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Assessment of the Mechanical and Time - Dependent Properties of Ambient Cured Metakaolin – Based Geopolymer Concrete

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Abstract
Development of geopolymer concrete using locally sourced kaolin can lead to a significant reduction of the carbon footprint of concrete and also increase the economic value of kaolin which is deposited in large quantity in most part of Nigeria. There is limited information available on the structural characteristics of geopolymer concrete that is based on other material than fly ash and blast furnace slag. Hence, assessment of mechanical and time – dependent properties of metakaolin based geopolymer concrete is important to ascertain the suitability of locally sourced material for the development of structurally viable geopolymer concrete. It has been established that the ambient cured metakaolin based geopolymer concrete (MGC) exhibited a higher strength than the conventional Portland cement concrete. The mechanical strength properties tests revealed that MGC recorded a rapid strength development by achieving approximately 90% - 95% of 28days strength within 3days of ambient curing, whereas, PCC could achieved approximately 43% of its 28days strength within 3days of water curing. At 28days the MGC recorded approximately 18%, 13% and 9% higher compressive, flexural and tensile strength respectively than PCC of equivalent mix content. The time – dependent tests revealed that the geopolymer concrete had a marginal 5% lower elastic modulus than PCC. At about 180 days, a lower shrinkage of about 66% and a reduced total creep coefficient of about 38% was recorded for MGC in comparison with PCC. It can be concluded that the metakaolin geopolymer concrete offers notable advantages in structural performance than the conventional Portland cement concrete.

Keyword: Ambient cured, Metakaolin geopolymer concrete, Mechanical properties, Time – dependent properties

1. Introduction

The entrenched production technology and processing of Portland cement has been identified as a major contribution to the greenhouse effect. Portland cement industry contributes approximately 5% - 7% of global CO_2 emissions (Kumar *et al*, 2018). However, there is an ever increasing demand for Portland cement/concrete to tackle the infrastructure deficit in Nigeria emphasises the need for alternative material to Portland cement/concrete which according to Anifowoshe and Akinremi, (2019) expanded by 2.1% to about 20.7mta in 2018. About 50% reduction of CO_2 emission is associated with the replacement of Portland cement with such supplementary cementitious materials (SCMs) as silica fume, fly ash and blast furnace slag in the range of 20% - 50% (van Deventer, *et al*, 2012). However, a more efficient approach to reducing the negative impact of Portland cement/concrete in sustainable infrastructural drive is the 100% substitution of Portland cement with an alkali – activated aluminosilicate rich material known as Geopolymer binder (Duxson, *et al*, 2007; Shi, *et al*, 2011).

Research on the use of kaolin for the development of geopolymer concrete is not yet popular in Nigeria. The characteristics of geopolymer concrete under different service conditions are still shrouded in uncertainty. Studies on the use of metakaolin – based geopolymer concrete as a suitable alternative material for serious structural application is scarce. Without adequate study on the metakaolin based geopolymer material, its mix formulation and performance properties as concrete product, it would be near impossible to achieve a viable commercialization of the product as a viable alternative to Portland cement and a greener solution in the concrete industry.

Geopolymer binder can be obtained from the alkali activation of materials that are rich in alumina and silica oxides. Such materials can be naturally sourced like metakaolin or by – product materials such as fly ash, blast furnace slag, silica fume etc. Metakaolin is produced from natural clays (Kaolin) by calcination at a moderate temperature. In addition to aluminosilicate material, there is need for alkali activators to initiate geopolymerization reaction and turn the source material to binder material. Most popular alkali activators in geopolymer mixtures are a combination of either sodium hydroxide (NaOH) and sodium silicate or potassium hydroxide (KOH) and potassium silicate (Albidah, *et al*, 2021).

