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# Workability and durability analysis of waste based geopolymer concrete

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***Abstract***
*Geopolymer concrete (GPC) as a promising material, started gaining momentous attention from the researchers and construction specialists because of its advantages in using by-products from agriculture and industries to replace cement thereby reducing greenhouse gas emission. Workability and durability tests were carried out, so as to ascertain the efficiency of the rice husk ash and rice husk as sodium silicate and super plasticizer, for solving efflorescence problem known with geopolymer concrete. The factory-made sodium silicate was also used and compared with laboratory-produced, before addition of other geopolymer binders such as kaolin clay and fly ash. Brunauer-Emmett-Teller (BET) scrutiny that delivers quantitative data on the specific surface area as well as porosity dispersal of solid materials revealed that correlation coefficient of RHA (0.994) was higher than that of cement (0.991). RHA has higher surface area (250.023 $m^3$/g) than the cement utilized which stood at 211.49 $m^3$/g. Though the c constant of cement exceeds the RHA values, RHA can serve as good pozzolanic material and cement substitute. It is seen that the workability of laboratory-produced sodium silicate and superplasticizer geopolymer concrete was enhanced significantly compared to that of prior geopolymer systems. It is observed that the workability of the geopolymer concrete was considerably enhanced compared to that of orthodox geopolymeric systems, especially at 2.5% rice-husk created superplasticizer and higher content of laboratory-produced sodium silicate.*



***Keywords:***
*Brunauer-Emmett-Teller;*
*Rice Husk;*
*Sodium Silicate;*
*Strength;*

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## INTRODUCTION

Geopolymer concrete (GPC) is a green material that possesses better strength properties and durability compared to normal concrete. Despite these advantages, the use of geopolymer concrete in practice is considerably limited [1][2]. The unique aspect of GPC as replacement of traditional concrete for upstream carbon capture and as viable development in the construction industry for developing nations cannot be overemphasized [2][3]. It satisfies green (high performance, and eco-friendly applications) and socio- economic (society approval, commercial, and rate) attributes of sustainability [3, 4, 5]. GPC is dual face material that combines aluminosilicate materials as binder and strong alkaline solutions as activator [1, 6, 7, 8]. It is more resistant to corrosion and fire, shrinks less than conventional concrete [9][10], has great compressive and tensile strengths [2][11], excellent durability, and it gains its full strength rapidly [13, 14, 15].

Past research on geopolymer had centered on the use of alkali-activated fly ash and slag as binder [3, 16, 17, 18], investigation of non-calcined natural kaolin as partial replacement of cement [19, 20, 21, 22]. Also, study on development of geopolymer concrete using ground granulated blast furnace slag [23, 24, 25] and palm oil fuel ash-fly ash [26, 27, 28, 29, 30, 31, 32]. Thus, this research will provide a sound platform which will reveal the full potential of the developed GPC as novel concrete that can provide enhanced strength and durability at different curing temperatures.

## MATERIALS AND METHODS

### Materials

Materials utilized in this research were kaolin clay, Portland cement, river sand, sodium hydroxide, factory-produced sodium silicate, laboratory-produced sodium silicate and super plasticizer, crushed granite, sand and potable water, as depicted in Figure 1.

### Methods

GP concrete was thoroughly mixed, production of four concrete mixes; viz: control concrete, pozzolanic concrete (factory-produced sodium silicate), pozzolanic concrete (laboratory-produced sodium silicate) and Geopolymer as listed in Table 1, before workability and durability tests were performed on the three categories of specimens produced based on BS 8100, BS 12350-2-2019, ASTM C 143 and ASTM C597/C597M-16 codes. The GP concrete was removed from the moulds after 24 hours, as seen in Figure 2, and cured in water for 3, 7, 14, 21, 28 and 56 days.

