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METAKAOLIN BASED GEOPOLYMER CONCRETE MIX DESIGN SUITABLE FOR REINFORCED CONCRETE USE

***Aliyu, I., Kaura, J. M., Abubakar, I. and Ocholi, A.**

Department of Civil Engineering, Ahmadu Bello University Zaria.

*Corresponding Authors email: ibrahmaliyu@abu.edu.ng (+2348036397717)

ABSTRACT
Geopolymer concrete (GPC) has no mix design code to guide in the choice of material proportions for specific compressive strengths, its properties have not been fully understood especially in Nigeria despite being blessed with abundant geopolymer Al-Si source material (Kaolin). This study presents the properties of Kankara kaolin before and after treatment. Minitab 18 factorial design was used to design a Metakaolin based Geopolymer Concrete (GPC) with target compressive strength of 25 N/mm^2 (Suitable for Reinforced concrete). After kaolin treatment XRF results indicate a marginal difference in Oxide composition with the kaolin before treatment, but FTIR results showed more of the difference because the transmittance of the band between 3619 and 3690 cm^{-1} which is typical of kaolin increased when treated to 94.484 % from 74.825 % when untreated and quartz interference at 1114.5 cm^{-1} and Si-O quartz at 674.6 and 752.9 cm^{-1} were no more after the treatment. A factorial design with four factors having 9 experimental runs recoded 39.5 N/mm^2 (Run No 7) as highest compressive strength and 4.1 N/ mm^2 (Run No 2) as the lowest. Results indicated that mixes with low alkaline liquid and low Molar concentration content exhibits lower strength than those with high alkaline liquid content and High Molar concentration. Re-analysing laboratory results and targeting a strength of 25 N/mm^2; a mix (M1) with 14M proposed for the validation attained 38.7 N/mm^2, another mix (M2) with 8 M also attained 30 N/mm^2, Both M1 and M2 are satisfactory going by ACI 211.4R.93 1998.

***Keywords*:** Compressive Strength, Minitab 18, Metakaolin, Factorial design, FTIR, XRF.

INTRODUCTION

Concrete is the most abundant manmade material in the world. One of the main ingredients in a normal concrete mixture is Portland cement. However, the production of cement is responsible for approximately 5% of the world's carbon dioxide emissions (Ernst et al, (2001)). In order to create a more sustainable world, engineers and scientists must develop and put into use a greener building material (Mathew and Joshua, 2012), it was also mentioned that production of 1 ton of cement generates about 1 ton of CO_2 and other GHGs (Celik et al, 2014, Chong et al, 2013, Pourkhorshidi et al, 2010 and Turanli et al, 2005). Moreover Concrete made up of cement, aggregates, water and, additives is the world's most consumed construction material since it is found to be more versatile, durable and reliable (AbdulAleem and Arumairaj, 2012). Concrete is the second most consumed material after water which required large quantities of Portland cement (Bharat and Kamal, 2015).

According to Hardjito et al, (2004), the search for environmentally friendly construction materials is imperative, as the world is facing serious problems due to environmental degradation; a significant expectation is on the industry to reduce carbon dioxide (CO_2) emissions to the atmosphere. Owing to the tremendous growth in development of infrastructure globally, consumption of cement as per International Cement Review's report was 3,294 million tons in 2010 which is increasing by nearly 12% annually, this will significantly create a huge shortage of limestone in future and therefore the need for alternative cementing materials in concrete and mortar. Geopolymer cement is such an alternative, Siddharth et-al, in 2016 stated ''The name geopolymer was formed by a French Professor Davidovits in 1978 to represent a broad range of materials characterized by networks of inorganic molecules. They depend on thermally activated natural materials like Metakaolinite or industrial by-products like fly ash or slag to provide the source of silica (Si) and alumina (Al)''

They are alternative cementitious materials synthesized by blending pozzolanic materials with strong alkali solutions such as sodium hydroxide (NaOH), potassium hydroxide (KOH) and soluble silicates to form a three-dimensional amorphous alumina-silicate network with strength similar to or higher than that of OPC concrete (Ken et al, 2015). Moreover Barath and Kamal, (2015) opined that Geopolymer concrete is better than normal concrete in many aspects such as compressive strength, exposure to aggressive environment, and exposure to high temperature. The study showed that the concrete it gains its full strength quickly and shrinks less than standard concrete. Thus, owing to these structural advantages it may be concluded that in near future Geopolymer concrete may be an effective alternative to standard cement concrete.

