# EFFECTS OF TEMPERATURE ON THE POZZOLANIC CHARACTERISTICS OF METAKAOLIN-CONCRETE

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**Abstract**

This paper focused on the thermal technique of activating pozzolanic activity of natural pozzolans, specifically calcined kaolinite clay in the form of metakaolin. The effect of heating temperatures over the range 450ºC-1050ºC in steps of 150ºC on the dehydroxylation of the kaolin and the pozzolanic activity of the resulting amorphous material were determined. The compressive strengths of metakaolin-concrete samples with 10%, 20% and 30% replacement of cement, calcined at different temperatures 450ºC, 600ºC, 750ºC, 900ºC and 1050ºC comparable to the control samples were analysed to determine the optimum temperature. Also, mixtures of mortar containing variable amounts of kaolin in the ration 5% to 30% in steps of 5% were calcined at the optimum temperature of 750ºC and investigated.

The results showed the optimum heating temperature for the kaolin to be 750ºC and the proportion of metakaolin in concrete should be 10% of the cement weight. For 10% metakaolin at temperature of 750ºC, the strengths were about 109% and 107% of normal mortar cubes at 28 days and 90 days respectively, while those of concrete cubes were 99% and 97% of normal concrete cubes. Also, with replacement proportion range of 15-25% of metakaolin (MK) in concrete, a relative concrete strength of 22.23MPa and 23.15MPa for 28 and 60 days respectively curing ages can be achieved

***Key words:* calcined kaolinite clay, metakaolin, thermal process, pozzolanic activity, blended mortar, concrete.**

## 1.0 Introduction

In Nigeria, annual cement consumption value is 19.5 million metric tonnes out of which only 9.5 metric tonnes are produced locally (Franklin, 2009). The abruptly high demand for cement owed to increased population and infrastructural development has resulted in the rapid depletion of unsustainable natural resources, problems of Carbon dioxide ($CO_2$) emission and high cost of cement. In order to solve these problems, as well as improve mortar/concrete performance, the exploration of cheaper materials that could be used as partial substitute for cement in mortar and/or concrete has become a focus point by researchers and specialists all over the world.

In Nigeria, kaolinitic clay seems to have the greatest overall potential as alternative pozzolanic material for concrete due to its availability in large quantity and the relatively low price. Nigeria has an estimated reserve of about two billion metric tons of kaolin deposit, scattered in different parts of the country and this includes Ogun, Kogi, Imo, Rivers, Anambra, Bauchi, Kebbi, Ondo, Ekiti,AkwaIbom, Katsina and Plateau States.

Kaolin clay group, which is represented chemically as Aluminosilicate hydrate ($Al_2O_3SiO_2 \cdot 2H_2O$), is normally converted to metakaolin by a process known as calcination (thermal treatment) at a temperature of about 700°C-900°C to drive out chemically-bound water and destroy the crystalline structure (Kakali et al., 2001). Clay calcination is necessary for converting clay to display cement-like behaviour. Metakaolin is more reactive with either acid or alkalis. It is generally known that pozzolan produced by calcination modifies the properties of lime and Portland cement mortars and concrete in a similar manner to natural pozzolan. The metakaolin reacts with calcium silica and calcium aluminate hydrates unlike other natural pozzolan (Zhang and Malhotra, 1995). This reaction takes place early in the hydration setting period and continues to occur during hydration, continuing to enhance the properties of the installed concrete.

Past studies have shown that the use of pozzolan-blended cement in concrete and mortar can increase compressive strength, flexural strength, resistance to chemical attack (e.g. sulphate) and even improve durability (Dahl et al., 2007 and Rodriguez, 2006 ).

Chin-Yi and Wei-Hsing (2002) reported that the result of dehydroxylation is a new phase called ametakaolinite. During this reaction, as X-ray Diffraction (XRD) showed, thehigher-order reflections lost their intensity and vanished inthe XRD background. This result led to the opinion that the metakaolinite can be amorphous, being a conception ofthe short-range order crystalline structure of metakaolinitepredominates.Justice (2005)found out that Metakaolin (MK) is not a by-product of an industrial process, unlike other supplementary cementitious materials. However, it is produced under carefully controlled conditions for specific purposes. Metakaolin is produced by heating kaolin, one of the most abundant natural clay minerals, to temperatures of 650°C-900°C. During the heat treatment, or calcinations, the structure of kaolin is broken down. The bound hydroxyls are removed from the disorderliness of alumina and silica layers, thus a highly reactive amorphous material with pozzolanic properties are then produced.

Hui et al (2008) determined the phase transformation amounts of active Silicate ($SiO_2$) and Aluminate ($Al_2O_3$) in kaolin using different pre-treated temperature regimes. This was done with the use of Fourier Transformation Infrared (FT-IR), chemical analysis and nitrogen adsorption.The results showed that the surface area and pore volume of kaolin, calcined at medium temperature, was higher than those at low and high temperature. This is due to the fact that with increase in calcination temperature from 300°C to 900°C, the amount of active Aluminate ($Al_2O_3$) in the kaolin microspheres increased rapidly and then decreased steadily while that of the active silicate ($SiO_2$) increased slightly.