(a) (b) (c)

Figure 1. (a) Cement, (b) laboratory-produced super plasticizer (from rice husk) and (c) Fly ash, rice husk ash and kaolin clay

(a) (b) (c)

Figure 2. (a) Mixing in the laboratory, (b) gas cooker used in making super plasticizer and (c) geopolymer concrete open dry for 24 hours

Table 1. Mix design proportions for geopolymer concrete

| Sample | Molarity (M) | BAR | FA (g) | RHA (g) | KC (g) | FA | CA | Na2SiO2/NaOH | Water |
|---|---|---|---|---|---|---|---|---|---|
| A1 (Control) | | 40:60 | cement | 240 | 240 | 360 | 720 | | 0.45 |
| A2 | 12 | 40:60 | 240 | 240 | 240 | 1080 | 2160 | 2.5 | 0.45 |
| A3 | 12 | 40:60 | 240 | 240 | 240 | 1080 | 2160 | 2.5 | 0.45 |
| A4 | 12 | 40:60 | 240 | 240 | 240 | 1080 | 2160 | 2.5 | 0.45 |

BAR - Binder Aggregate Ratio, FA – Fine Aggregate, CA – Coarse Aggregate.

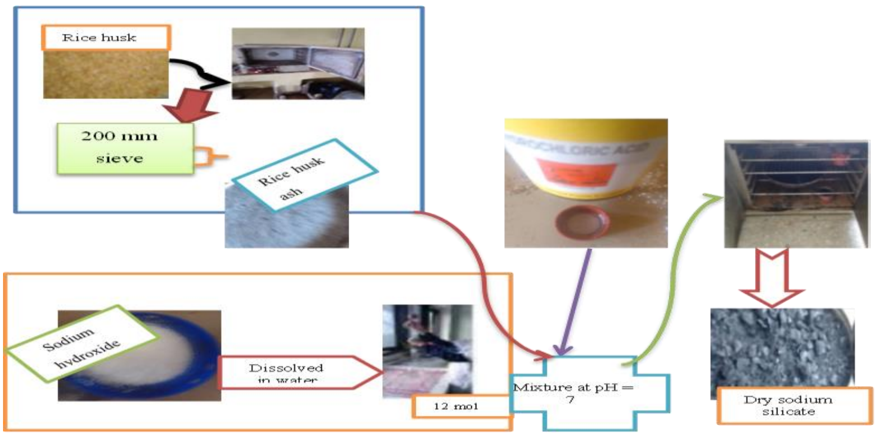



Figure 3. Preparation of laboratory-produced sodium silicate from RHA.

### Preparation of Laboratory-produced Sodium Silicate

Firstly, RHA was filtered through 200 sieves in order to achieve standardization in size. Additionally, sodium hydroxide of 12Mol was dissolved inside water based on the standard concentration. Then Rice husk ash of 100 grams were measured and mixed with a 600 ml sodium hydroxide solution, then hydrochloric acid was added to the proportion of RHA and sodium hydroxide solution at 1:20 respectively, so as to control the pH to neutral. This material was boiled for over 1 hour at 110 °C, before the solution was sifted, dried and kept at room temperature as shown in Figure 3.

### Preparation of Laboratory-produced Superplasticizer

For producing the locally made superplasticizer in the laboratory, the rice husk was boiled together with sodium hydroxide, a highly alkaline solution in water. 500g of sodium hydroxide was weighed and dissolved in 1000 ml of water to create 12M, before addition of100 g of rice husk to the solution. The whole mixture was boiled in a vessel placed on a low- burner for one and half hours continuously. The rice husk comprises organic constituents – explicitly, hemi-cellulose, lignin, cellulose and mineral silica hoarded on the superficial surface of the rice husk epidermis.

