Assessment of Isan-Ekiti Metakaolin as a Pozzolan in Concrete

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ABSTRACT: The continuous search for alternative binding materials in place of cement has led researchers into looking for both artificial and naturally occurring materials that can be used to partially replace cement in the construction industry. Materials such as slag, fly ash, rice husk ash, wood ash and corncob ash which have cementitious properties, have been investigated, while works have been intensified on the use of metakaolin as cementitious materials in the last decades. This work looked into the use of metakaolin from the Isan-Ekiti kaolin deposit as a cementitious material. Chemical, slump and mechanical tests were conducted on the calcinated kaolin that gave the metakaolin. The chemical result showed that the metakaolin has pozzolanic characteristics and is classified as class “N” pozzolan. The mechanical tests revealed that cement can be partially replaced in concrete with metakaolin for up to 20% by weight.

Keywords: Metakaolin, Calcinated, Pozzolan, Cement, Mechanical test.

INTRODUCTION

The use of Portland cement concrete for various construction works in recent time has risen to millions of tons per year. Efforts are on to reduce the use of Portland cement in concrete production because its production is one of the major reasons for CO$_2$ emission into the atmosphere. A tonne of Portland cement produced is accompanied by the emission of 1 tonne of carbon dioxide (CO$_2$) which is a greenhouse gas, into the atmosphere, (Ramlochan et al., 2013).

Recently, there is an increase in the utilization of pozzolanic supplementary cementitious materials (for instance silica fume, fly ash, slag and rice husk ash) in cement concrete production because these materials do not only help to bring about reduction in the use of cement but also reduce the cost of construction, its environmental impact due to CO$_2$ emission during the production of cement and enhance both the strength and durability properties of concrete. Concrete made with such pozzolanic materials as supplementary cementitious materials (SCMs) with silicon and aluminium as the main constituents are eco-friendly and they enhance the strength of concrete (Badogiannis et al., 2002).

The term pozzolan can be defined as siliceous material, which are in finely divided form and chemically react with calcium hydroxide in the presence of water to form cementitious compounds (Justice, 2005).

The growth in demand for Portland cement concrete has led to the escalation in its production costs with its basic materials like Portland cement and aggregates becoming increasingly very expensive to obtain because of the huge costs incurred in the Portland cement production, aggregates sourcing, sand excavation process, pretreatment and transportation (Romer, et al., 2011).

Kaolin is one of the industrial minerals that can be found in commercial quantities in Nigeria. It was estimated by Raw Material Research Council...
of Nigeria (RMRDC) that the country has a reserve of about (3) three billion metric tonnes of kaolin deposit scattered in difference parts of the country which includes Ogun, Edo, Plateau, Nassarawa, Katsina, Ekiti, Kogi, Abia, Kano, Niger, Bauchi, Sokoto, Kaduna, Oyo, Delta, and Borno states. The market for kaolin is large, sustainable and expanding because of the numerous applications of its products. Good prospects exist in kaolin mining and prospecting in Nigeria (RMRDC (TB) 2008).

According to Vikas et al (2012), Metakaolin is a waste/non-conventional material which can be utilized beneficially in the construction industry. The advantages of this material as partial replacement of cement in concrete cannot be under estimated. Metakaolin may be used as a cement replacing material in concrete, to reduce cement consumption, to increase strength and the rate of strength gain, to decrease permeability and to improve durability (Khatib and Wild, 1998; Aquino et al., 2001; Asbridge et al., 2001; Boddy et al., 2001; Justice, et al., 2005).

Niveditha and Manivel, (2014) in their work reported that metakaolin produced by calcination of pure kaolinite clay can greatly influence both the mechanical and ductility properties of concrete. Biljana et al., (2010), also reported that the development of pozzolanic reactivity in fired kaoline clay, measured by indirect method based on strength development occurring with reaction time, which is the main characteristics of the produced metakaolin, depends on the nature and abundance of the kaolinites in the kaolin clay materials. Velosa et al (2009), in their study on influence of chemical and mineralogical composition of metakaolin on concrete characteristics, reported that concrete using metakaolin is rich in Si02 and Al2O3, and being also poor in alkali but showed better mechanical results.