The chemical composition of the silicate ($SiO_2$), aluminate ($Al_2O_3$) and ferric ($Fe_2O_3$) summed up to 82.3% in finely ground calcined clay, when considered as pozzolan material (Dahl et al, 2007). The obtained pozzolan reduced alkali aggregate reaction, increased sulphate and chloride ingress resistance. Addition of 15% 5µm Micro Light Weight Aggregate in cement content will result to good resistance to high freezing and thawing cycles.

Having reviewed some of the various works carried out on the use of pozzolan as admixture in concrete and mortar mixes, it is evident that the introduction of pozzolan as cement replacement materials in recent years is widely acceptable. Metakaolin has not been in use in the Nigerian construction sector, apart from the fact that kaolin clay varies in physical properties and composition, depending on the environment. However, kaolin is in vast quantity in all the geopolitical zones of Nigeria – a reserve of two billion metric tons deposit. Further, the high cost of cement in developing countries coupled with the environmental pollutionhave called for investigation into its partial or full replacement in concrete.

## 2.0 Materials andExperimentalMethodology

### 2.1 Materials

Kaolin used in this study was obtained from vast open hilly land mass in Ewekoro, Ogun State area of Nigeria, where it occurs naturally. The light brown coloured kaolin samples naturally deposited in lumps had yellowish white streaks in it.

Cement used was Ordinary Portland Cement, with properties in accordance with BS 12 (1991). The aggregates were selected based on the limitation of BS 882. The fine aggregate used is natural river sand and the coarse aggregate is crushed granite stones, obtained from the quarry. The sizes for sand range from 75micron to 9.5 mm while for granite chippings, ranged between 2.0-19.0 mm. These materials were dried to control the water content in the concrete. The water used for this work is clean, clear and fit for drinking (portable water) which satisfies the BS 5328 standard for water requirements for mixing concrete.

## 2.2 ExperimentalMethodology

### 2.2.1 Determination of Chemical Composition of Metakaolin (MK)

Each kaolin sample was thermally treated at temperatures of $450^0$Cto$1050^0$C at interval of $150^0$C for duration of 30and60 minutes using Carbolite GPC 1200 oven to form metakaolin. After thermal treatment, the metakaolin was left to cool to room temperature and stored in plastic bags. The colour formation observed is as follows: at $450^0$C, light brown to slightly brown colour; at $600^0$C, light brown to brown colour; at $750^0$C, light brown to dark brown colour; at $900^0$C, light brown to slightly reddish brown colour and at $1050^0$C, light brown to reddish brown colour. Figure 1 shows kaolin samples exposed to different calcination temperature. The major and minor oxides present in the metakaolin samples were determined through chemical tests.


**Figure 1: Kaolin Samples with different calcination temperature**

### 2.2.2 Blain Fineness Value and Standard Consistency

The densities of metakaolin samples were checked on the scale of the blain meter cell volume ($1.889cm^3$) in accordance with NF EN 196-6 1990 to determine the mass of each sample to be used to determine its blain fineness value. The measured mass of the sample was put in the air permeability cell and compressed with a plunger. A filter paper was then placed on the cell unit and transferred to the Blain analyser. Finally, the Blain Fineness Value for the sample was read on the screen attached to the machine.Standard consistency and setting times (initial and final) of paste, made from each of the samples, were determined in accordance with BS 12-1991.

### 2.2.3 Compressive Strength of Metakaolin Concrete/Mortar

Six series of concrete mixes were considered in this study. The 150mm cube specimen were prepared with the water-cement ratio of 0.6 and mix design for the concrete grade $25N/mm^2$ to evaluate the mechanical properties for up to 60 days and records taken accordingly on 7, 28, 42 and 60 days respectively.

The first series (12 Nos concrete cube specimens) was the control mix, i.e. normal concrete, without metakaolin replacement. The second series (36 nos concrete cube specimens) was concrete with pre-defined replacement ratio with metakaolin (10%, 20% and 30%) treated at temperature of $450^0$C. The same methodology was applied to the third, fourth, fifth and sixth series of concrete for the study of the strength development of concrete containing different percentages of metakaolin at $600^0$C, $750^0$C, $900^0$C and $1050^0$C. A total of 192 cube specimens were cast. The effect of calcination on the strength of metakaolin-concrete was observed and slump test carried out to determine the consistency of concrete and to check its uniformity from batch to batch

To ascertain pozzolanity of metakaolin, 70mmsandcrete mortars cubes were prepared from each sample in the ratio 1:3 (pozzolan blended cement: standard sand) with a water/cement ratio of 0.4 as specified by BS 4550-3.4-1978. Compressive strength test was performed according to BS 1881: Part103:1983 on the mortar cubes at the ages of 1, 3, 7, 28 and 90 days.

## 3.0 Results and Discussion

### 3.1 Chemical Composition of Metakaolin (MK)

Table 1 shows the results of the chemical composition of calcined kaolin (metakaolin) at different temperatures for varied calcination duration, uncalcined and ordinary Portland cement. From observation, the silica content of the metakaolin increases with increase in both temperature and time of burning i.e. the amount of silica ranges from 49.15% ($600^0$C at 30mins) to 51.02% ($1050^0$C at 60mins). The results infer that temperature and the duration of burning have significant effect on the chemical composition of the metakaolin. Also, it can be observed that the maximum value of Loss on Ignition (LOI) i.e. maximum of 1.7% is far less than the maximum value 6% recommended by standards. Hence, all the resulting samples are suitable pozzolans (Shetty,2006). At higher temperature and longer time of calcination, silica content is with relative low LOI. Since amorphous silica is an essential requirement, observations made revealed that temperature of $750^0$C and 60 minutes calcination, producing 0.68% of LOI could be considered as appropriate conditions to produce amorphous silica.