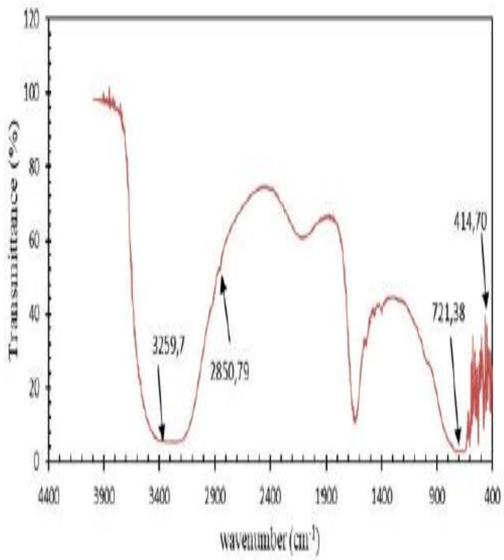
During the making of the agro-waste based super plasticizer, extreme -temperature alkaline hydrolysis that is available within rice husk with organic constituents occurs. Besides, this leads to leaching into the solution of some quantities of inorganic silica from the rice husk as dark-brown-colored solution, then sifted and collected in a vessel. This superplasticizer is specifically created for geopolymer systems as the ready-made or conventional superplasticizer lessens the engineering features of geopolymers.

(a) (b) (c)

Figure 4. (a) Alkaline activator (sodium silicate and sodium hydroxide), (b) laboratory-produced sodium silicate, and (c) Sodium hydroxide

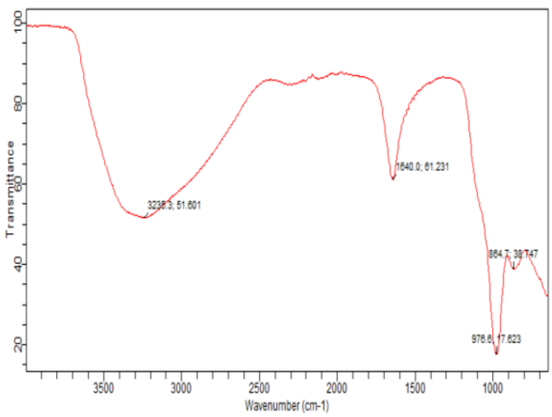
The graphic representation, raw materials and technique for this innovative material are described in Figure 4.

Rice-husk-created superplasticizer (Figure 5) is then scrutinized for categorization studies, such as BET, XRD and FTIR, to analysis the formation of various mineral levels, chemical connection and microstructure correspondently.




(a)

Sample ID:Na SILICATE ACTIVATOR
Sample Scans:30
Background Scans:16
Resolution:8
System Status:Good
Method Name:Transmittance
User:Admin
Date/Time:2021-06-11T08:23:07.582-07:00
Range:4000 - 650
Apodization:Happ-Genzel
File Location:C:\Program Files\Agilent\MicroLab PC\Results\Na SILICATE ACTIVATOR_2021-06-11T08-23-07.a2r




(b)

Figure 5. FTIR spectra of (a) sodium silicate from rice husk ash of NaOH 12M concentration, (b) ready-made sodium silicate

## RESULTS AND DISCUSSION

### Outcome of FTIR analysis

Fourier transform infrared spectroscopy (FTIR) characterization was utilized to ascertain the availability of various inorganic and organic bonds in the binders, alkaline activator, laboratory-produced sodium silicate and super plasticizer, and the spectrum obtained is displayed in Figure 5. Figure 5 illustrates the key absorbance band in the region between 950 and 3250 $cm^{-1}$. The combined vibrations are noticeable owing to the presence of various components. All the same, witnessed bands (within 1800 –980 $cm^{-1}$) were falteringly assigned in the IR investigation of kaolin clay, 3250 $cm^{-1}$ - 988 $cm^{-1}$ for RHA and 3200 – 950 $cm^{-1}$ for sodium silicate. The Si–O stretching vibrations were observed for Si-O stra, Si-O-Al stra., and Si-O stra, Si – O- Fe stra., displaying the ample amount of quartz [7][19]. Al-O-H (stra.), Al-)-H (inter-octahedral) and H-O-H stra. ascertain a sturdy band at 3695-8 $cm^{-1}$, 3623-4 $cm^{-1}$ and 3450·3 $cm^{-1}$ designate the leeway of the hydroxyl connection. Similarly, an all-encompassing band at 3450·3 $cm^{-1}$ and 1635.2 $cm^{-1}$ for H-O-H stra in the gamut of kaolin clay proposes the likelihood of water of hydration inside the adsorbent. The presence of bands at 2100 $cm^{-1}$, and 1800 $cm^{-1}$, for H-O-H stra., as well as Si-O-Si, Si-O str., likewise symptomatic presence of gypsum, while waves detected at 980 $cm^{-1}$ point toward the prospect of having hematite [4, 7, 10]. Hence, the outcomes of IR are quite supportive in the discovery of numerous kinds of minerals available within the sorbents utilized.

