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Durability and Microstructural Performance of Metakaolin-based Geopolymer Concrete under Elevated Temperature Exposure.

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Abstract
Concrete materials exposed to harsh environment usually experience changes in the microstructure and physical appearance. Geopolymer concrete specimens of grade 20 N/mm^2 was targeted at a mix ratio 1:2:4 for metakaolin, fine and coarse aggregate respectively and the alkaline liquid ($NaOH:Na_2SiO_3$) which key start the geopolymerisation process was varied from 1:1 to 1:4. The water content of the Na_2SiO_3 was found to be 40.80 % with a pH value of 12.8 and oxide value of 3.89.This implies that, the commercial sodium silicate use in this study exhibited acceptable pH and silica modulus for geopolymerization. The XRD pattern of the unheated (MKGPC) sample exhibits the characteristic peaks of conventional metakaolinite functions at Bragg's angles of 11.44, 12.72, 20.45, 25.28, 27.02, 35.88, 38.83, 55.58 and 62.37°. The peak at 12.72° was due to present of the undisturbed crystalline silica in the analyzed sample. The XRD pattern shows a highly amorphous characterization with no sharp peaks for the heated sample. The SEM micrograph shows some changes in the microstructure as the sample transform from crystalline to amorphous for the heated geopolymer concrete. The sample was inserted in a furnace and the temperature was elevated from100^{O}C to 1400^{O}C at a varying period ranging from 2 to 8 hours in accordance with BS EN 12390-2. The optimum mix ratio of 1:3 gave the highest compressive strength, for 12M concentration of (NaOH), there was no significant changes in the compressive strength of 30 N/mm^2 obtained at 200 ^{0}C until at a temperature of 500^{0}C after which it continue to drop. However, at lower and higher temperature ranging from 100^{O}C to 400^{O}C and 1000^{O}C to 1400^{O}C respectively, the compressive strength was constant as it shows no significant effect even at hourly increase in elevated temperature ranging from 2hrs to 8 hours. More so, the density loss was constant at lower temperature and decreases as the temperature increases.

Keywords: purposes. Geopolymer concrete, elevated temperature, metakaolin, density, XRD, durability.

1. Introduction

Concrete structural members are usually designed to accommodate different or various environmental problems categorized from mild to a worst-case scenarios. That is, severe conditions. Fire is reported to be categorized among one of the most environmental issues that affect concrete or structural members, such as, tunnels which have very close structural conduits (Di *et al.*, 2021). Concrete when exposed to harsh environment (elevated temperatures) will experience physical, chemical and mechanical changes. Physical changes include; change in coloration, surface cracking and Spalling of concrete cover that is, expulsion of some portions of concrete from the surface, degradation of the compressive strength, thermal expansion and chemical dehydration of concrete. Also the chemical changes result to the breakdown of ions responsible for binding the material constituent together and changing the structural morphologic. When all these mechanisms are understood, it gives an overview to the nature and extent of the concrete deterioration or degradation.

Geopolymer is considered as an alternative material to Portland cement owing to its benefits in terms of environmental protection and energy conservation since 43% - 59% energy reduction is achieved during production as compared to conventional concrete (Thamilselvi, *et al.*, 2017) in addition to the excellent durability resistance. Metakaolin is used in this research as the source material and according to (Yetunde *et al.*, 2019), metakaolin is obtained by heating kaolin, a natural, finely divided, alumina siliceous mineral and it is abundant in large deposit in Nigeria. One of the most

weighty constituents material in geopolymer concrete mixed formulation are the silicates to alumina ratio constituents of metakaolin, which is anticipated to be in the range of 1.5 to 5.08% upon mixing to form geopolymer solution (Bello *et al.,* 2023). Matakaolin is produced in a control (temperature) manner to refine its colour and remove residual impurities so as to achieve higher degree of purity and to enhance pozzolanic reactivity (Yetunde *et al.,* 2019).

However, many researchers investigate the chemistry of geopolymers paste while some studies the structural performance of geopolymer concrete elements when exposed to service loading, which includes; slabs, beams, columns, steel fibre reinforced geopolymer concrete, high performance geopolymer concrete and material properties of geopolymer but there are fewer study or research on the microstructure and durability performance of metakaolin based geopolymer concrete subjected to harsh environment (elevated temperature).