The results of chemical composition of metakaolin also showed that it is comparable with standard (ASTMC618, 1993) and other materials as the combined silica, alumina and ferric content (85%) was above 70% and other criteria such as moisture content and LOI limits also indicates that metakaolin has pozzolanic potential.It is further revealed in Table 1 that the least silica content of metakaolin (42.50%) with varying calcined temperature is more than twice that of the ordinary Portland cement (19.05%), but ordinary Portland cement has greater value of calcium oxide content (63.45%), as compared to that of metakaolin (9.83%). The calcium oxide content in calcined kaolin decreases with increase in temperature. The high content of calcium oxide in OPC is the main reason for its cementitious characteristics over any known pozzolan.

**Table 1: Effects of Calcining Temperature on Chemical Composition of Kaolin**

| Temperature (°C) | Time of Calcination (min) | Oxides % | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | CaO | $SiO_2$ | $Al_20_3$ | $Fe_20_3$ | Mg0 | $Na_20$ | $K_20$ | $S0_3$ | LOI | TOTAL |
| 450 | **30** | 9.83 | 42.50 | 32.13 | 1.95 | 1.24 | 0.16 | 0.15 | 0.13 | 1.70 | **89.79** |
| | **60** | 9.75 | 46.51 | 29.00 | 1.87 | 1.14 | 0.15 | 0.15 | 0.10 | 1.67 | **90.34** |
| 600 | **30** | 9.21 | 45.09 | 31.26 | 1.08 | 1.09 | 0.13 | 0.12 | 0.12 | 1.69 | **89.79** |
| | **60** | 8.85 | 49.15 | 29.88 | 1.04 | 1.01 | 0.12 | 0.11 | 0.10 | 1.01 | **91.27** |
| 750 | **30** | 7.57 | 52.45 | 30.17 | 0.21 | 0.94 | 0.10 | 0.09 | 0.07 | 1.00 | **92.60** |
| | **60** | 7.27 | 54.35 | 29.50 | 0.00 | 0.92 | 0.11 | 0.11 | 0.08 | 0.68 | **93.02** |
| 900 | **30** | 5.91 | 46.31 | 26.80 | 0.12 | 0.80 | 0.07 | 0.06 | 0.55 | 0.60 | **81.22** |
| | **60** | 5.20 | 50.48 | 24.88 | 0.10 | 0.78 | 0.06 | 0.10 | 0.12 | 0.20 | **81.92** |
| 1050 | **30** | 6.61 | 46.81 | 25.60 | 0.04 | 0.66 | 0.05 | 0.15 | 0.11 | 0.30 | **80.33** |
| | **60** | 6.77 | 51.02 | 24.50 | 0.02 | 0.64 | 0.04 | 0.03 | 0.02 | 0.15 | **83.19** |
| **Uncalcined kaolin** | | 0.00 | 55.00 | 29.00 | 1.00 | 0.50 | 0.02 | 3.10 | 0.00 | 8.80 | **97.42** |
| **Ordinary Portland Cement** | | **63.58** | **19.05** | **4.98** | **0.64** | **1.96** | **0.75** | **0.43** | **0.50** | **0.018** | **91.91** |

### 3.2 Effects of Metakaolin on Physical Properties of OPC

The effects of metakaolin on fineness and specific gravity of cement was assessed. The results are shown in Table 2. The obtained Blain fineness value for metakaolin alone is 335$m^2$/kg and is lower than that of OPC (355$m^2$/kg). Substitution of metakaolin for cement at different proportion caused reduction in the surface area (fineness) and reduction in the Blain Value of the resulting cement accordingly.

The hydration process of Portland cement do not involve the complete dissolution of the cement grains; rather, the reactions take place between water and the exposed surfaces of the cement particles. The fineness of the cement has a considerable effect on its rate of reaction, as this will determine the surface area exposed to water. Thus, partial substitution of OPC with metakaolin (MK) will cause delay in hydration reaction. The effect of this, is that the strength development is slowed down. Cost of grinding and adiabatic temperature rise in finer cement, are limiting factors in the fineness of cement; metakaolincould be used as adiabatic temperature conditioner like any other pozzolanic material.

For the standard consistency test results, initial and final setting times for metakaolin blended cement pastes are also shown in Table 2. The trend shows that, as the MK content increases, more water will be required to produce consistence paste. For instance, when 25% metakaolin is used, about 35.5% water consistence is needed as against 27% used with OPC only. This could be attributed to the delay in rate of hydration reactions as a result of presence of MK. Similarly, metakaolin causes delay in setting times (initial and final) of cement paste. For example, 5% MK content has initial and final setting times of 128 and 202 minutes as against 110 and 193 minutes for normal Portland cement. It implies that MK can be used in arid environment and in mass concrete conditions.