Geopolymers are polymers which undergo polycondensation and set fast at low temperature within a few minutes. They are hard, inorganic non-inflammable as well as stable at temperature up to 1250 ^{0}C (Joseph, 2002). Zhang et al, (2014) showed that the concrete exhibits comparable bending and compressive strength as that of ordinary Portland cement specimens both at ambient temperature and after exposure to high temperatures. Thus, the authors concluded that MK–FA based geopolymers offer a feasible alternative to conventional Portland cement in practical building applications.

Chanh et al, (2008) is of the opinion that the exact mechanism of setting and hardening of the geopolymer material is not clear. However, most proposed mechanism of the chemical reaction may comprise; dissolution (to form reactive precursors) of Si and Al atom from source materials through actions of hydroxyl ions, then Transportation. Orientation or condensation of precursor ions in monomers, and finally setting or poly-condensation/Polymerisation of monomers into polymer structures

Despite geopolymer concrete being a promising alternative to Portland/lime cement concrete, it has no code or mix specification to guide the choice of material mix proportion for specific grades of concrete; there is the need to produce a mix design (Material Proportion) that will achieve a specified strength.

This study examines the properties of the source material (Metakaoilin processed from Kankara Kaolin Katsina state) using X-Ray Florescence (XRF) and Fourier-Transform Infrared (FT-IR) analysis. A factorial design of experiment approach in Minitab 18 Statistical software was used to produce mix design for Metakaolin-based Geopolymer concrete that will achieve a characteristic compressive strength of 25 N/mm^2. This is necessary because Geopolymer concrete is a new research area in Nigeria, not much study has been done to investigate the properties of the concrete despite the fact that Nigeria is blessed with abundant Kaolin clay deposits, e.g the Kankara Kaolin clay deposit in Northern Nigeria, the Ajebo kaolin clay deposit in the South-west and in Abeokuta, the Ibere and Oboro clay deposits.

Ephraim, (2016) and Jacob, (2017) worked on metakaolin-based geopolymer concrete but the mix design approach adopted by the authors was for fly-ash based geopolymer as contained in Anuradha et al, (2012). This study focused on coming up with a mix design proportions for Metakaolin-based geopolymer structural concrete.

MATERIALS AND METHOD

Materials

Metakaolin: The metakaolin used for this study was processed from Kankara Local Government Katsina state Kaolin. The process involved beneficiation and calcinations at 800 ^oC which were all done at the Chemical Engineering Department Ahmadu Bello University, Zaria

Table 1: Properties of the Metakaolin

PROPERTY	VALUE
Physical form	Powder
Colour	Baby Pink
Specific Gravity	2.35
Strength Activity Index (SAI)	93% at 28 days (>= 70% (ASTMC618-05))

X-Ray Fluorescence (XRF) of treated and untreated Kaolin: This was conducted at the multi-user laboratory of Chemistry Department A.B.U Zaria, using X-Supreme 8000 Oxford Instrument to obtain the oxide composition of the source material (Kaolin and Metakaolin) by illuminating samples with an X-ray beam. The portion of the energy emitted by the samples which is unique to the chemistry of the samples was analysed for the oxide composition.

Fourier-Transform Infrared Spectroscopy (FT-IR) of Untreated and Treated kaolin: this was also conducted at the multi-user laboratory of Chemistry Department A.B.U Zaria using Cary-630 of Agilent Technologies. Infrared light was passed through the sample and each molecular bond produced a transmittance spectral (Stretching or Vibration) which is a finger print of the sample

Fine and Coarse Aggregates: The aggregates used were sourced from the concrete laboratory of Civil Engineering Department A.B.U Zaria. The coarse aggregate is graded aggregate of a nominal maximum size of 20 mm. Their properties are as shown in Table 2 and 3.