For silicate, the momentous peaks which were perceived at 615.8, 704.8 and 776.9 $cm^{-1}$ were ascribed to Si–O–Si together with O–Si–O bending vibrations of on site-synthesized inorganic silicate. Even though the intensities of their bands were small relative to the ones from the other bands, their existence confirms the in-situ materialization of sodium silicate through the reaction of sodium hydroxide on inorganic silica from rice husk. Then again, one strengthened band positioned at 1427.4 $cm^{-1}$, this was of vibrations of sodium carbonate and a band distinctive of O–C–O stretching, which was also assumed to be instigated by the action of sodium (Na) with atmospheric carbonate. Its great intensity was a sign of the extremely reactive character of sodium to form compounds, for instance sodium carbonate. The other lesser dispersed bands at 917 - 1001 and 1252 $cm^{-1}$tallied with Si–O broadening in silicate glasses as well as weak C–O stretching in natural cellulose, correspondingly.

Some physiognomies weak summits in the laboratory-produced superplasticizer range at the frequency section of 1654.8 $cm^{-1}$ were ascribed to quadrant ring stretching in lignin as well as carbonyl stretching.

The C=O stretching coining from organic fractions was available in the agro-waste based superplasticizer, but minor bands at 2307 and 2939 $cm^{-1}$ were linked with –CH stretching vibrations from both fructose and natural cellulose. These outcomes again confirm the XRD results and showed the disposition of inorganic and organic compounds in the agro-waste superplasticizer.

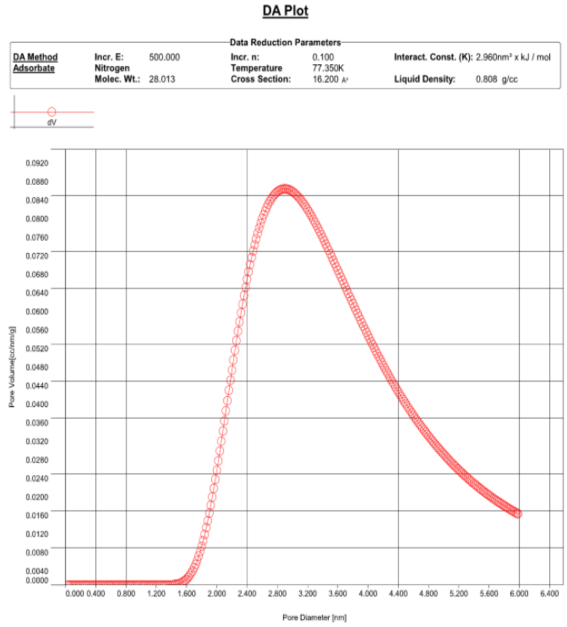
Likewise, FTIR scrutiny signified that the introduction of the agro-waste based superplasticizer to the GPC concrete lessened the viscidness and enhanced the flow manners of GPC, owing to the impact of the available organic fractions in the factory-produced super-plasticizer, which was imparted through alkaline absorption of rice husk. The range and peak intensity at 3259.9 and 2854.08$cm^{-1}$ reveal the hydrogen bonds, viz.; C-H and OH. This bond attested to the fact that with rising NaOH concentration, the intensity of the hydrogen bonds created also enhanced. Though, the materialization of hydrogen bonds was instigated by breaking the reminance of the amorphous fraction of the RHA. At this period, there was also a rising in the silica produced.

The peak intensity at 722.96 $cm^{-1}$indicated that Si-O-Si connection, which is the key component of sodium silicate, i.e Si-O-Si was distributed at 414.90 to 721.86 $cm^{-1}$ and this transpired in all sodium silicate yields. The practical group analysis exhibited that sodium silicate had been successfully manufactured and appropriate for use as alkaline activator in geopolymer concrete.