2.0 Materials and Methods

2.1 Materials

The various materials adopted during this research work were sodium silicate (Na_2SiO_3), Sodium Hydroxide (NaOH) of analytical grade England, portable water source form the department of Civil Engineering, kaolin obtain from Kankara in Kastina State, metakaolin by the calcination of kaolin which was done at Chemical Engineering A.B.U Zaria, fine aggregate and coarse aggregate all source from Zaria.

2.2 Methods

2.2.1 Preparation of geopolymer concrete samples.

The mechanical test performed on the geopolymer concrete is compressive strength test. Also functional group characteristics of the hardened geopolymer concrete at 56 days using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) were also performed. Approximately about 336 samples of geopolymer concrete cubes of size 100 mm by 100 mm was casted by mixing the metakaolin content and alkaline liquid solution ($NaOH:Na_2SiO_3$). The effect of change in molar concentration and the alkaline liquid ration was checked in order to obtain a homogenous mix. The molar concentration of 12M (NaOH) was used and the alkaline liquid ratio was varied from 1:1 to 1:4. The Alkaline liquids was prepared and mixed in the required proportion while allowing it to rest for 24 hours. The fine and coaurse aggregate, metakaolin and alkaline liquids with the required additional water was mixed in an electric concrete mixer and later placed in the mould and compacted using an electric table vibrator per BS EN, 12390-2 (2019).



Plate 1. Preparation of geopolymer concrete samples

2.2.2 Curing of geopolymer concrete.

Oven curing was carried out after 24 hours of casting and then de-mould and insert in a polyethylene bags to enhance the polymerization process which result to higher compressive strength. The samples were then put in an electric oven at $60^{O}C$ for the next 24 hours after which is allow to rest for the next curing days and was carried out in accordance with BS EN, 12390-2 (2019).



Plate 2. Oven cured samples

2.2.3 Compressive strength properties.

The compressive strength tests was carried out based on BS EN 12390-2 (2019) on concrete cubes (100mm × 100mm) specimens for 12M molar concentration of (NaOH) with the aid of universal testing machine of 2500kN maximum capacity. The tests were performed at 7, 14, 28 and 56 days curing period for both ambient and oven cured. From the result obtain after 56 days of curing for both ambient and oven cured, the 12M concentration of (NaOH) at a mix ratio 1:3 for oven cured gave a better strength performance which was subjected to elevated temperature to check it durability performance.



Plate 3. Compressive strength test in progress



Plate 4. Crushed geopolymer concrete cube

2.2.4 Physical appearance of geopolymer concrete after exposure to elevated temperature

It could be seen from plate 7 below that physical changes such as change in coloration, yield lines and or minor cracks and spalling of concrete cover occur as a result of high thermal stress the geopolymer concrete undergoes. Also expulsion of some portions of the concrete from the surface and gradual degradation of the compressive strength was recorded.

2.2.5 Compressive strength analysis of the impact of elevated temperature on the geopolymer.

The geopolymer concrete cube sample of size 100 × 100 mm were insert into the oven and the temperature was elevated to the desired ^{O}C at various hourly increase in temperature and then allow to cool. After which the compressive strength analysis of the heated (elevated temperature) geopolymer concrete was done at a temperature ranging from 100 ^{0}C to 1400 ^{0}C.

$$RCS = {}^{F_L}/_{A} \qquad 1$$

Where RCS is the Residual Compressive Strength. (N/mm^2), F_L is the failure load of the heated sample subjected to elevated temperature. (N), A is the area of geopolymer concrete cube subjected to elevated temperature. (mm^2).



Plate 5. Control samples



Plate 6. Samples in furnace



Plate 7. Samples after subjected to elevated temperature.

2.2.6 XRD analysis of the harden geopolymer concrete.
X-ray diffraction (XRD) test was carryout on the geopolymer concrete to determine the crystalline nature of the material, arrangement of atoms, lattice parameters and phase analysis. The upper and center parts of the specimen is cut into a 10mm disc, then it is crush and grind into powder form. It is then place in the XRD machine with monochromatic radiation by scanning the 2 angle from 10 to 70. The sample and detector or tube are move in other to change the diffraction angles, after which the intensity is measured and the diffraction data are recorded.

2.2.7 SEM analysis of harden geopolymer concrete.
Scanning Electron Microscopy (SEM) was used to determining the structural morphology of the geopolymer concrete for the heated (elevated temperature) and unheated specimen. The samples were cut to the size of the sample holder, which is 10 mm by 10 mm standard. The samples were inserted in Quorum (Q150R ES) machine where the specimen was coated with a thin layer of gold. Thereafter the specimen were view via a monitor connected to the machine, where different magnification where capture, in which 100 µm was selected. The operating voltage used was 10 kV.