Table 2: Properties of fine aggregate

		Result	Code	requirement
Fine Aggregate	Sieve Analysis	Conforms to code requirement	BS812-103.1-1985	
	Specific Gravity	2.78	BS812-2-1995	2.4-2.9
	Silt and clay content	2%	BS812-2-1985	< 5%

Table 3: properties of coarse aggregate

Properties	Coarse aggregate	**Code**	**Requirement**
Flakiness	20.50%	BS812-105-1989	35% Max
Elongation	14.10%	‘’	35% Max
Aggregate Crushing Value	24.10%	BS812-110-1990	30% Max
Aggregate Impact Value	18.50%	BS812-112-1990	≤ 45 %
Moisture content	0.54%		
Specific gravity	2.57	BS812-2-1995	2.4-2.9

Sodium Silicate (Na_2SO_3): Commercially available Industrial grade sodium silicate was purchased at Sabon-gari market, it is a whitish to greenish liquid gel, with a water content of 40.8%, Silica to Sodium oxide ratio 3.89. (i.e Water is 40.8%, SiO_2 is 46.2% and Na_2O is 11.88%)

Sodium Hydroxide (NaOH): Sodium hydroxide pellets with a purity of approximately 99% was also purchased at Sabon-gari market. The properties are as shown in Table 4.
Water: The water used for preparing the NaOH solution as well as casting operation of the specimens was sourced from A.B.U Zaria, the water is fresh, drinkable and portable, has no definite taste or odour, free from dust, oils, chemicals, and vegetable matter or other major impurities in accordance to BS EN1008-2002

Table 4: Properties of NaOH

PROPERTY	VALUE
Appearance	White
Odour	Odourless
Molar Mass	40 g/mol (Chandra P.K et al, 2017)

METHODS

Design of Experiment (DOE)

Table 5: High and Low limit used in for the Design of Experiments

FACTOR	Lower Limit	Higher Limit
A (Metakaolin content (Kg/m^3))	400	480
B (Molar Concentration (M))	8	14
C (Alkaline Liquid: Metakaolin Content)	0.4	0.65
D (Na2SiO3: NaOH)	1.5	3.0

Table 5 shows the exact values used in Minitab for the design of the experiment. The Metakaolin content range used was 400 to 480 kg/m^3, a molar concentration of 8M to 14 M was used. The fine and coarse aggregate ratio was fixed at 1:2, and the aggregates were proportioned in Kg/m^3, the extra water and superplastcizer used were additives.
Mix Designs by Minitab: A fractional factorial design was used with the 4 factors. 9 runs (8 base designs and 1 centre point) point were proposed by Minitab 18 (table 6). The number of blocks is one (1) because aspects of the experiment (preparation of alkaline liquid, Mixing and casting, curing and testing) were done consecutively in days.

Table 6: Half Fractional Factorial design for four (4) factors with one centre point

RunOrder	Center Pt	Blocks	Metakaolin Content (Kg/m^3)	Molar Concentration (M)	Alkaline liquid :Metakaolin Content	Na2SiO3:NaOH
1	1	1	400	8	0.65	3
2	1	1	400	8	0.4	1.5
3	1	1	480	8	0.65	1.5
4	1	1	400	14	0.4	3
5	1	1	400	14	0.65	1.5
6	1	1	480	8	0.4	3
7	1	1	480	14	0.65	3
8	0	1	440	11	0.525	2.25
9	1	1	480	14	0.4	1.5

Source; Minitab 18 (2018)

Alkaline liquid preparation: The alkaline liquid used composed of Sodium Hydroxide (NaOH) and Sodium Silicate (Na_2SiO_3), NaOH is 40 g/mol; 320 g, 440 g, and 560 g of NaOH was dissolved per litre of the solution for 8 M, 11 M, and 14 M respectively, the solution was allowed at ambient temperature for 24hrs before mixing began, it was mixed with the required proportion of Na_2SiO_3 before use.
Mixing and casting: Metakaolin, Fine and coarse aggregates were measured and dry mixed in a plastic container and then in a concrete mixer for 4 minutes, the NaOH and Na_2SiO_3 solution were poured to the dry-mix and mixing continues. The mix was dry and to obtain a mix that can be cast in a 100*100*100 mm mould, a specified content of super-plasticizer was added together with extra water gradually added to get mouldable cohesive mix. The fresh mix was placed in the moulds in 3 layers each giving 25 blows with a tamping rod and the surface was trimmed.
Materials added in the laboratory (Water and Super-Plasticizer): It was noted that the fresh mix was very stiff for most of the mixes which may be due to the fineness of the metakaolin used as stated by Mohammed et al, (2014) that the finer the particles of the pozzolan, the more water it will

require. In order to improve the workability of these mixes a Glenium based Super-Plasticizer with name Hydro-Plast 500 was added as a percentage of Metakaolin content and extra water added was recorded for each as shown in Table 7