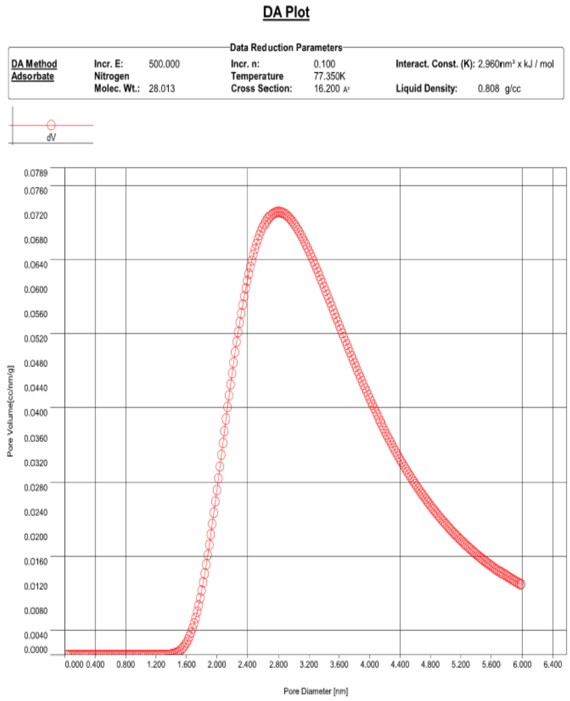
### Outcome of Brunauer-Emmett-Teller (BET) Analysis on Cement and Geopolymer Binders

Brunauer-Emmett-Teller (BET) scrutiny is a physical categorization technique that makes available quantifiable data about the porosity dispersal of solid materials and specific surface area. The process is fit for aeclectic range of solid matrices from catalytic agent powders to colossal materials.

Table 2 and Figure 6 shows that correlation coefficient of RHA (0.994) was higher than correlation of cement (0.991). Besides, RHA has a higher surface area (250.023 $m^3/g$) than cement (211.49 $m^3/g$) utilized. Though the c constant of cement exceeds the RHA values, RHA can serve as good pozzolanic material and cement substitute.




(a)




(b)

Figure 6. Brunauer-Emmett-Teller (BET) for, (a) RHA and (b) cement.

Table 2. BET outcome for cement and RHA

| | RHA | Cement |
|---|---|---|
| Slope | 9.99 | 13.28 |
| Intercept | 3.455 | 3.189 |
| Correlation Coefficient | 0.994 | 0.991 |
| C constant | 3.891 | 5.165 |
| Surface Area | 250.023 $m^3/g$ | 211.49 $m^3/g$ |

## Workability/Slump Test

Concrete workability is a very decisive factor; besides it is well-known to affect the compatibility, constancy, pumpability and flow capability of a concrete mixture. It also denotes an effort needed to influence a freshly manufactured concrete blended with least loss of homogeneousness. One of the main concerns peculiar to fly-ash-created geopolymers is their workability during on-site practical usage. In contrast to conventional concrete systems, geopolymeric material is disposed to relax fast, rendering extremely viscous unmanageable concrete mixtures. Conversely, the hindrance can be slightly resolved with the introduction of ready-made commercial super plasticizers, but highly handles with waste produced type– that contains, naphthalene, reformed lignosulfonates, polycarboxylates, and so forth, which are organic in nature and to a certain degree display compatibility with geopolymeric Si–O–Al–O set-ups. Similarly, workability is a most important disadvantage of fly-ash-created geopolymers owing to extreme viscosity, and none of orthodox superplasticizers was found operational in sustaining the viscosity of the mix. Thus, a new kind of superplasticizer was created to solve this workability concern. The waste created by superplasticizer was manufactured through rice husk as raw material. Categorization investigation via BET, XRF and FTIR of the material specified that the amorphous silica from rice husk reacts with the sodium hydroxide which is carefully chosen alkaline activator for this research displays that there is an in-situ synthesis of lignin and sodium silicate.