3.0 Results and Discussion.
This section entails the presentation and discussion of results obtained during the analysis.

3.1 Physical properties of the commercial sodium silicate.
It was analyzed and verified that, the silicate tested was viscous and whitish to greenish in colour and a musty odour which is similar to what was reported in the work of (Aliyu *et al.*, 2019). Also, the water content of the sodium silicate was obtained as 40.80 % while the pH value obtain fall with the strong alkaline medium recorded as 12.8. Therefore, the commercial sodium silicate obtained is good for its intended purpose owing to the physical properties it exhibited. These were further justified from the oxides value of its silicate to sodium which was recorded as shown in Table 1 to be 3.89.

Table 1: Properties of the Sodium Silicate Gel/Paste

S/No	Property	Value
1	Nature	Viscous Paste
2	Colour	Whitish to greenish
3	SiO_2:NaO	3.89
4	Water Content	40.80 %
5	Odour	Musty
6	Ph	12.8

3.2 The beneficiated kaolin (Metakaolin) for geopolymer formulation feedstock
Table 2 presents the analyzed results of the beneficiated kaolin to form metakaolin. The significance of this beneficiation process is to reduce the impurities in the raw kaolin and makes it pure as much as possible for effective performance in the geopolymer concrete formation.

Table 2: Chemical composition of the metakaolin (beneficiated kaolin)

Chemical Oxides (%)	SiO_2	Al_2O_3	CaO	Fe_2O_3	M_gO	SO_3	TiO_2	K_2O	Na_2O
% composition	57.21	36.71	4.69	0.69	0.02	0.00	0.091	0.06	0.11

As shown in Table 2, the amount of impurities such as SO_3, MgO and K_2O have reduced from 0.42, 0.13 and 0.26 % to 0.00, 0.02 and 0.06 % respectively, which gave rise to increase in silica oxide content from 54.9 to 57.21 % and subsequently, increased the silica to alumina ratio from 1.37 to 2.55 which is within the range (2-2.65) for a good compressive strength as reported in the works of (Bashar *et al.,* 2019; Hengels *et al.,* 2021).

3.3 Effect of 12M Concentration of (NaOH) at Ambient and Oven Cured Conditions on Geopolymer Weight

Figure 1, shows the effect of oven and that of ambient cured condition on the weight of the developed geopolymer concrete for 12M concentration (NaOH) of mix-ratio (NaOH: Na_2SiO) 1:1, 1:1.5, 1:1.2, 1:1.2.5, 1:3, 1:3.5 and 1:4 respectively. As shown in the figure, the initial weight of 2.55 kg was reduced to 2.37 and 2.33 kg against ambient and oven cured condition respectively after 7-days for mix ratio formulation 1:1. Furthermore, at the end of the 14th day, the weight of both oven and ambient cured conditions reduced to 2.33 kg in each case, which gradually reduced to 2.3 kg at day 28 as shown in Figure 1. More so, it was observed that, there was no further decrease in the weight as they remained at 2.3 kg at the end of the 56th day curing.

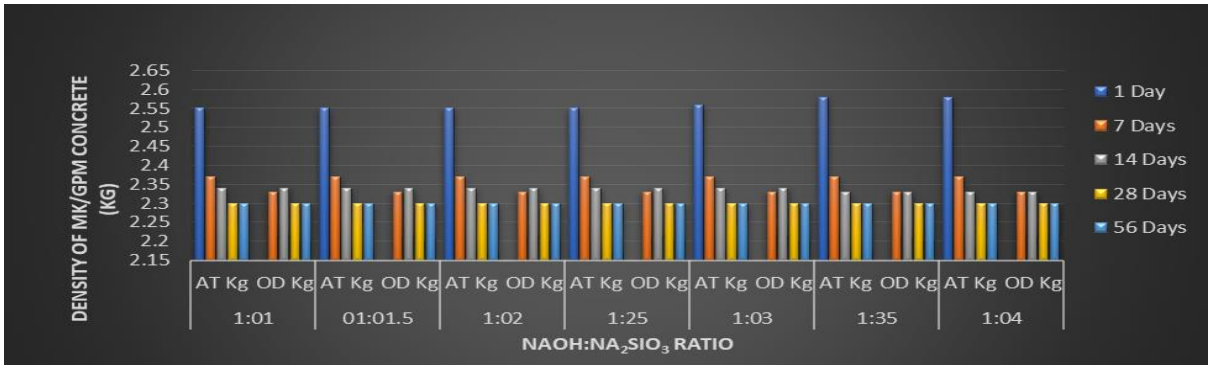


Figure 1: Effect of Weight Comparison at Ambient and Oven Cured Condition for 12M Concentration varying ($NaOH:Na_2SiO_3$) mix ratio.