**Table 2: Effects of Metakaolin on Physical Properties and Setting Times of OPC**

| % Content of Metakaolin | Blain Value ($m^2$/kg ) | Specific Gravity | Standard Consistency (%) | Setting time (min) | | Retardation Relative to Control, (min) | |
|---|---|---|---|---|---|---|---|
| | | | | Initial | Final | Initial | Final |
| **0** | 355 | 3.78 | 27.00 | 110 | 193 | 0 | 0 |
| **5** | 353 | 3.77 | 29.00 | 128 | 202 | 18 | 9 |
| **10** | 347 | 3.74 | 31.00 | 142 | 223 | 32 | 30 |
| **15** | 342 | 3.72 | 32.50 | 159 | 224 | 49 | 31 |
| **20** | 338 | 3.69 | 34.00 | 217 | 255 | 107 | 62 |
| **25** | 337 | 3.67 | 35.50 | 256 | 295 | 146 | 102 |

Table 3 shows that the addition of metakaolin in various quantities of 10%, 20% and 30% of cement and temperature over the range 450-1050$^0$C affects the workability favourably. The workability improved with increase in the percentage of MK content. On the average, the different calcination temperatures affected the workability in the form: 1050$^0$C resulted in high degree of workability with MK content more than 10%, 900$^0$C resulted in low degree of workability with MK content less than 30%. 450$^0$C, 600$^0$C and 750$^0$C resulted in medium degree of workability in all cases of MK content in concrete. Slump appeared to be true in all cases for metakaolin-concrete. Normal concrete had shear slump. Average compacting factor of 0.90 was recorded for all samples.

**Table 3: Workability, Slump and Compacting Factor of MK-Concrete.**

| % MK | Calcination Temperature | Slump (mm) | Slump Type | Degree of Workability | Compacting Factor |
|---|---|---|---|---|---|
| 10 | 450°C | 25.00 | true | very low | 0.78 |
| | 600°C | 67.00 | true | medium | 0.92 |
| | 750°C | 100.00 | true | high | 0.95 |
| | 900°C | 30.00 | true | low | 0.85 |
| | 1050°C | 50.00 | true | medium | 0.92 |
| 20 | 450°C | 52.50 | true | medium | 0.92 |
| | 600°C | 75.00 | true | medium | 0.92 |
| | 750°C | 75.00 | true | medium | 0.92 |
| | 900°C | 45.00 | true | low | 0.85 |
| | 1050°C | 115.00 | true | high | 0.95 |
| 30 | 450°C | 75.00 | true | medium | 0.92 |
| | 600°C | 83.00 | true | medium | 0.92 |
| | 750°C | 50.00 | true | medium | 0.92 |
| | 900°C | 50.00 | true | medium | 0.92 |
| | 1050°C | 125.00 | true | high | 0.95 |
| **Normal Concrete** | | **150.00** | **shear** | **150.00** | **1.95** |

### 3.3 Pozzolanic Activity

For a cement to be considered pozzolanic as per ASTM C618, it must exhibit a strength index greater than 75% after 28 days. This index is calculated as the ratio of the compressive strength of a mortar with 20% added metakaolin to that of a reference mortar with no additional of metakaolin (ASTM C311). The kaolin samples heated at temperatures up to 750$^0$C exhibit a strength index of 94% after 28 days curing.

The results of strength development of mortar cubes show that the strength development of the blended mortar cubes is relatively close to the control. This can be clearly revealed with 5% and 15% replacements achieving compressive strength of 19.17 MPa and 17.72 MPa respectively compared to 19.32 MPa of the control at 28 days. The 15% replacement also exhibits similar strength development as the control. Compared to 5% replacement, the amount of metakaolinite exists in the metakaolin-blended mortar cubes are probably too high. Likely, the quantity of calcium hydroxide, produced from the hydration of cement, is not enough to react with all the metakaolinite to produce extra CSH. The calcium hydroxide has been reduced to the minimum level while some metakaolinite are left out without any chemical reaction.