Table 7: Super-Plasticizer (SP) and Water added during Mixing

Runs	Metakaolin content (kg/m3)	SP %	SP added (kg/m3)	Water added (kg/m3))
1	400	0	0	47.5
2	400	1.5	6	127.11
3	480	0	0	57
4	400	1.5	6	140.125
5	400	1	4	66.5
6	480	1	4.8	148.675
7	480	1	4.8	61.75
8	440	1.02	4.488	101.65
9	480	1.5	7.2	127.3

Source: Laboratory work (2018)

Temperature and duration of curing: The specimens were allowed a resting period of 24 hours under ambient temperature after which they were de-moulded weighed, wrapped in a polyethylene bag and placed in an oven and cured for 24 hours at 60 ^{o}C. The specimens were then removed weighed again and kept under ambient temperature and humidity until the day of crushing (Testing)

Testing The compressive strength test was conducted at 14 days after casting (13 days ambient and 1 day heat), the choice is based on findings and recommendations of Vijai et al, (2010) who stated that the strength of heat cured GPC doesn't increase significantly from 7 days but for ambient temperature cured GPC,. Pawan and Surendra, (2016), states that ''the rate of strength gain after 7 days is slow''. Harditjo et al, (2004) reported that after heat curing, the strength of geopolymer concrete does not vary with age because the chemical reaction of the gel is due to substantially a fast geopolymerisation process. Aaron, (2010) also stated that '', any strength attained at 14 days will not be significantly different from that at 28 days''.

RESULTS AND DISCUSSION

Table 8: Comparison of oxide composition

Oxides	Untreated (Kaolin)	Treated (Metakaolin)	ASTMC618-05	Getso.A. I (2013)
Al2O3+SiO2 + Fe2O3	96.51%	98.42%	≥70%	94.90%
CaO + MgO	0.995%	0.43%	≤ 5%	0.29%

Source: Laboratory work (2018)

From the oxide composition (table 8) of the treated and untreated kaolin, it can be seen that, the difference in oxide composition after the treatment is not much, this agrees with Bhaskar and Gopalakrishnarao's findings in 2010 who reported with XRF the difference after beneficiation is marginal , this is so because the kaolin contained a small proportion of the deleterious oxides, but as can be seen the percentage of the useful oxides increased by about 2% and that of non-deleterious oxides reduced by 0.565 %, the essence of the beneficiation is to reduce the presence of the unwanted oxides