Most recent works on geopolymer explore fly ash – based geopolymer (Hardjito and Rangan, 2005; Arioz *et al*, 2012; Chuah, *et al*, 2016; Lahoti *et al*, 2018), slag – based geopolymer (Atis, *et al*, 2009; Allahverdi *et al*, 2010; Kathirvel and Kaliyaperumal, 2016), rice husk ash – based geopolymer (He, *et al*, 2013; Zabihi, *et al*, 2018), kaolin (Heah, *et al*, 2011; Okoye *et al*, 2015). Most popular research works on metakaolin – based geopolymer are limited to mortar and paste (Weng and Sagoe – Crentsil, 2007; Yao *et al*, 2009; Kamalloo *et al*, 2010; Medri, *et al*, 2010; Zhang, *et al*, 2016). Available information on metakaolin – based geopolymer material are limited to the study conducted by Pouhet, (2015) who reported the development of geopolymer mortar using flash – calcined metakaolin and sodium silicate. From this report a very rapid reaction of the alkalis was observed in the geopolymer paste and the addition of sodium hydroxide in the mix led to greater dispersions of the strength values. Nur *et al*, (2017) investigated the performance of metakaolin geopolymers exposed to seawater and found that the material remains structurally intact. Albidah *et al* (2021) studied the characteristics of metakaolin – based geopolymer concrete for different mix design parameters. Different properties like workability, density, compressive strength, water absorption, split tensile strength, ultrasonic pulse velocity were determined and some useful models were proposed for predicting these properties.

Most studies on metakaolin – based geopolymer concrete are limited to parametric studies and the effect of various mix parameters on the mechanical properties of the geopolymer material. Studies on both mechanical and time – dependent properties of metakaolin geopolymer concrete are very scarce. The objective of this study therefore is to assess the mechanical and time – dependent properties of metakaolin – based geopolymer concrete and to compare the results with the conventional Portland cement concrete. Such mechanical properties as compressive strength, flexural strength and split tensile strength were determined and the time – dependent properties such as elastic modulus, drying shrinkage and compressive creep were also determined.

2.0 Materials and Methods

2.1.0 Metakaolin

The kaolin was locally sourced from Ikpeshi, in Edo state, Nigeria. The raw kaolin was pinkish white and smooth grained in texture. The kaolin was calcined at a temperature of 750^0C for 3 hours in the furnace. Figure 1 shows sample of the kaolin and metakaolin. X – ray fluorescence test was conducted on the metakaolin sample to obtained it oxide composition as presented in table 1. The metakaolin can be classified as low – calcium precursor based on the ASTM C618 criteria i.e. SiO_2+Al_2O_3+Fe_2O_3 = 87.4 ≥70.

Figure 1: (a) Raw Kaolin Powder (b) Calcination of Kaolin in furnace (c) Metakaolin powder

Table 1: Chemical composition of metakaolin

Composition	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**	**TiO_2**	**LOI**
Percentage	49.10	37.93	0.38	0.34	0.11	0.28	1.32	1.14	1.03

2.1.1 Alkaline Activator

Alkaline activators used in this study is a mixture of sodium hydroxide and sodium silicate solutions of industrial grades. The sodium hydroxide was obtained as a bag of solid flake and the NaOH solution was prepared prior to casting, using condensates from air conditioner system. The sodium silicate solution was obtained from a local supplier.

(a) (b) (c)

Figure 2: (a) Bag of sodium hydroxide flakes (b) Sodium hydroxide solution (c) Sodium silicate solution

2.1.2 Aggregates

The fine and coarse aggregates used in this study include river sand and crushed granite respectively, and they were obtained from local aggregate market. The physical properties of both coarse and fine aggregate such as particle density, specific gravity, water absorption, crushing and impact value were determined in line with relevant standard procedures as presented in table 2.

Table 2: Physical properties of aggregates

Property	**Coarse Aggregate**	**Fine Aggregate**
Particle size distribution	BS 882 – 103 (1992)	BS 882 – 103 (1992)
Particle density (kg/m^3)	AS 2758.1 (2014)	AS 2758.1 (2014)
Bulk Specific gravity	ASTM C127 (2007)	ASTM C128 (2007)
Water absorption (%)	ASTM C127 (2007)	ASTM C128 (2007)
Aggregate crushing value (%)	BS 812 – 110 (1990)	-
Aggregate impact value (%)	BS 812 – 112 (1990)	-

2.2 Methods

2.2.1 Specimen Preparation

Concrete specimens were prepared using mix proportion shown in table 3. The mix proportion for geopolymer concrete was obtained from Taguchi optimization method considering four synthesis parameters such as alkali/metakaolin ratio, sodium silicate/sodium hydroxide ratio, sodium hydroxide concentration and aggregate content.