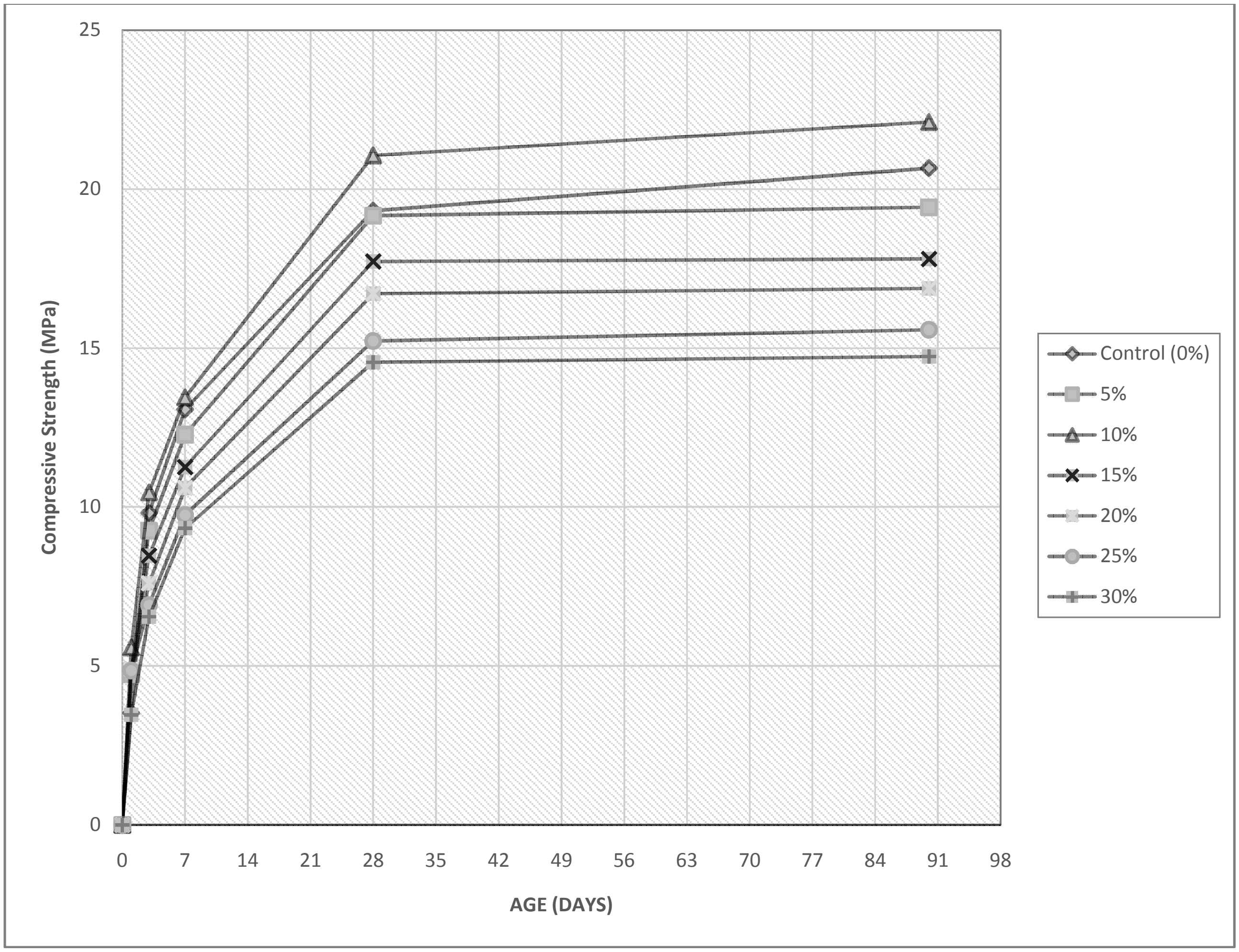


**Figure 2: Compressive Strength of different Metakaolin (MK) Blended Mortar**

The mortar cubes with 10% replacement exhibits the best strength performance in this study. The strength development for concrete with 10% replacement appeared even higher than the normal mortar cubes (the control) as shown in Figure 2. The compressive strength of mortar cubes with 20%, 25% and 30% replacement are generally lower than the control at all test ages. The development of strength with age is consistent in all cases.

It is known that kaolin has the particle size ranging from 0.2 – 15 microns with the specific area of 10000 – 29000 $m^2$/kg which is much finer than cement. These finely divided cement replacement materials have a physical effect in that they behave as fillers. At the cement paste-aggregate particle interface, they produced more efficient packing, causing the concrete to be more homogenous and denser. There is a reduction in the amount of bleeding, resulting in reduced initial and narrower transition zone microstructure. *Zhang et al (1995)* reported that achieving higher compressive strength and reduced porosity, reduced calcium hydroxide content and reduced width of the interfacial zone between the paste and the aggregate. Metakaolin has demonstrated the same attributes as it also contributes to latter strength development of mortar. Partial replacement by metakaolin results an increase in the strength of concrete, possibly due to an improved transition zone.

Metakaolin rapidly removes calcium hydroxide i.e. $Ca(OH)_2$ from the system and accelerates the ordinary Portland cement (OPC) hydration. The hydration of cement is accelerated by the presence of particles of metakaolin which acted as nucleation site for the reaction products (calcium hydroxide). However, the results show that the strength development is retarded at the third day. The compressive strength of all mixes except 10% replacement is lower than the control. This is generally caused by the "dilution effect", which refers to the lowering of initial hydraulic reactivity, subsequently improving homogeneity by way of reducing coagulation and increasing dispersion of clinker particles resulting into improved hydration process. As the replacement rations exceed 10%, the amount of metakaolinite is in excess to react with calcium hydroxide. These extra metakaolinite produce an immediate dilution effect such that the water-cement ratio is reduced. Concrete strength is reduced in approximate proportion to the degree of replacement. Against this backdrop, the 30% replacement endures the most critical strength loss.

### 3.4 Effects of Metakaolin on Compressive Strength of Concrete

The presence of metakaolin (MK) in the concrete leads to a decrease in the strength values with the increase in MK content for the first 28 days of curing. The compressive strength reduces from 18.85MPa (0% MK) to 13.87MPa (for $750^0$C calcination and 10% cement replacement) and 25.95MPa (0% MK) to 25.73MPa (for $750^0$C calcination and 10% cement replacement) at 7 and 28 days curing ages.The MK content increases the volume content of tricalcium aluminate ($C_3A$),resulting into retardation in concrete setting.Thisis in consonance with the work of Hui et al, (2008), where with increase in the calcination temperature, the amount of active Aluminate ($Al_2O_3$) increased rapidly and later reduced steadily while the active Silicate($SiO_2$) in kaolin microspheres increased slightly.