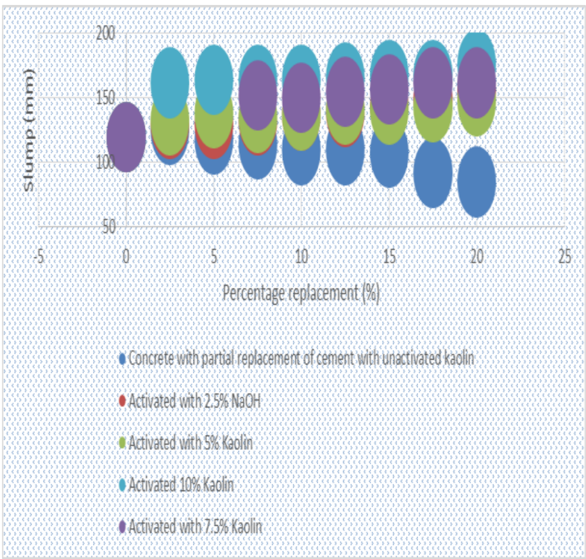



Figure 7. Disparity in slump gain through usage of various doses of GP concrete

The manufactured agro-waste superplasticizer was utilized in various proportions in fly-ash-created geopolymeric systems in order to ascertain its engineering physiognomies (Figure 7).

Evaluation of each binders utilized demonstrated that FA has a spherical particle shape, whereas RHA and KC have angular particle shapes, which have a direct effect on the workability as observed during the study; however, the large surface area and high porosity of the RHA particles could have affected the workability of GPC [5, 11, 13, 19]. The presence of intra-structural pores must have stored the water generated from geopolymerization process, making the mixture more cohesive; similar observation was found by Menya in their study (2018). The workability of the freshly prepared GPC was ascertained via the slump cone technique, and it is confirmed that the workability of the GPC was enhanced considerably compared to that of orthodox geopolymeric systems.

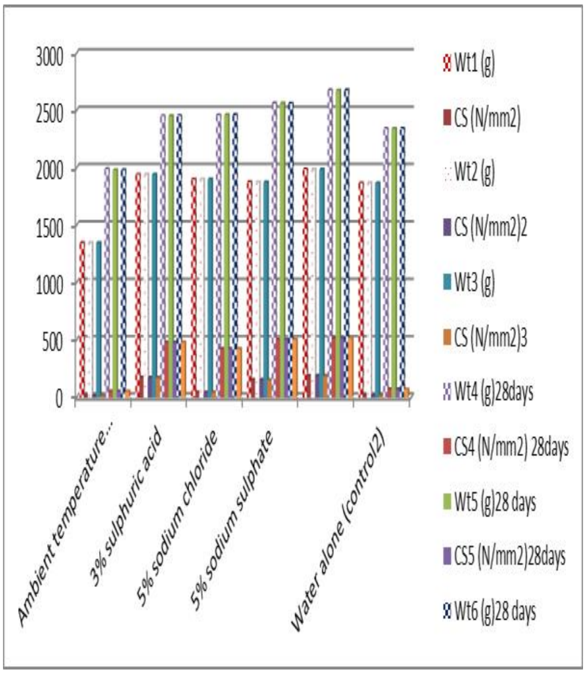
### Outcome of Durability Test

The results from immersion of geopolymer concrete specimens in four dissimilar chemical solutions: 5% sodium sulphate, 5% sodium chloride, 3% sulphuric acid, and 5% sodium sulphate plus 5% magnesium sulphate adopted based on Cong and Cheng [2] and Patankar et al., [11] findings, were presented in Figure 8.

Also, various chemicals used are displayed in Figure 9. Throughout the immersion time, the change in mass in compressive strength for 7 days shows that GPC soaked 5% magnesium sulphate + 5% sodium sulphate has highest weight of 1985.1g and crushing value of 187.0N/mm$^2$, followed by 5% sodium sulphate (1874.3g and 151.4N/mm$^2$), ambient temperature has lowest weight of 1345g and compressive strength of 13.7N/mm$^2$.