Table 3: Mixture proportion of PCC and MGC

(kg/m^3)	**Cement**	**MK**	**F.A**	**C.A**	**SS**	**SH**	**H_2O**
PCC	715	-	715	715	-	-	255
MGC	-	558	725	725	235	157	-

The preparation of the geopolymer concrete involves mixing the dry components including fine aggregate, metakaolin powder and coarse aggregate in that order in an electric mixer for about 3mins. Then the combined alkali solutions were added albeit gradually while the mixing continue until homogeneous mix was achieved. The concrete mixture was placed in slump cone for slump test after which it was remixed and placed in respective moulds for each of compressive strength test (100mmx100mmx100mm), flexural strength test (100mmx100mmx500mm), split tensile strength, elastic modulus and creep test (100mmx200mm), and drying shrinkage test (50mmx50mmx200mm). After casting the concrete in the mould, they were placed on vibratory table for consolidation and then covered with polythene sheet for the next 24hrs, after which they were demoulded. The geopolymer concretes were kept at a safe place in the lab for ambient curing at 24^0C – 28^0C and 50 – 75% relative humidity, while the control specimens were cured in water until they were tested.

2.2.2 Test Method

For the determination of the mechanical and time – dependent properties of concrete samples, relevant standards were used as guide and average of three specimens were tested at each age of testing. The details of the test program for each test is presented in table 4.

Table 4: Test program for mechanical and time – dependent properties of PCC and MGC

Type of Test	Reference Standard	Age of test (day)
Compressive strength	EN 12390 – 3 (2002)	3, 7, 14, 28, 56, 90
Flexural strength	EN 12390 – 5 (2000)	3, 7, 14, 28, 56, 90
Split tensile strength	EN 12390 – 6 (2000)	3, 7, 14, 28, 56, 90
Elastic modulus	ASTM C469/C469M (2014)	3, 7, 28, 56, 90
Drying shrinkage	ASTM C157/C157M (2008)	7, ………180
Creep	ASTM C512 (2018)	1, ……….180

3.0 Results and Discussions

3.1 Compressive Strength

The results of compressive strength of both PCC and MGC is as presented in figure 3. It is clear from figure 3 that the MGC achieved 90% - 95% of its 28day compressive strength at 3days. This observation aligned with the finding of Albidah, *et al*, (2021) in which MK – based GPC specimens achieved 90% – 95% of the 28day strength at 7day of ambient curing. There is no significant increase in strength of geopolymer beyond 3day, and this indicates that ambient curing does not contribute to the strength development of geopolymer concrete unlike the increase in strength of Portland cement concrete due to continuous hydration process of Portland cement. It is also instructive that even though the MGC recorded higher early strength than PCC, at later age, the strength gap closes substantially. It cannot be said that MGC is better than PCC in terms of their attainable strength value, but it can be argued that the former is advantageous over the latter due to its early strength development which could results in rapid construction and saving construction cost.