At 60 days of curing, the compressive strength of the metakaolin-concrete cubes produced from 10%, 20% and 30% MK were 27.35, 24.94 and 21.35MPa respectively which are 96.9%, 88.4% and 75.7% of normal concrete. This shows the significance of pozzolanic effects as the strength increases with age or at longer period of curing. However, Figure 3 shows that the optimum compressive strength for different percentages of metakaolin at different ages at a temperature of 750$^0$C; except for 7 days where the process of pozzolanity has just commenced.

**Table 4: Compressive Strength of Metakaolin-Concrete with Age and Temperature of Calcination.**

| % MK | Calcination Temperature (°C) | Average Strength at Age of Concrete (MPa) | | | |
|---|---|---|---|---|---|
| | | 7 days | 28 days | 42 days | 60 days |
| 10 | 0 (uncalcined) | 8.44 | 15.84 | 16.08 | 16.88 |
| | 450 | 12.90 | 22.55 | 23.65 | 25.52 |
| | 600 | 12.95 | 23.24 | 24.46 | 25.90 |
| | 750 | 13.87 | 25.73 | 26.96 | 27.35 |
| | 900 | 18.00 | 20.65 | 22.03 | 26.03 |
| | 1050 | 18.85 | 20.00 | 21.74 | 25.52 |
| 20 | 0 (uncalcined) | 6.19 | 12.39 | 13.61 | 13.74 |
| | 450 | 9.86 | 21.88 | 21.90 | 22.29 |
| | 600 | 10.50 | 22.46 | 22.61 | 22.90 |
| | 750 | 12.00 | 23.54 | 23.88 | 24.94 |
| | 900 | 16.61 | 18.46 | 20.86 | 22.46 |
| | 1050 | 16.63 | 17.54 | 20.00 | 23.59 |
| 30 | 0 (uncalcined) | 4.50 | 9.66 | 11.57 | 11.00 |
| | 450 | 6.48 | 18.98 | 18.61 | 17.71 |
| | 600 | 6.93 | 19.02 | 18.96 | 18.96 |
| | 750 | 8.17 | 20.92 | 19.17 | 21.35 |
| | 900 | 15.07 | 19.14 | 17.55 | 16.94 |
| | 1050 | 15.11 | 18.44 | 17.13 | 16.83 |
| **Normal Concrete** | | **18.85** | **25.95** | **27.13** | **28.22** |

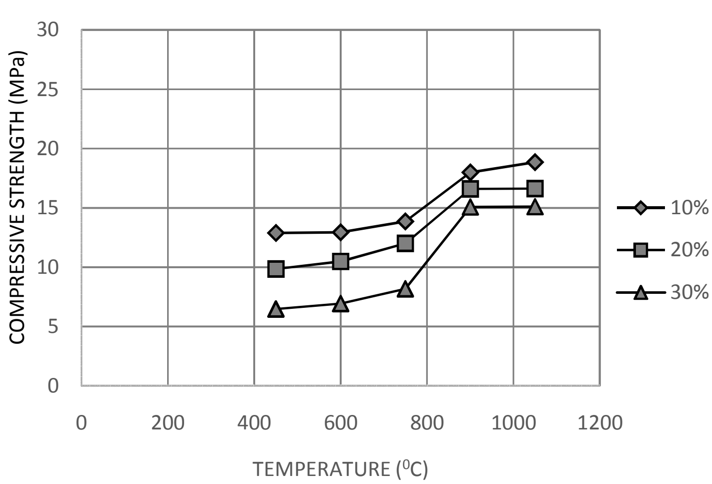


Figure 3(a): Compressive Strengths of Metakaolin-Concrete at Different Calcination Temperatures at 7 days Curing Period

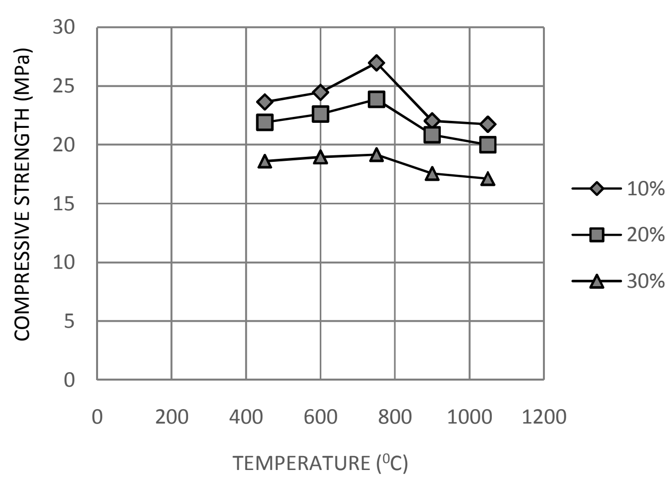


Figure 3(c): Compressive Strengths of Metakaolin-Concrete at Different Calcination Temperatures at 42 days Curing Period