Additionally, similar trends were observed in the mix-ratio 1:1.5, 1:2, 1:2.5, 1:3, 1:3.5, and 1:4 as depicted in Figures 1. The stability in weight of the developed geopolymer concrete despite variations in oven and ambient cured conditions after the 14th day of the study can be attributed to several factors. Firstly, geopolymer concrete undergoes a curing process involving chemical reactions between the alkali activators and the precursor materials, leading to the formation of a hardened matrix. However, it was observed that, after about the 14th day of the investigation, these reactions may reach completion, resulting in a stabilized weight. This is also in a close agreement with the works of (Enes, 2023; Ahmet *et al.,* 2023).

Similarly, it has been established literature (Ahmet *et al.,* 2023) that, at the initial curing period, significant changes occur in the internal structure of the concrete as its densifies and bonds strengthen, thereby once the internal structure is established, further weight changes may be minimal. Moreover, as reported by (Ahmet *et al.,* 2023). More so, moisture content within the geopolymer concrete may fluctuate in the early curing stages due to evaporation, hydration, or ambient cured conditions. However, after a certain point, the concrete reaches a moisture equilibrium where the rate of moisture loss equals the rate of moisture absorption, resulting in a stable weight. Additionally, the internal microstructure of geopolymer concrete evolves during curing, leading to the development of a more compact and stable material. Based on these, once the microstructure reaches a certain level of maturity, further changes may be negligible.

However, these factors collectively contribute to the observed phenomenon of the weight of the developed geopolymer concrete remaining unchanged despite variations in curing conditions after the 14th day of the study. These changes

may be due to intermediate interactions of the variable in the mix with the temperature profile as also reported by (Bashar *et al.*, 2019).

3.4 Effect of Alkaline Liquid Ratio on the Compressive strength of Geopolymer for 12M concentration (NaOH). Figure 2 shows the effect of 12M **(NaOH)** concentration on the compressive strength of metakaolin based geopolymer concrete. However, higher compressive strengths were obtained for oven cured samples at a mix ratio 1:3 formulation. This is evidence from the initial compressive strength value of 21.2 and 22.1 N/mm^2 obtained on the 7th day at ambient and oven cured temperature conditions respectively. The compressive strength of the geopolymer concrete increased at the end of the 14th day to 24.2 and 24.4 N/mm^2 for ambient and oven cured temperature conditions respectively as shown in Figure 2.

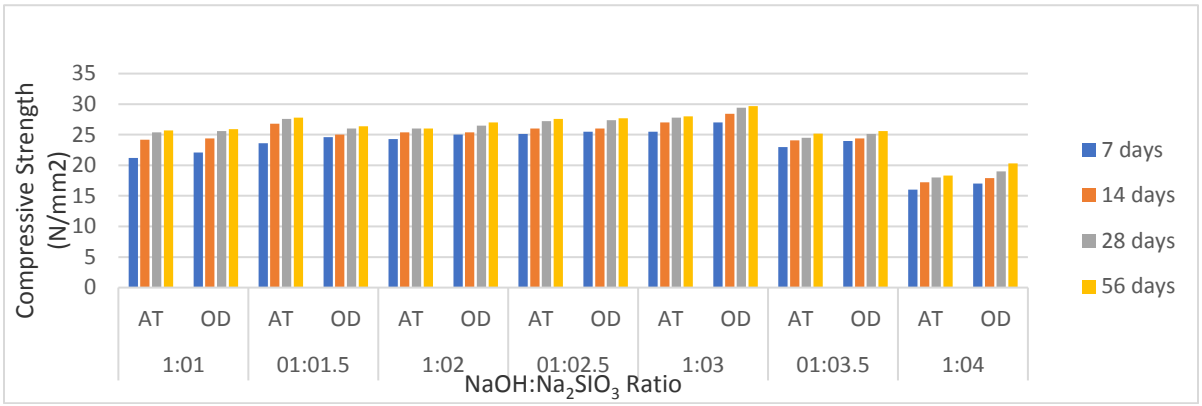


Figure 2: Relationship between Compressive Strength and Alkaline Liquid Ratio (NaOH:Na_2SIO_3) at Various Curing Age for 12M **(NaOH)** Concentration.