FTIR Results
Kaolin

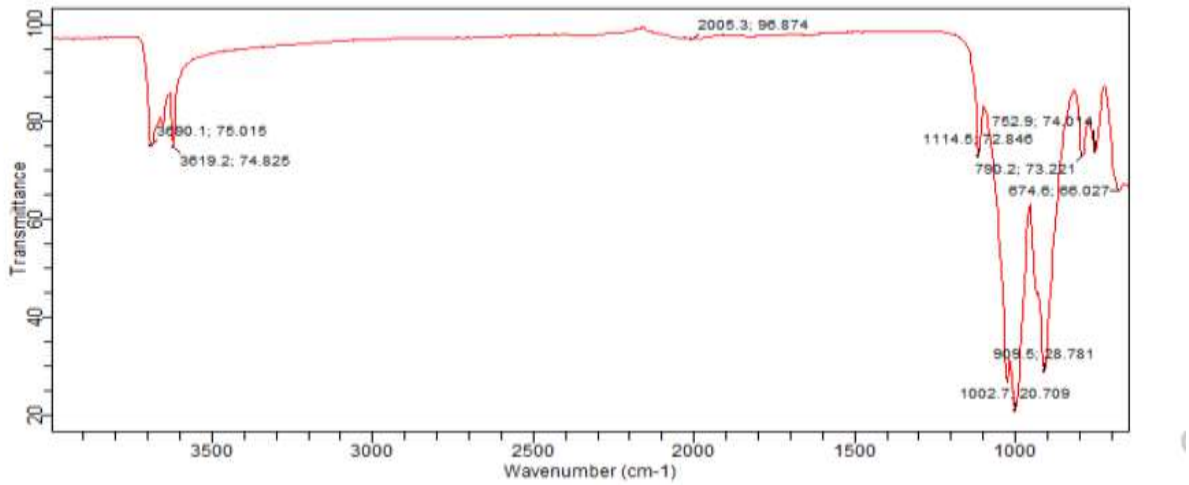

Fig. 1: FTIR of Raw Untreated Kaolin

Bhaskar and Gopalakrishnarao, (2010) stated that the IR spectrum of a typical kaolin is at 3697, 3669, 3645 and 3620 cm^{-1}, the band on Figure 1 between 3619 and 3690 cm^{-1} cover s these range indicating kaolinite clay minerals, two characteristic bands were exhibited near 3669 and 3620 cm-^{1}, The band at 3690 cm^{-1} on the figure indicates Al-O-S stretching and at 3619 cm^{-1} be OH Stretching (Crystalline hydroxyl). the band at 2005 cm^{-1} could be assigned to the OH^{-} vibration mode of a hydroxyl molecule and at 1114.5 cm^{-1} could be an interference due to the presence of quartz; OH deformation linked to $2Al^{3-}$ can be seen at 909.5 cm^{-1}, that at 790.2 cm^{-1} is due to Si-O-Si inter tetrahedral bridging bonds in SiO_2 and around 674.6 and 752.9 cm^{-1} may be Si-O quartz.

Metakaoiln

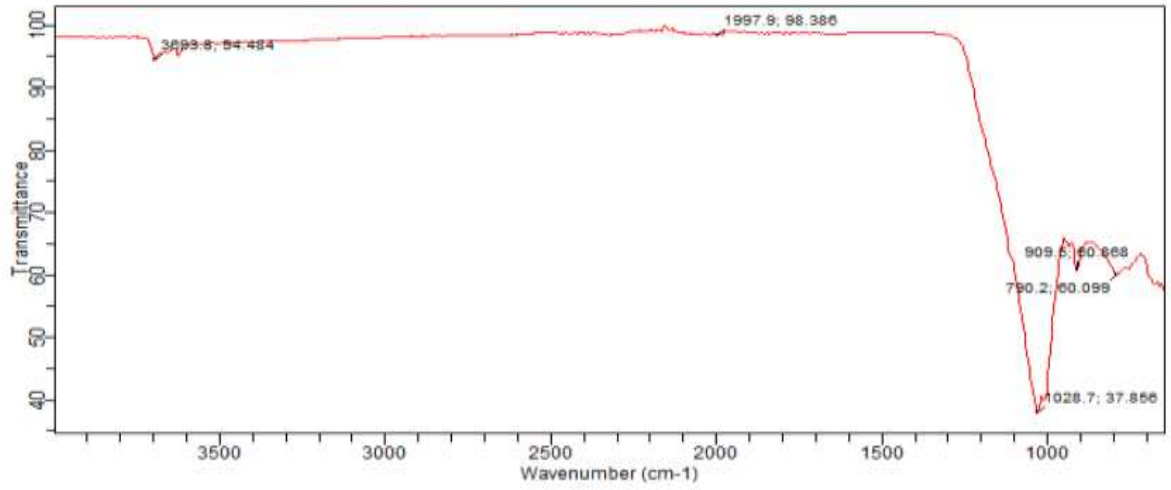

Fig. 2: FTIR of Treated Kaolin (Metakaolin)