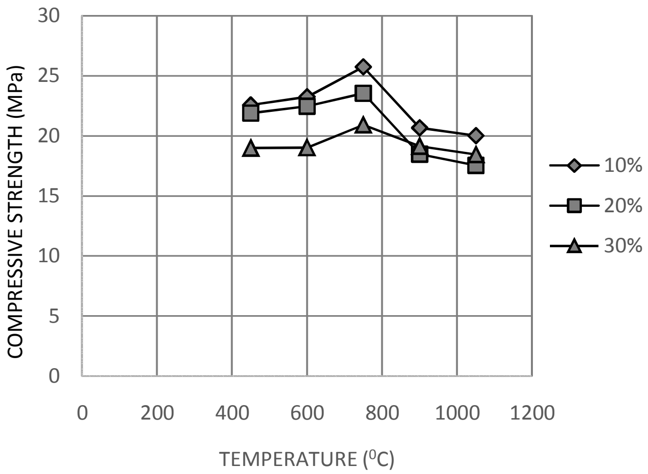


Figure 3(b): Compressive Strengths of Metakaolin-Concrete at Different Calcination Temperatures at 28 days Curing Period

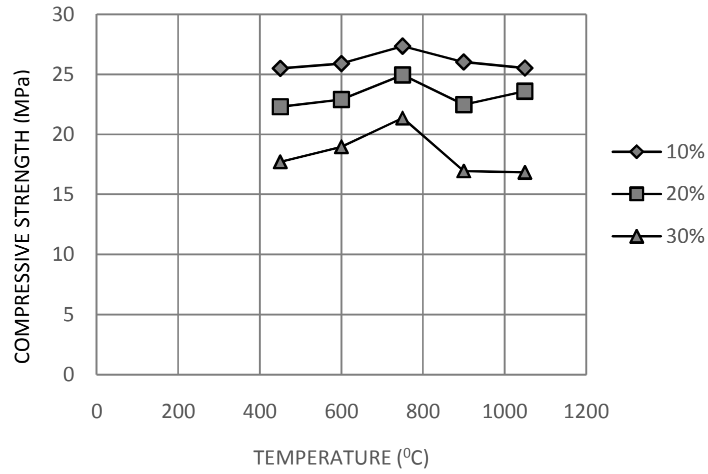


Figure 3(d): Compressive Strengths of Metakaolin-Concrete at Different Calcination Temperatures at 60 days Curing Period

### 3.5 Effectof Calcination Temperature on Metakaolin-Concrete

Calcination is needed to improve the performance of kaolin, converting it to metakaolin. Through calcination, kaolin will become reactive with calcium hydroxide to enhance the strength of concrete.The results of compression test for varying cement replacement are shown in Table 4, while Figure 4 shows the strength performance of 10% MK calcined at different temperature. Metakaolin at 750$^0$C has the best performance among other samples. At the 7th day, metakaolin-concrete at 750$^0$C reached strength of 13.87 MPa. After that, the strength developed to 25.73 MPa on the 28th day, 26.96 MPa on the forty-second day and reaches an ultimate strength of 27.35 MPa on the 60 days. Contrast to the well-performed metakaolin at 750$^0$C, metakaolin at 450$^0$C is the least performed metakaolin. It only manages to attain strength of 13.39 MPa at the seventh day. The further strength development for metakaolin-concrete at 450$^0$C is 22.55 MPa (28 days), 23.65 MPa (42 days) and 25.50 MPa (60 days).

Sabir et al (2001) studied that the burning or calcining temperature of clays is crucial and affects the pozzolanic reactivity of the resulting product. The calcining temperature, producing the active state, is usually in the range of 600-800°C. Similarly, in this paper, starting from 450$^0$C to 750$^0$C, the compressive strength of the concrete increases with the rise in calcination temperature until the optimum temperature of 750$^0$C. After the calcination temperature increased to 900$^0$C, the concrete strength begins to drop. Figure 4 shows the strength performance with 10%MK calcined at different temperatures.