Furthermore, the compressive strength of the material generally increased as the curing age increased against the respective mix ratio 1:1.5, 1:2, 1:2.5, 1:3, 1:3.5 and 1:4. However, as it was also observed from the figure 2 for 12M concentration formulation, the highest compressive strength of 29.7 N/mm^2 was obtained at the optimum mix ratio of 1:3.

3.5 Effects of elevated Temperatures on the Compressive Strength of Geopolymer concrete for 12M concentration of (NaOH) at 56 days curing period.

The effects of varying elevated temperatures on the compressive strength of geopolymer materials is depicted in Figure 3.

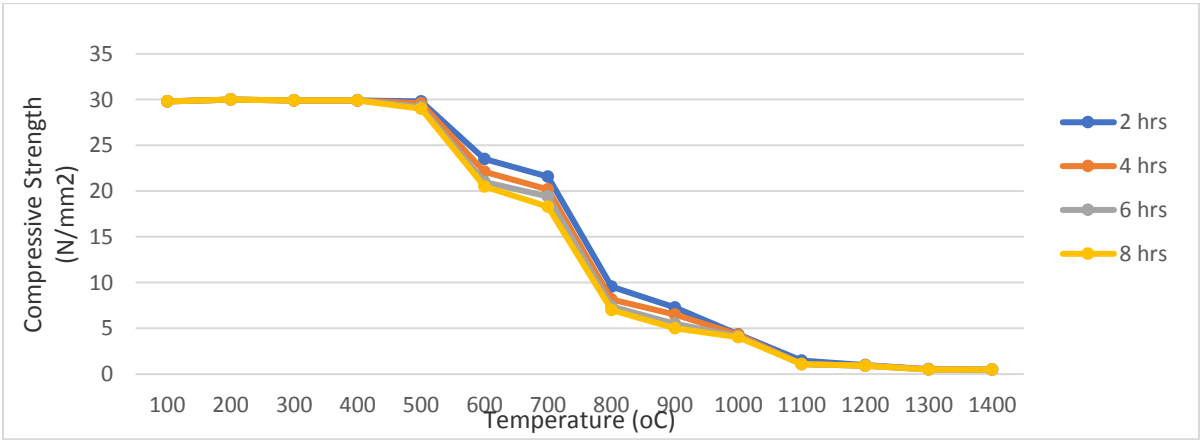


Figure 3: Effect of hourly change in elevated temperatures on the compressive strength of geopolymer materials.

As shown in Figure 3, the initial compressive strength of the geopolymer materials tested at $100^{O}C$ elevated temperature for a period of 2, 4, 6 and 8 hours is 29.70 N/mm^2. Additionally, as the elevated temperature was increased to 200, 300, $400^{0}C$ the compressive strength remained (constant) unchanged as the value recorded all through was 29.70 N/mm^2 for the whole chance in time investigated as shown in Figure 3. However, there was a noticeable change in the compressive strength of the materials at an elevated temperature of $500^{0}C$ as the compressive strength of 29.6 N/mm^2, 29.3 N/mm^2, 29.2 N/mm^2, and 29 N/mm^2 were recorded for change time of 2, 4, 6 and 8 hours respectively, indicating that the increase in the hourly change in elevated temperature at this stage was slightly significant, which is consistent with the report of (Kwabena and Morteza, 2023). The compressive strength of the material as shown in Figure 3 was observed to have further decreased to 23.5 N/mm^2 at an elevated temperature of $600^{O}C$ for change in time of 2 hours. However, the compressive strength changed throughout the hourly change in time of 4, 6 and 8 hours as shown in Figure 3.

Furthermore, at $800^{O}C$ elevated temperature, the compressive strength values at 2, 4, 6 and 8 hours significantly drop further to 9.55, 8.14, 7.40 and 7 N/mm^2 respectively and this might be attributed to the breakdown of the aluminosilicate ion responsible for binding the material constituent for the geopolymer concrete. Also, a continuous degradation of the compressive strength was recorded at the operating calcination temperature of $1000^{O}C$. At temperatures ranging from $1100^{O}C$ to $1400^{O}C$, the compressive strength values converge to almost zero and constant as the value obtained are less than 0.5 N/mm^2.