The aim of this research is to investigate the use of locally available calcinated kaolin (metakaolin) of Isan-Ekiti as cementitious material, by partially replacing cement in concrete at certain percentages.

**MATERIALS AND METHOD**

**Calcination of Kaolin samples**

The Isan-Ekiti Kaolin was grinded to powder using local grinding machine. The kaolin powder was fired (calcinated) at Federal Institute of Industrial Research Oshodi (FIIRO), Lagos State, Nigeria to form Metakaolin. The chemical and mineralogical analysis tests on Kaolin and the Metakaolin were carried out. The calcination was based on the recommendation of ASTM C 618-12,(2008).

Prior to the calcinations of the kaolin clay material, Twenty five (25) fire treated pots with each having the capacity to hold at least 2.5 kg of kaolin clay powder at a time were produced from clay mineral soil materials at FIIRO laboratory. The pots having been molded were allowed to solidify for 7 days before being fire treated and soaked at 1000°C for 3 hours and allowed to cool down at room temperature to ensure that the pots were strong enough to resist high temperature usage. About 2 kg of kaolin clay was measured into the fire treated pot and placed inside the electrically fired Kiln and fired to 700°C and soaked (maintained) for one hour after which the furnace (kiln) was switched off and the resulted metakaolin brought out and allowed to cool at room temperature. The resultant residue is a highly reactive Alunino-Silicate pozzolanic material rich in Silica and Alumina. After cooling, the resultant metakaolin powder Plates 1, 2 and 3 was further ground, sieved, passed through 150 µm and retained in 100 µm sieve size and sample of it was taken to Ojuolape Laboratory at Ring road, Ibadan, Nigeria for chemical analysis.

**Slump test**

The metakaolin formed from the calcination of kaolin was used to replace cement in concrete samples at 0%, 5%, 10%, 15%, 20%, 25%, 30%,
35%, and these were designated as $S_0$, $S_5$, $S_{10}$, $S_{15}$, $S_{20}$, $S_{25}$, $S_{30}$, and $S_{35}$, respectively. Slump test which gives the indication of the quality of concrete with respect to consistency was carried out according to BS.1881-2: (1996) to measure the workability of all concrete specimen mixtures for this work. The apparatus used for the test included a mould (which is a frustum cone 300 mm high with 100 mm diameter opening at the top and base opening of 200 mm diameter). The mould was cleaned, lubricated and firmly held on its base plate (600 mm by 600 mm square), and was filled with concrete in three layers with each layer tamped 25 times with 16 mm diameter steel rod. The cone surface leveled up with the steel rod was slightly raised vertically by the handles and allowed the slipping of the concrete in 10 seconds. The cone was turned upside down and the rod placed across the upturned cone. The slump was measured by subtracting the height of the mould and that of the highest point of the subsided concrete.

Splitting tensile test
Another 48 cylindrical concrete specimens were cast to determine the splitting tensile strength of concrete in line with BS EN. 12390-6 (2009), cylinder specimens of size 150 mm diameter × 300 mm length with mix proportion of 1: 2: 4 at water-cement ratio of 0.5 having 0, 5, 10, 15, 20, 25, 30 and 35 % metakaolin replacement for cement with curing ages of 28 and 90 days were cast for split tensile strength test. The constituent materials having been proportioned using weight batching and thoroughly mixed using mobile mixer was filled into the already prepared cylinder in three layers. Each layer was tamped 25 times with the tamping rod in accordance with the code. The mould with the content were covered with polythene bags for 24 hours, the specimens were demoulded after 24 hours of cast and were transferred to the curing tank where they were allowed to cure for the test ages (28 and 90 days). The cylinder specimens were allowed to dry and
crushed at loading rate of 100 kN/m by using a compressive testing machine (Plate 4).