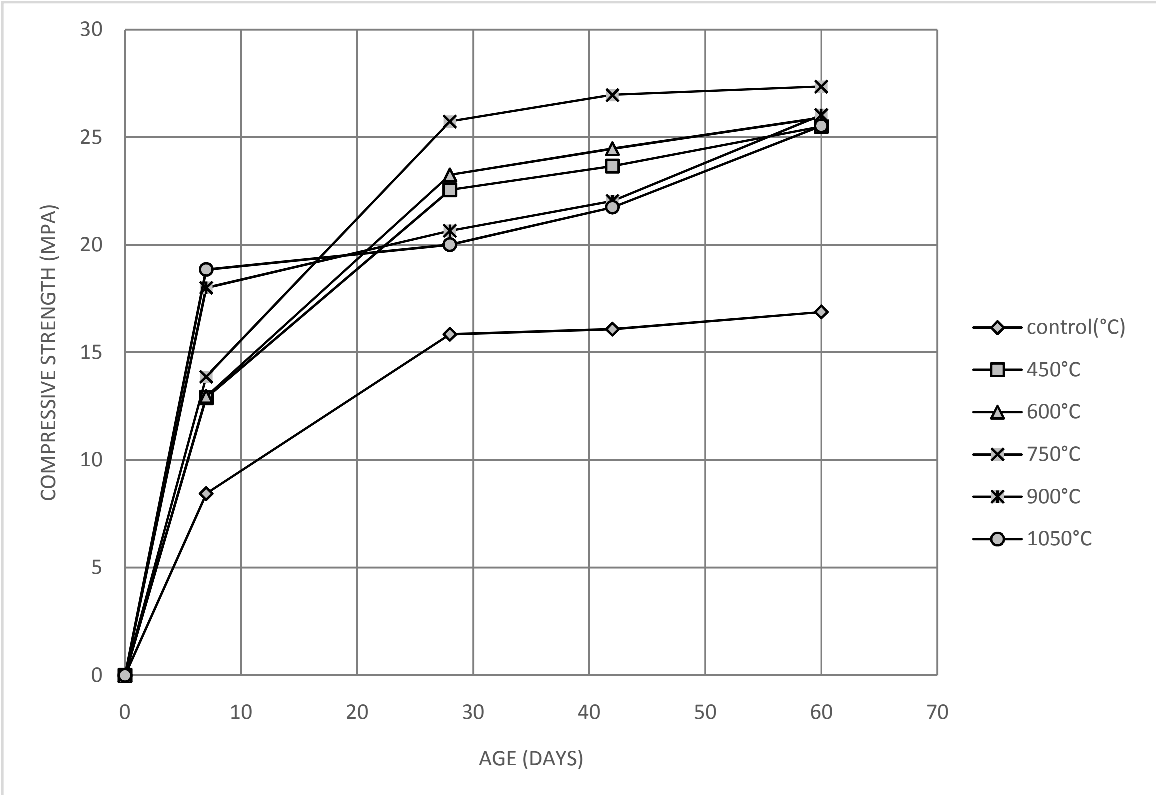


**Figure 4: Strength Performance with 10% MK Calcined at Different Calcination Temperature.**

Other than the amount of metakaolinite, the calcination temperature also affects the reactivity of metakaolinite. From the study done by Kakali et al. (2001), metakaolinite has a highly disordered structure and reacts particularly well with lime and forms, in the presence of water hydrate compounds of Calcium and Aluminium Silicates. However, the amorphous metakaolinite of high surface area is actually the result of the thermal treatment. The calcination temperatures determine the degree of disorder of metakaolinite and hence affect its chemical reactivity.

Increased calcination temperature results in higher content of metakaolinite with higher reactivity. Metakaolin at $750^0$C has higher early and ultimate strength than metakaolin at $600^0$C. The improved performance is not proportional to the temperature increment, where negligible improvement is observed for temperature of $450^0$C and $600^0$C (i.e. 25.50 MPa to 25.59 MPa at 60days) but the improvement within temperature $600^0$C and $750^0$C (i.e. 25.59 MPa to 27.35 MPa at 60days) is more conspicuous. This indicates that the performance of metakaolin is not linearly proportional to the calcination temperatures.

When the calcination temperature reaches $750^0C$, the peak performance of metakaolin-concrete is achieved. Thus, based on the experimental data, the temperature of $750^0C$ is the optimum calcination temperature for this study. At this stage, the metakaolin has a maximum amount of metakaolinite and also the highest chemical reactivity. This enables it to have the highest early and ultimate strength, about 13.87 MPa or 10.7% higher than metakaolin at $600^0C$ at 28 days.

As the calcination temperature is increased to $900^0C$ and above, it is observed that the strength of the concrete declines. This is as a result of the recrystallization which occurs when kaolin is heated at the temperature above the optimum temperature, hence causing a reduction in the amount and reactivity of metakaolinite. In this condition, kaolin activity is reduced.

## 4.0 Conclusions & Recommendations

Based on the results of our investigations, the following conclusions could be made:

i. Different colour formations were observed for different granular kaolin samples at different calcination temperatures. Metakaolin in cement paste causes reduction in rate of hydration and retard setting times, as the fineness of blended cement is slightly reduced. On the average, $750^0C$ resulted in medium degree of workability (true slump) in all cases of MK content in concrete. Water-binder ratio of 0.5 may be appropriate

ii. Metakaolin mortar exhibits a strength index greater than 75% after 28 days in conformity with ASTM C618. Kaolin samples heated at temperatures between 600 and $750^0c$ exhibit a strength index of 94%. Hence, metakaolin is a pozzolan.

iii. The compressive test results shows that concrete reaches highest strength at early ages (1-7 days) at the calcining temperature of $1050^0C$ due to the favourable chemical condition that is promoted by the high content of silica ($SiO_2$). At the $1050^0C$ and $450^0C$, the percentages of silica content ($SiO_2$) are 51.02% and 46.51% respectively.

iv. Compressive strengths of concrete and mortar cubes for different replacement ration of metakaolin at 28 days and 60/90 days respectively are comparable to their control

v. The optimum temperature among the range applied in this study i.e. $450^0C$, $600^0C$, $750^0C$, $900^0C$ and $1050^0C$ is $750^0C$ because the highest strength of metakaolin concrete and MK blended mortar cube for 28 days, 25.73MPa and 21.06MPa respectively were reached at $750^0C$ and 10% optimum percentage substitute of metakaolin.

Based on the results obtained in this work, it is recommended that further investigation be carried out to determine the effect of metakaolin Particle size distribution on the concrete strength, while it is important to study the different behaviour of metakaolin-concrete, calcined from different kaolin(s) to look into the effect of geographic area and bedrock on the compositions of the clay minerals.

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