FA-RHA based GCP exhibits superior protection from the chloride solution, there was no deterioration found on the specimen surface in the presence of sodium chloride arrangements from early till 56 days. It was also observed that there was no significant change in mass and compressive strength. Whereas the change of mass was obtained by subtracting the mass of cube before immersion and mass after immersion.

The result of resistance to sulphate solution for the performance of FA-RHA based GPC subjected to aggressive chemical environments demonstrated that, the GPC was good resistant to sulphate environment. Indeed, after the introduction of these specimens for 56 days to 5% sodium sulphate solution, there was no deterioration to the surface, this is similar to the observation by Kong and Sanjayan [7], Muhammad *et al.,* [9] and Aggarwal et al., [17]. This outcome demonstrates that the use of superplasticizer GPC in the seawater zone construction will result in an excellent outcome. At the point when contrasted with OPC concrete, GC has incredible mechanical properties and solidness.




Wt – Weight ; CS – compressive strength; 1 – 3 for 7 days and 4-6 for 28 days.

Figure 8. Optimum of super plasticizer blended geopolymer at 7 and 28 days using laboratory-produced sodium silicate

(a) (b) (c)

Figure 9. (a)&(b) Absorption of geopolymer concrete inside 4 different chemicals, c) Different chemicals used for producing geopolymer concrete, Sodium hydroxide, hydrogen chloride and chemicals for durability tests

**Outcome of X-Ray Fluorescence (XRF) on Binder Materials**

X-ray fluorescence (XRF) shows the elemental/chemical composition of the materials. Table 3 and Figure 10 display the XRF results of cement, fly-ash, kaolin clay and the rice husk ash.

The result shows that cement consists majorly of calcium (II) oxide ($Ca(OH)_2$) at 62.5% followed by Silicon dioxide ($SiO_2$) at 24.5% and traces amount of Aluminum oxide ($Al_2O_3$) at 7.8%.

Fly-ash consists majorly of Silicon dioxide ($SiO_2$) at 52.11% and Aluminum oxide ($Al_2O_3$) at 23.59% with slight quantity of Iron (III) oxide ($Fe_2O_3$) at 7.39%. Kaolin clay consists majorly of Silicon dioxide ($SiO_2$) at 52% and Aluminum oxide ($Al_2O_3$) at 35% with slight quantity of Potassium oxide ($K_2O$) at 2%. Kaolin clay on the other hand consists majorly of Silicon dioxide ($SiO_2$) at 93.23% and Potassium oxide ($K_2O$) at 3.40% with slight quantity of $P_2O_5$ at 2.05%.

Based on the proximity of silicate and alumina compositions obtained showed that kaolin used has silicate and aluminate levels near other kaolin utilized by other researchers such as Faluyi *et al.,* [3], Dewi et al., [13], Arum *et al.,* [18], although there are still many other materials such as $K_2O$, $Fe_2O_3$, $TiO_2$, MgO, $SO_3$ etcetera in lesser contents.

Table 3. X-ray fluorescence (XRF) values of various aluminosilicates material and ashes

| **Chemical Composition (%)** | **PC** | **FA** | **KC** | **RHA** |
|---|---|---|---|---|
| Silicon dioxide ($SiO_2$) | 24.5 | 52.11 | 52.00 | 93.23 |
| Potassium oxide ($K_2O$) | 0.67 | 4.08 | 2.00 | 3.40 |
| Aluminum oxide ($Al_2O_3$) | 7.8 | 23.59 | 35.00 | 0.03 |
| Titanium dioxide ($TiO_2$) | 0.04 | 0.88 | 0.90 | 0.1 |
| Iron (III) oxide ($Fe_2O_3$) | 0.5 | 7.39 | 1.00 | 0.16 |
| Calcium oxide ($Fe_2O_3$) | - | 2.61 | <0.05 | 1.03 |
| Magnesium oxide (MgO) | 2.5 | 0.78 | 0.70 | 0.18 |
| $Na_2O$ | 0.42 | 0.42 | 0.05 | 0.02 |
| $SO_3$ | 2.11 | 0.49 | 0 | 0.25 |
| $P_2O_5$ | 0.24 | 1.31 | 0 | 2.05 |
| Loss in ignition (LOI) | 0.82 | 3.21 | 0.09 | 0.08 |
| Fineness: % retained on 45 /70 mm sieve | - | 27.9 | - | 70.3 |
| Specific gravity | 3.12 | 2·4 | 2.58 | 2.08 |
| Blaine's specific surface area ($m^2/kg$) | 349.8 | 359.8 | - | 321.5 |
| Density ($Kg/m^3$) | 2350 | 740.5 | - | 158.5 |