However, it can be deduce from the chart that at lower and higher temperature ranging from $100^{O}C$ to $400^{O}C$ and $1000^{O}C$ to $1400^{O}C$ respectively, the compressive strength is constant as it shows slight or no significant effect even at hourly increase in elevated temperature. Also, the hourly increase in elevated temperature is only effect at moderate temperatures ranging from $500^{O}C$ to $900^{O}C$ as variations in the compressive strength were record as shown in the figure 3. This is consistent and in good agreement with the work of (Bashar *et al.*, 2019; Hengels *et al.*, 2021; Alida *et al.*, 2021).

3.6 Effects of developed density loss on the geopolymer concrete after exposure to elevated temperature for 12M at 56 days curing period.

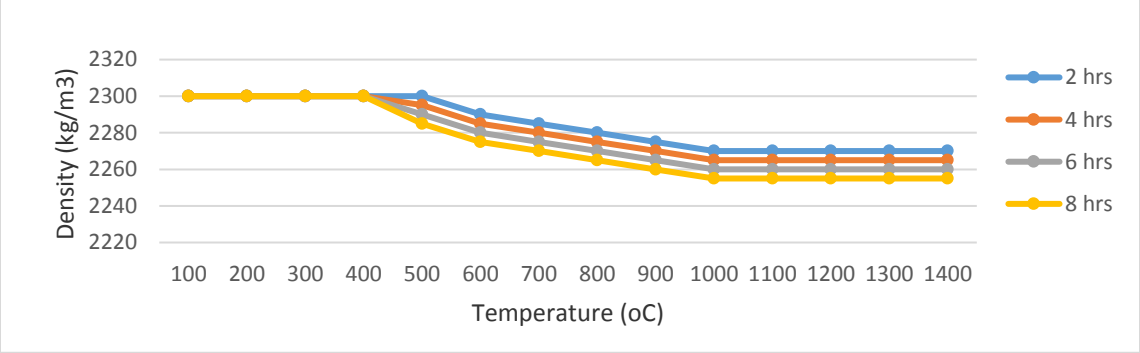


Figure 4: Relationship between Density and Temperature at Various time duration for 12M (NaOH) Concentration.

In this figure, the effect of hourly change in elevated temperature on the density of geopolymer concrete specimens as well as the mechanical were checked. It could be observed that a constant trend was seen at an elevated temperature ranging from $100^{0}C$ to $400^{0}C$ and this shows that there is a stable microstructure formation present in the specimen, while a significant drop or loss of density was recorded at temperature ranging from $500^{0}C$ to $900^{0}C$. The significant drop in density is attributed to the expansive of the specimen which break down the ionic bond responsible in binding the mineral constituent together. Also at higher temperatures ($1000^{0}C$ to $1400^{0}C$) the density remains constant. This is a clear indication that the presence of impurities and voids present in the specimen must have been dissipated. The change in the hourly elevated temperature is less effective on the density at low temperature but more significant and pronounce at high temperature as shown in the figure 4.

3.7 Structural and Morphological Properties of the Geopolymer Concrete.

X-ray diffraction (XRD) and scanning electron microscope (SEM) were carried out on the unheated and heated geopolymer concrete specimens developed to determine the effects of hourly change in elevated temperature in terms of structural and morphological properties respectively.

3.8 XRD analysis of unheated geopolymer concrete specimen (control) for 12M.

The XRD analysis of the geopolymer concrete specimen before subjected to elevated temperature of 800^{O}C is shown in Figure 5. It was observed that, the XRD pattern exhibits the characteristic peaks of conventional metakaolinite functions at Bragg's angles of 11.44, 12.72, 20.45, 25.28, 27.02, 35.88, 38.83, 55.58 and 62.37°. The peak at 26.67° was due to present of the undisturbed crystalline silica in the analyzed sample, which is in similar agreement when compared to the work of (Tuan *et al.*, 2021). The high intensity peaks shown in Figure 5 signifies the present of stable structure as it has not been subjected to elevated temperature which may distort or affect its orientation. The lower brag angle peaks shown in Figure 5 indicate the presence of other composition such as MgO, Na_2O, Fe_2O_3 K_2O and water contents at lower compositions. This is justified from the intensities shown at peaks Bragg's angles of 12, 20, 25, and 35˚. Thus, it was observed that, the peaks had broadly lower intensities compared to the silica peak, which shows that the unheated sample contained high concentration of crystalline silica also known as quartz.