Figure 2 showed the FTIR result of treated (beneficiated and calcined) Kaolin. The kaoilinite mineral band between 3619 and 3690 cm^{-1} increased in transmittance when treated to 94.484 % from 74.825 % when untreated, this is in agreement with Marounane et al, (2019). The OH vibration mode of the hydroxyl molecule shifted to 1997.9 cm^{-1}. The interference due to quartz at 1114.5 cm^{-1} was no more after treatment. The Si-O stretching of Kaolinite clay minerals is which was at 1002.7 cm^{-1} shift to 1028.7 cm^{-1}. The bands at 674.6 and 752.9 cm^{-1} which may be due to Si-O quartz have reduced intensity, the OH deformation linked to $2Al^{3-}$ remains at 909.5 cm^{-1}, and the Si-O-Si inter tetrahedral bridging bonds in SiO_2 can also be seen at 790.2 cm^{-1}. The shift in wave number is an indication of bond change from kaolin to metakaolin.

Density of the Geopolymer Mixes

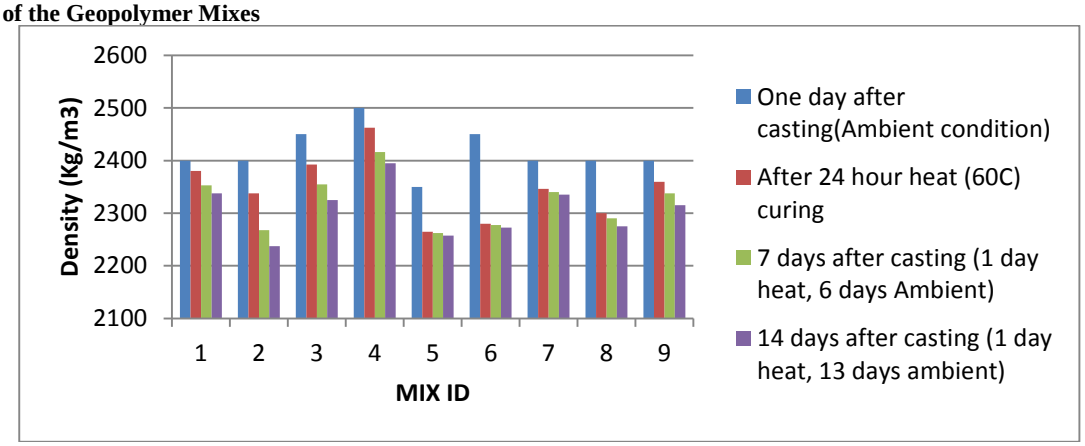

Fig. 3**:** Change in density MBGPC Mixtures

The bulk density of the geopolymer is shown in Figure 3, the density values are similar to that of conventional concrete, The moisture loss is highest after 24 hour heat curing at 60^{o}C and therefore the drop in density is more pronounced; when left under ambient temperature, the density continued dropping, this is due to progressive moisture loss of the metakaolin based geopolymer concrete mix with time. The highest and lowest densities recorded after a day under ambient temperature are 2500 kg/m^3 and 2350 kg/m^3 respectively, after 14 days (1-day heat and 13 days ambient) these densities dropped by 4.2% to 2395 kg/m^3 and 4% to 2257.5 kg/m^3 respectively.