Note: Fly ash (FA); Kaolin clay (KC); Rice husk ash (RHA).

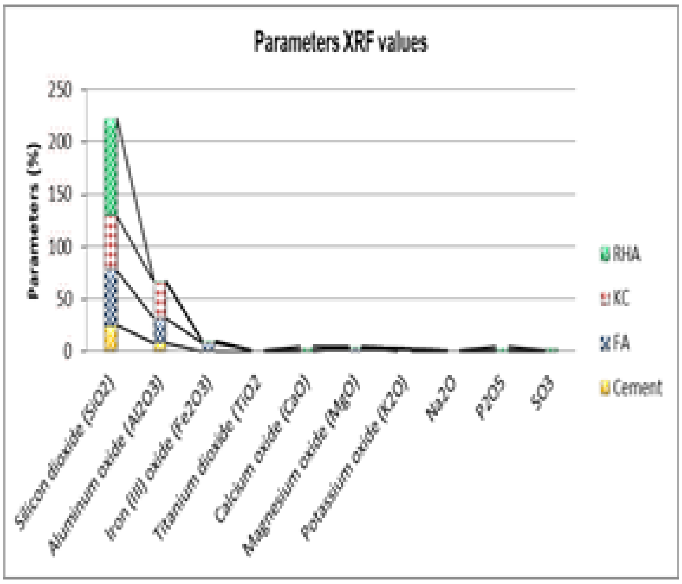


Figure 10. XRF drawing shows the chemical features.

These compositions are in agreement with the result of FTIR, which shows that the chief elements of the functional group for the binders are alumunium, silicon and iron. These compares displays that the selected pozzolanic binders can be used instead of cement, either for partial or total replacement.

## CONCLUSION

This paper discussed the outcome of experimental analysis carried out on conventional and laboratory-produced super plasticizer created geopolymers concrete. Fourier transform infrared spectroscopy (FTIR) characterization illustrate the major absorbance band at the region between 950 and 3250 $cm^{-1}$.Some characteristic weak peaks in the laboratory-produced superplasticizer spectrum at the frequency region of 1655 $cm{-}1$ were attributed to quadrant ring stretching in lignin and carbonyl stretching including C=O stretching originating from organic fractions present in the factory-produced superplasticizer, whereas small bands at 2309 and 2940 $cm^{-1}$ were associated with –CH stretching vibrations from fructose and native cellulose. Throughout the exposure period, the change in mass in compressive strength for 7 days shows that GPC soaked 5% magnesium sulphate + 5% sodium sulphate has highest weight of 1985.1g and crushing value of 187.0N/$mm^2$, followed by 5% sodium sulphate (1874.3g and 151.4N/$mm^2$), ambient temperature has lowest weight of 1345g and compressive strength of 13.7N/$mm^2$. Likewise 28 days immersion followed the same trend. The XRD spectrum of binders materials demonstrated that fly ash contains silicon dioxide, aluminum oxide (3Al2O3·SiO2) and iron (III) oxide as minor phases, while kaolin clay have kaolinite, illite, coesite, garnet, muscovite, nacrite and vermiculite in that order and the major mineral composition of rice husk ash are quartz, garnet, spinel, hanksite, lime, muscovite and clinochlore in that order. Thus, the superplasticizer of 1·5% by weight of fly ash proved to be optimum for fly-ash-based geopolymer concrete. Also, inclusion of 7.5% kaolin clay and 10% RHA to 5% fly ash based geopolymer concrete can enhance the early compressive strength of concrete.

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