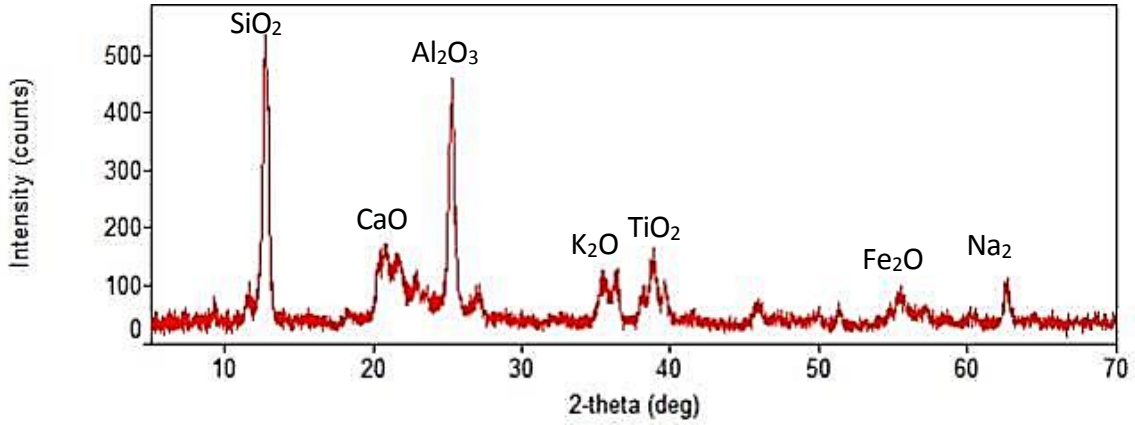


Figure 5: The XRD result of unheated geopolymer concrete sample (control) for 12M

3.9 XRD analysis of the heated (elevated temperature) specimen effect on the developed geopolymer concrete.

Figure 6 depicted the XRD analysis of the heated geopolymer concrete specimen at a temperature of 800^{O}C and an hourly change in elevated temperature time of 8 hours as shown in the figure 6, the XRD pattern shows a highly amorphous characteristic. Also, it was observed from Figure 6 that, no sharp peaks were present in the formation as when compared to the peaks observed in Figure 5 for the unheated geopolymer concrete sample. These observations proved that, the elevated temperature has significant effect on the geopolymer concrete, changing its orientation from crystalline nature to amorphous, which is in a good agreement with the work of (Rajini, 2021). It also indicate the deterioration of the major elements responsible for strengthening the geopolymer concrete. As shown in Figure 6, the major peak present in the sample was observed at Bragg's angles of 25.25^{O}, which could be due to the presence of residual strength and changes in the orientation of the formulation due to destabilization in the microstructure as a result of the elevated temperature.

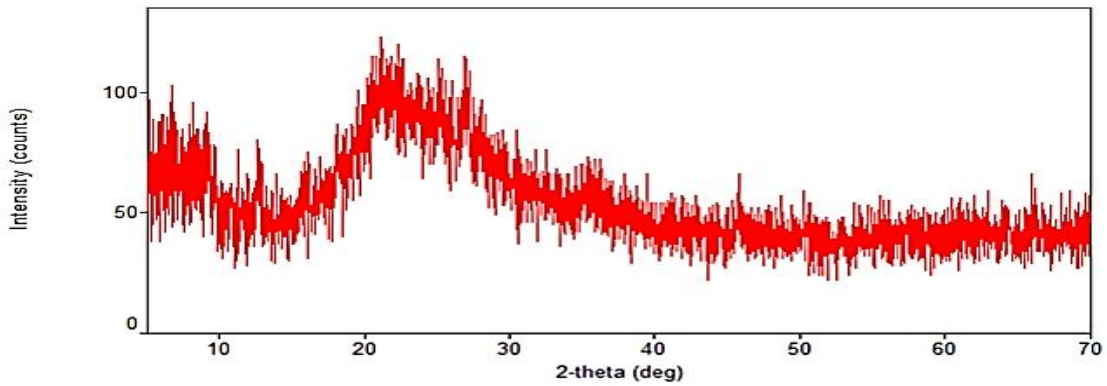


Figure 6: XRD analysis of the heated geopolymer concrete specimen for 12

3.10 Morphological property of the geopolymer concrete

Figure 7 depicts the scanning electron micrograph of the heated geopolymer concrete developed at a magnification of 7,000x. The micrograph shown in the figure 7 shows the morphological properties of the geopolymer concrete, in which the microstructural components in the formulation were coarsely dispersed as a result of the breakdown of the aluminosilicate bond, thereby resulted in the change of chemical composition. It also reveals a highly porous structure with larger voids and rough surfaces.