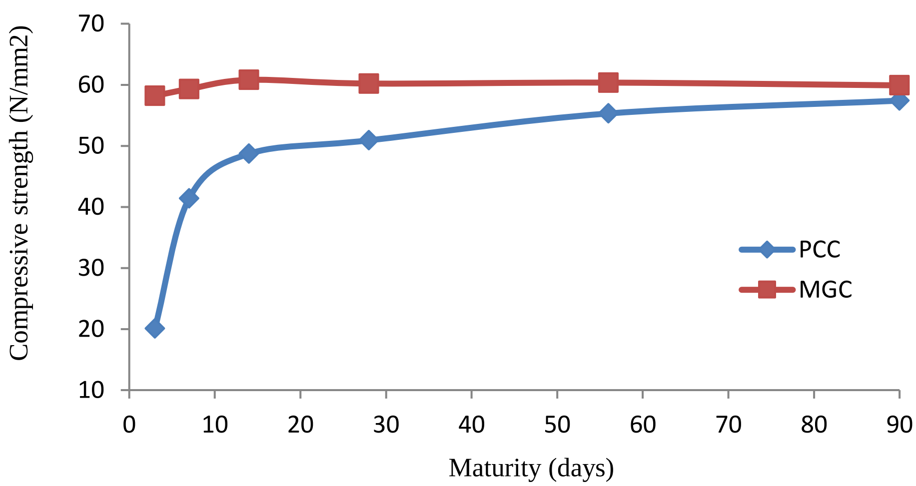

Figure 3: Compressive strength of PCC and MGC

3.2 Flexural Strength

Figure 4 shows the results of the flexural strength characteristics of PCC and MGC. The figure indicates that the flexural strength of MGC is relatively higher than that of PCC albeit by small margin. The higher early flexural strength of MGC can be traced to the higher early compressive strength of MGC in comparison with PCC. This pattern of flexural strength property of MGC is in agreement with the findings of Gautam *et al*, (2015) where it was found that the average flexural strength of geopolymer concrete at 7day is about 74% of the 28days strength whereas, that of conventional concrete of grade M50 was about 39% of its 28days strength.

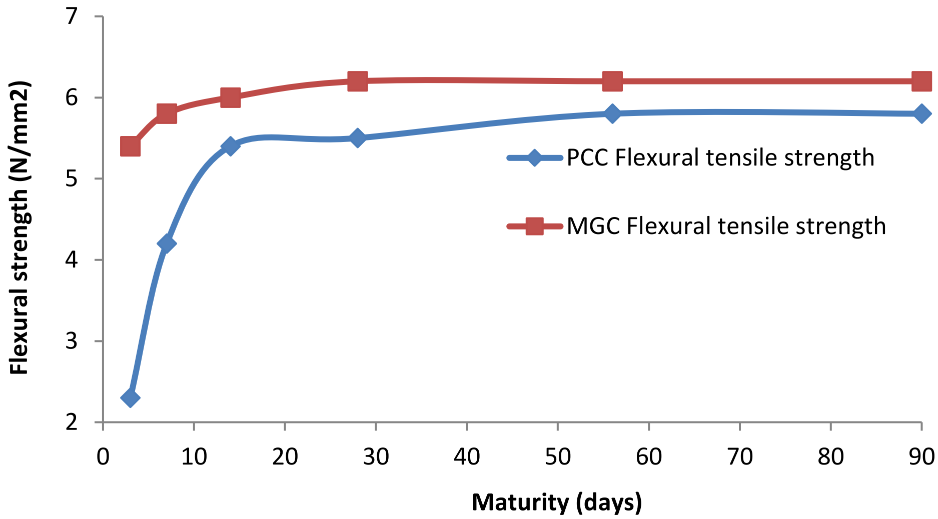

Figure 4: Flexural strength of PCC and MGC

3.3 Split Tensile Strength

Figure 5 presents the results of the split tensile strength property of PCC and MGC, indicating that the tensile strength of MGC is marginally higher than that of PCC. The pattern of behaviour of MGC and PCC is similar to compressive and flexural strength earlier discussed, and it validates the findings of other authors like Gautam, *et al* (2015), where average splitting tensile strength developed by GPC at 7day is 60% of its 28days strength while the split tensile strength of Portland cement concrete of equivalent strength was 45% of its 28days strength.

