	27.32	29.14	31.05
Fe	13.05	16.14	3.3	1.32	0.84

Note:  $A_{28}$  = geopolymer product obtained from formulation  $A_{100}$ , cured for 28 days,  $B_{28}$  = geopolymer product obtained from formulation  $B_{100}$ , cured at 28 days and  $D_{14}$  = geopolymer product obtained from formulation  $D_{30:70}$ , cured at 14 days.

**Compressive Strength of produced Geopolymer samples:** The outcome of the strength properties of the geopolymer pastes produced is displayed (Table-5, Figure-1). The table illustrates that as the curing day increases, the strength of the synthesized geopolymer increased. This means a continuous chemical reaction in the geopolymer samples gave rise to better strength. The 100% kaolin formulation ( $B_{100}$ ) showed the peak strength at 14 days (22 N/mm<sup>2</sup>) and 28 days (32.73 N/mm<sup>2</sup>) respectively. This strength is comparable to those obtained from previous studies of Tseng et al.<sup>17</sup> even with different formulation conditions such as calcination temperatures, curing temperatures and duration. The compressive strength obtained after 7 days for sample  $D_{14}$  is closely related to the value obtained in a study by Eric<sup>16</sup>.

**Table-5:** Compressive strength (N/mm<sup>2</sup>) of produced geopolymer paste samples.

Mix design	7days (N/mm <sup>2</sup> )	14 days (N/mm <sup>2</sup> )	28days (N/mm <sup>2</sup> )
A <sub>100</sub>	2.78	5.19	5.44
B <sub>100</sub>	8.04	22.88	32.73
C <sub>30:70</sub>	2.78	1.31	1.00
D <sub>30:70</sub>	0.71	12.81	9.35

Note:  $A_{100}=100\%$  calcined spent catalytic cracking catalyst (SFCC<sub>800</sub>),  $B_{100}=100\%$  calcined Kaolin (CK<sub>650</sub>),  $C_{30:70}=30\%$  SFCC<sub>800</sub> and 70% calcined alumina,  $D_{30:70}=30\%$  calcined Kaolin (AC<sub>650</sub>) and 70% calcined alumina.

However, researchers like Hong Jiang *et al.*, described synergistic effect played in the strength and toughness of polymers blends like poly (1, 4-cyclohexylene dimethylene terephthalate glycol) which showed marked improvement in their properties due to the blending. The compressive strengthgain for geopolymer could also be as a result of the level of the raw material dissolution through geopolymerization, that is, the discharge of Si and Al species into the geopolymer gel<sup>4</sup>. In a better embodiment, compressive strength of 15 to 60 N/mm<sup>2</sup> on or before 28 days should be exhibited by a geopolymer composition<sup>3</sup>.



**Figure-1:** Compressive strength of geopolymers produced using different ratios.

The compressive strength of the samples made from SFCC<sub>800</sub> revealed the maximum strength of 5.44 N/mm<sup>2</sup> for 28 days. This value is lower than values from previous studies<sup>8,15</sup>. The variation may be owing to formulation conditions. The study done by Carla et al.<sup>15</sup> showed increased strength of the SFCC pastes on incorporating 30% Ca(OH)<sub>2</sub> to binder composition in the first 28 days of reaction, hence essential for an increased strength. The geopolymer samples of this study were formulated with SiO<sub>2</sub>:Al<sub>2</sub>O<sub>3</sub> of 2.9 and Na<sub>2</sub>O/SiO<sub>2</sub> of 0.48. The strength reduction in spent catalyst-based geopolymer sample can be attributed to excess silicates in the mix as the polymerization degree of silica is high with a slow rate of reaction required for optimal compressive strength. The higher Na<sub>2</sub>O/SiO<sub>2</sub> ratio increases the alkalinity, thereby promoting a higher degree of dissolution which gives rise to higher availability of Al-rich species. This results to more Si-O-Al bonds instead of Si-O-Si bonds which are stronger<sup>13,15</sup>. The low compressive strength also shows that the spent catalyst is an equilibrium catalyst  $(E_{cat})$ which is generally lower in strength to electrostatic precipitator catalyst (Ep<sub>cat</sub>)<sup>17</sup>. Despite the low strength of spent catalyst based-geopolymer, they can be used as plasters, coatings and partial cement replacement in cement and concrete<sup>17,18</sup>.

The 30:70 formulations which consist of 30% starting material and 70% calcined alumina gave a reduced compressive strength. This strength infers that the dosage of calcined alumina added to improve compressive strength of the geopolymer pastes was excessive unlike the dosage used in earlier studies<sup>19</sup>. When alumina content is excessively increased, that is, low Si/Al ratio, the geopolymer samples exhibit low strength<sup>20,21</sup>. The compressive strength value obtained B<sub>100</sub> formulation can be used as building bricks in severe, moderate and negligible weathering conditions and as paving bricks in a moderate and no weathering condition. The geopolymer samples with low compressive strength can give an artificial stone for use as decorative piece<sup>2</sup>.

# Conclusion

The X-ray Fluorescence (XRF) chemical analysis showed that the major constituents of kaolin are Si, Al, Fe and Ti in major quantities with many other elements in trace concentrations. The experimental results also showed that geopolymer production is possible with alumina silicate material like calcined kaolin because it met the minimum value of 70% for the sum total of silica, alumina and iron oxide making up the pozzolanic materials with loss on ignition (LOI) of less than 10% (2.9). The properties of the produced geopolymer is controlled by its Si: Al ratio. Thus, the geopolymer obtained from metakaolin with Si:Al of 2.1 is a Polysialate type and the compressive strength of the produced geopolymer increased as the curing day increases. This means a continuous chemical reaction in the geopolymer samples giving rise to better strength which makes them to be considered as an important material for building, pottery and major components in material and technological industries.

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