However, dispersed microstructure is usually characterize with a lower strength degradation due to the compelling exposure to harsh (elevated temperature) environments. As shown in Figure 7, the geopolymerization reaction as a result of the temperature effect is demonstrated, as evidence from the uniformly dispersed silicate grains that are clearly visible in the SEM image. This is due to the presence of residual minerals such as silica and alumina content combine as the two main constituents of geopolymer concrete formation.

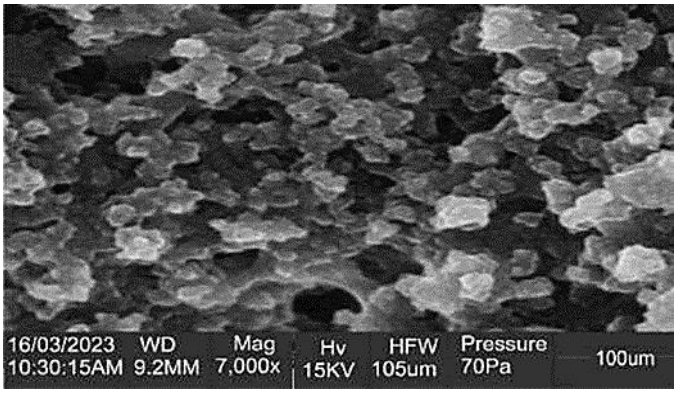


Figure 7: SEM micrograph of the heated geopolymer concrete at a magnification of 7000×.

More so, Figure 8 shows the morphological properties of the unheated geopolymer concrete at a magnification of 8,000x. As shown in the figure, the surface agglomeration was not dispersed when compared with the heated (elevated temperature) sample as shown in Figure 7, which differ significantly in the degree of bond formation and dispersed of the chemical constituents as a result of geopolymerization reaction. It shows larger agglomerates with a smoother surface, with little visible voids and more consolidated and compacted structure. This clearly indicate that, the sample has a more compact morphology at this scale, which enhance the material's compressive strength. The SEM micrograph depicted in Figure 8 shows no change in the microstructure when compared to the heated geopolymer concrete due to its exposure to temperature effect. The trends observed in these investigation are similar to the work of Marta and Mateusz, (2021).

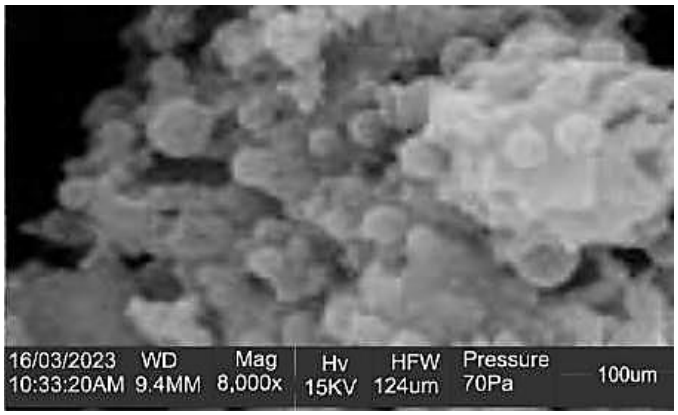


Figure 8: SEM analysis of the unheated geopolymer concrete at a magnification of 8000×.

4.0 Conclusions

The following conclusions were drawn based on the outcome of the results carried out.

The best strength properties or performance of the compressive strength is achieved at 29.7 N/mm^2 at an oven cured condition. However, the compressive strength increased with increase in the sodium silicate content at a mixed ratio 1:3.The maximum compressive strength increases to 30 N/mm^2 at the elevated temperature of 200^{O}C at best operating time of 2 hours was recorded. The compressive strength is slightly constants at lower and higher elevated temperatures and it shows little or no variability in the hourly increase in time change. More so, the hourly rise in temperature is only significant at moderate temperature. Density loss remains constant at lower temperatures but decreases at higher temperatures. It can also be concluded based on the microstructural analysis that, the elevated temperature have effects on geopolymer concrete as it transformed from crystalline form to amorphous, which was confirmed from the XRD and SEM analysis.

5.0 Recommendation

It is recommended that, basalt fibres may mitigate strength loss at >600°C due to their high thermal stability which might also resist cracks of the geopolymer concrete materials.

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