Compressive strength of the Geopolymer concrete Mixes

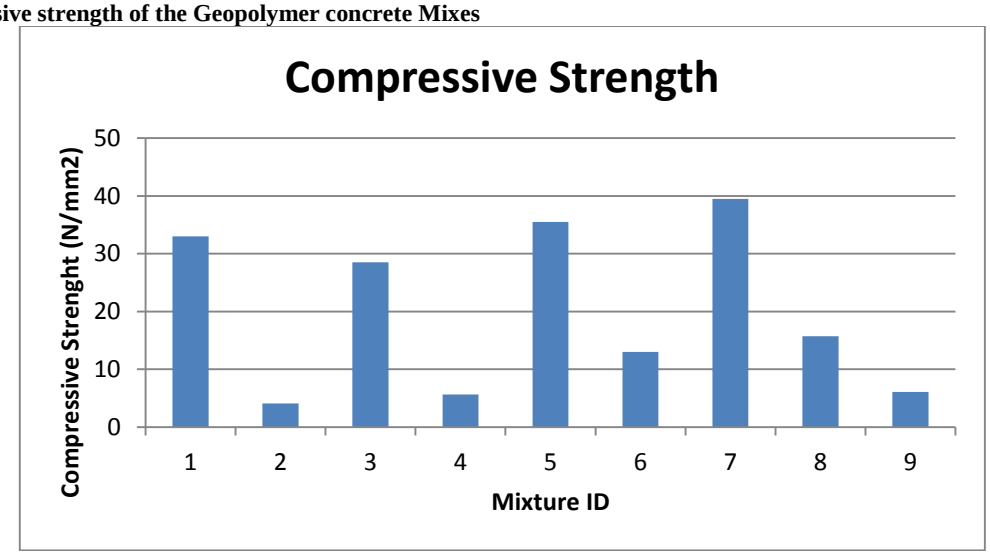

Fig. 4**:** Compressive strength values of Mix 1 to 9

Figure 4 showed the compressive strength results for each mixture ID, the highest and lowest compressive strength values are 39.5 N/mm^2 and 4.1 N/mm^2, four mixes have values greater than 25 N/mm^2 and five below this value, the centre point mix (ID8) has a compressive strength of 15.75 N/mm^2 which is an approximate average of the compressive strengths. Mixes with the high values have a high alkaline liquid content (0.65) while others have low alkaline liquid content (0.4) except ID8 which is a centre point; this is because a high alkaline liquid content makes the geo-polymerization more complete than with low alkaline liquid which has insufficient activator for the dissolution of the source material. The two highest compressive strength values (ID5 and ID7) were obtained with 14M concentration followed by 8M concentration (ID1 and ID3), this agrees with Shankar and Khadiranaikar, (2013) that a higher compressive strength is obtainable with higher molar concentration, moreover the compressive strength value was higher with Na_2SiO_3: NaOH of 3 than with 1.5, this agrees with reports by Zarina et al, (2015), Harditjo et al, (2005), that the reaction will be enhanced and strength increased with an increased Na_2SiO_3 content.

Minitab's Proposed Mix Proportions For 25N/mm^2 Compressive Strength

The results from the laboratory work were re-analyzed by Minitab 18 with a set target compressive strength and mix proportions for the set target were proposed by Minitab 18

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Table 9: Minitab's Input Parameters

Response	Goal	Lower	Target	Upper	Weight	Importance
Cs	Target	4.1	25	39.5	1	1

Table 10: First Five mix proportions proposed after a factorial results analysis by Minitab 18

Solution	Mc	Mo	AMr	ALr	Sp	Wa	Cs Fit	Composite Desirability
1	479.60	9.61	0.40	2.42	7.08	145.72	25.00	1.00
2	440.00	11.00	0.53	2.25	3.60	91.89	25.00	1.00
3	479.89	13.99	0.65	3.00	1.49	47.50	25.00	1.00
4	**400.20**	**14.00**	**0.65**	**3.00**	**0.00**	**55.26**	**25.00**	**1.00**
5	478.82	8.11	0.41	2.99	6.00	148.68	25.00	1.00
6	478.32	8.23	0.65	2.98	0.00	66.45	25.00	1.00

Table 11: Response surface analysis prediction

Solution	Mc	Mo	AMr	ALr	Sp	Wa	Cs Fit	Composite Desirability
1	409.416	8.0051	0.649615	1.57290	0.03484	49.293	25	1

Where Mc stands for Metakaolin Content, Mo is Molar Concentration, AMr is the alkaline liquid Metakaolin ratio, ALr is Na_2SiO_3:NaOH, Sp is the superplasticizer dosage and Wa is the water content. The choice of the mix proportion for use to achieve the compressive strength of 25 N/mm^2 was based on the effect and cost of constituent materials as observed during the laboratory work, the processes involved in producing the metakaolin used make the cost of producing metakaolin significant, the alkaline liquids (Sodium silicate and Sodium hydroxide solution) were purchased at reasonable prices. Therefore the choice of the mix proportion used was based on:

- Mix proportion with low metakaolin content
- Least possible superplasticizer (SP) requirement (Because the SP used has no significant positive effect)
- High alkaline liquid content (Because of strength and workability of the mix)

Mix No 4 on table 10 seems to be the optimum and therefore it was chosen for the validation.