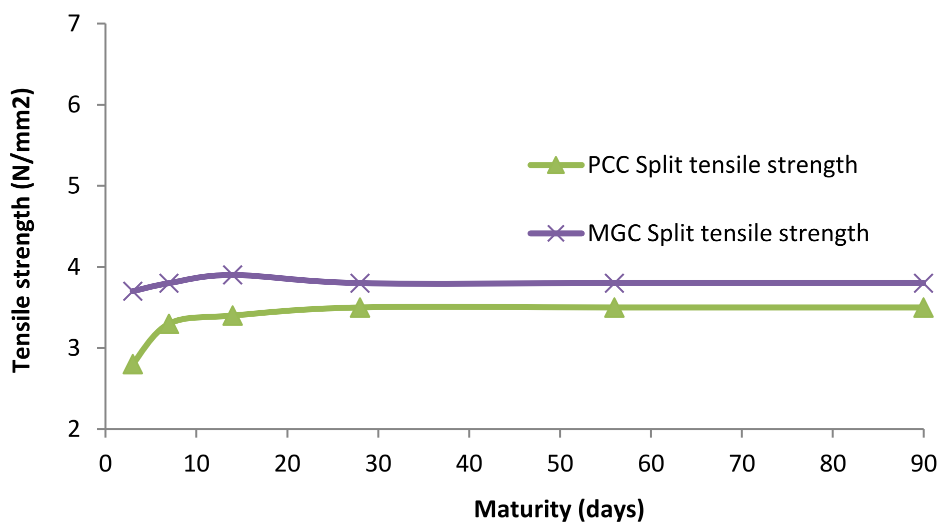

Figure 5: Split tensile strength of PCC and MGC

3.4 Elastic Modulus

The results of the elastic modulus of PCC and MGC is as presented in figure 6. The average elastic modulus value of MGC is marginally lower than that of PCC despite the higher early strength of the former. This indicates that MGC shows early sign of distress under load prior to deformation, hence, it can be said to be more ductile in compression than PCC. This observation agrees with the findings of Sarath Chandra *et al* (2018) where it was reported that geopolymer concrete from blended fly ash and blast furnace slag was more ductile than Portland cement concrete. A ductile material plastically deforms to a large extent before fracture under load and it absorbs higher energy before fracture.

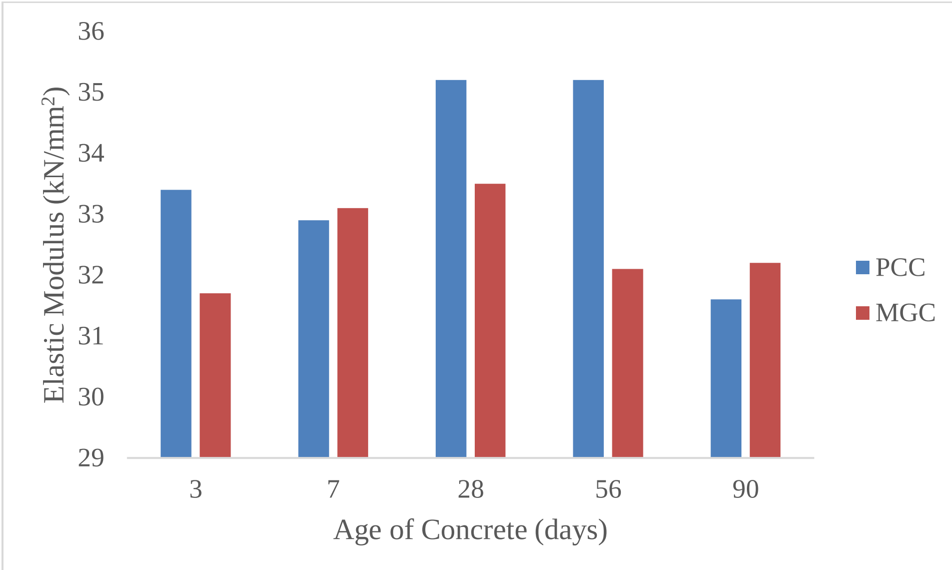

Figure 6: Elastic modulus of PCC and MGC

3.5 Drying Shrinkage

Figure 7 shows the drying shrinkage behaviour of PCC and MGC. The shrinkage value of both PCC and MGC was found below 1000 microstrain which is the acceptable limit specified in standard Australia AS 1379, (2007). However, MGC specimens shrank less than the PCC specimens under the same curing condition by an average 66%. The lower shrinkage value of MGC emphasizes its volume stability due partly to its high early strength characteristic. At 7 days of curing, MGC has attained higher maturity than PCC, and it is prone to less moisture with the environment due to absence of excess water in its mixture.

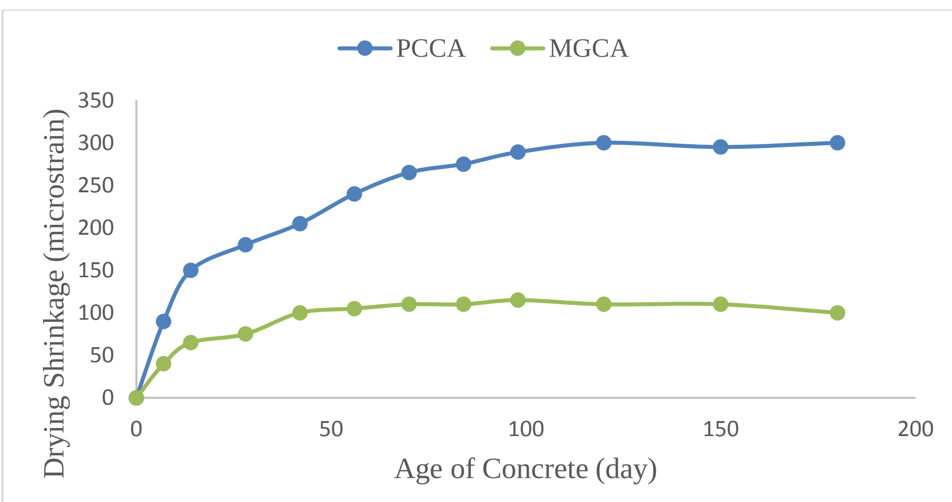

Figure 7: Drying shrinkage property of PCC and MGC

3.6 Creep

The net creep strain at any age is the subtraction of the drying shrinkage strain and the instantaneous strain from the total strain. Creep coefficient represents the magnitude of creep strain as a function of instantaneous strains. Total and basic creep coefficient were obtained as the ratio of total and basic creep strain and their corresponding instantaneous strain after application of load. Figure 8 shows the results of the total creep coefficient of PCC and MGC. Total creep coefficient of PCC is higher than MGC by an average of 43% over test period. Membrane covering may be necessary to limit the total creep coefficient of PCC when the concrete structure is creep sensitive, but this is not necessary for MGC as it adapt better to the environment.

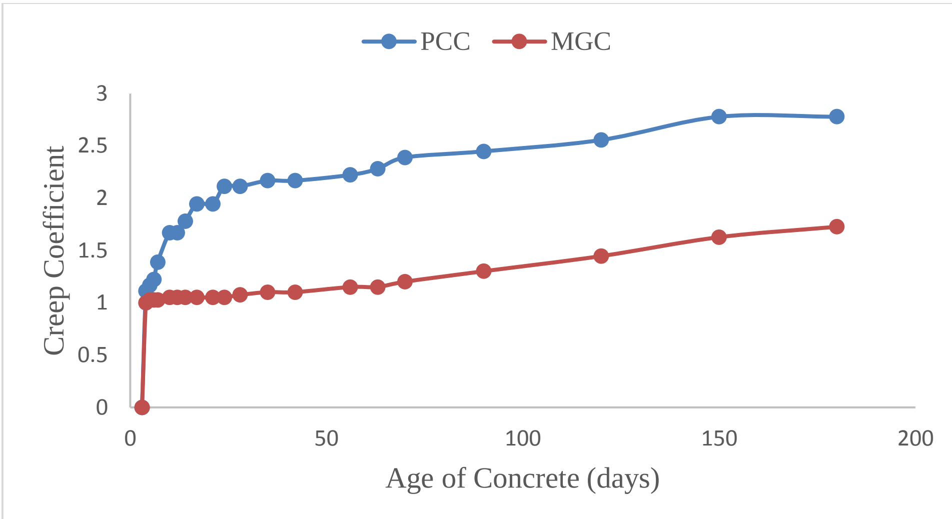

Figure 8: Total creep of PCC and MGC

4.0 Conclusions

In this study, mechanical and time – dependent properties of metakaolin geopolymer concrete was assessed in comparison with conventional Portland cement concrete with a view to ascertain its suitability for important structural application. The findings in this study addressed some issues concerning the general view that geopolymer concrete from low – calcium material requires elevated temperature curing regime to achieve significant strength at early age. It was found that ambient cured MGC has high early strength advantage over conventional PCC, making it suitable for fast construction. Also, it was established that the mechanical properties of MGC follow the same trend and they are relatively higher than PCC.

However, it was found that PCC exhibited about 5% higher elastic modulus than MGC. The behaviour of MGC under load suggests that it is more ductile than PCC, for showing higher capacity to sustain load under deformation before it reaches its ultimate strength capacity. The drying shrinkage property indicates that MGC is a stable material under normal environmental condition and it is significantly more stable than PCC, hence it is more suitable for general purpose application. The creep behaviour of MGC indicates that MGC recorded less strain under load than PCC under similar loading condition. The creep coefficient of PCC recorded an average of 38% higher than MGC over the period of testing. MGC is thus a stable material under sustained load and in the face of changing temperature and relative humidity condition.

5.0 Recommendation

Further studies in relation to durability – related properties and extensive microstructural study on metakaolin – based geopolymer concrete would provide increased knowledge on the general characteristics of the concrete for general and specific uses.

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