Another mix was made to having molar concentration as 8M and all other factors the same as No 4; the reason for this choice is because results from trial mixes indicated that good compressive strength values can be achieved with 8M (requiring less NaOH Content), also workability was noticed to be higher with low Molar Concentration which agrees with Sivakumar, (2013), that higher Molar concentration is associated with rapid loss of consistency, in addition mix no 6 (Table 10) proposed a mix design with a difference to No 4 only in metakaolin content (478 kg/m^3), and finally, response surface method analysis also predicted a most desired mix with 8M (Table 11).

The two mixes (Table 12) were prepared, cast and cured for validation of the Design of Experiment (DOE)

Table 12: Mix choice for 25 N/mm^2 validation

Solution	Mc	Mo	AMr	ALr	Sp	Wa	Cs Fit
M1(4)	400.196	**13.9964**	0.650000	2.99801	0.00000	55.262	25
M2	400.196	**8.00000**	0.650000	2.99801	0.00000	55.262	25

Compressive strength results Mix M1 and M2

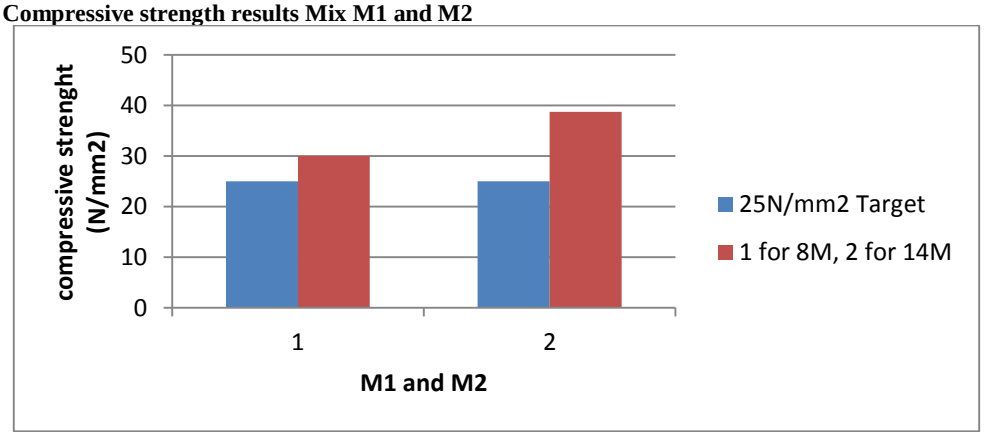

Fig. 5**:** Compressive strength against Mixes M1 and M2

Figure 5 presents compressive strength results targeting 25 N/mm^2 for M1 and M2, M2 having a higher compressive strength because of higher molar concentration. It can be seen that both mixes met the target value of 25 N/mm^2, the Mix proposed by Minitab 18 (M1) achieved a compressive strength of 37.5 N/mm^2 exceeding the target by 55 % while the second mix (M2) has a compressive strength of 30 N/mm^2 exceeding the target by 20 %, this implies that both mixes are satisfactory going by (ACI 211.4R.93, 1998). Which states ‘’to meet the specified strength requirements, the concrete must be proportioned in such a manner that the average compressive strength results of field tests exceed the specified design compressive strength by an amount sufficiently high to make the probability of low tests small’’ from the foregoing the mixes proposed (M1 and M2) achieved the target compressive strength and can be used to produce structural reinforced geopolymer concrete.

CONCLUSIONS

The results of this study revealed that treatment of kaolin clay (Beneficiation and calcinations) greatly reduces the presence of quartz and brings about a molecular bond change as indicated by increased transmittance and a shift in wave-number; on the other hand, the XRF analysis showed a marginal difference in oxide composition after treatment. The compressive strength of the Metakolin-based geopolymer increases with an increase in the ratio of Alkaline liquid:Metakaolin content and Na_2SiO_3:NaOH respectively to the maximum of 0.65 and 3.0 checked, also an Alkaline liquid ratio metakaolin of 0.65 is sufficient for compressive strength values of up to 40 N/mm^2. Finally the mix proportions (M1 and M2) achieved the target compressive strength and can be used to produce structural reinforced geopolymer concrete.

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