In each batch, three cylinder specimens each were removed from the varying percentages at the end of each age (28 and 90 days) air dried for two hours in the laboratory and the weights determined with the digital weight balance for their densities before the splitting tensile strength.

Split tensile strengths were calculated using equation 1 below:

\[ \text{Split Tensile (N/mm}^2) = \frac{2P}{\pi dl} \]  

Where \( P \) = Failure Load kN, \( d \) = Diameter of cylinder, \( l \) = Length of cylinder

**Compressive strength test**

All concrete cube specimens of size 150 mm x 150 mm x 150 mm were prepared in line with BS EN 12390-3 (2009). Concrete samples designated as \( S_{0}, S_{5}, S_{10}, S_{15}, S_{20}, S_{25}, S_{30}, S_{35} \) were cured for 7, 14, and 28 days in water at room temperature of 23 ± 2 °C. Water-cement ratio of 0.5, was used in the production of various concrete cube specimens for the compressive strength test. The moulds of cube size 150 x 150 x 150 mm were prepared (lubricated with oil) and each mould for the cube was filled with concrete in three layers using a trowel. Each layer was compacted using 16 mm diameter tamping rod to tamp 25 times. The specimens were then demoulded after 24 hours and transferred into a curing tank containing clean water where they were stored till the respective testing ages at curing temperature of 20 ± 1°C.

Three cubes each were removed from the varying percentages at the end of each age, air dried for two hours in the laboratory and the weights determined with the weight balance for their densities before testing for their compressive strengths using compressive testing machine at loading rate of 405 kN/m. The result of the compressive strength tests carried out on all the concrete cubes at the various ages and their densities were then recorded.

**Crack width measurement**

A total of 48 reinforced concrete beams of size 150 mm x 150 mm x 500 mm were cast with 12 mm diameter high yield steel reinforcing bars, in line with BS EN 12390-3: (2009) for determination of modulus of rupture and crack width after 28 and 90 days. The beam specimens were tested using third point loading method; this test was mainly to help determine the size and pattern of cracks formed by various specimen of metakaolin concrete. Three specimens were tested for each batch, the average of the loads at which the specimen failed was determined and experimental crack width at failure load was measured, while maximum theoretical crack widths at service load were determined by analysis of section using equation 2 according to BS 8110.

\[ w_{\text{max}} = \frac{3a_{cr} \varepsilon_m}{1 + 2 \left( \frac{a_{cr} - c_{\text{min}}}{h \times x} \right)} \]  

Where: \( a_{cr} \) = the average spacing between reinforcements \( \varepsilon_m \) = average concrete strain \( c_{\text{min}} \) = the minimum cover to reinforcement \( h \) = the depth of beam \( x \) = the neutral axis.

**RESULTS AND DISCUSSION**

**Chemical Composition of Metakaolin**

The chemical composition analysis of Metakaolin revealed major oxides of Silicon (Si), Aluminum (Al), Ferrous iron (Fe), and Calcium (Ca), which are the major components of pozzolans, provided that their percentage composition is more than 70% in a cementitious materials, this is according to ASTM C618-84. From Table 1, the combination of these oxides, \((\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3)\) is equal to 95.64%, this is...
more than the 70% recommended for pozzolans, and it can be classified as class “N” pozzolan because the metakaolin material is naturally occurring.

Therefore it is evident that the metakaolin produced from kaolin sourced from Isan-Ekiti in Ekiti State of Nigeria, which was used for this work can be said to be a pozzolan on the basis of chemical composition and can be used as supplementary cementitious materials (SCMs) for cement replacement in concrete production.

**Slump test result**

The mixture consistency based on the slump test record in Table 2 showed that the concrete mixes were plastic and the workability of the mixtures decreased by replacing cement with metakaolin. The slump height for the control concrete specimens was 91 mm and the slump for $S_{20}$ was 64 mm which was 27 % lower than the control specimen. Also the slump for $S_{35}$ was 54 mm which was 37 % lower than the control concrete specimen. Thus metakaolin addition stiffened the concrete mixture and therefore lowered the workability of the mixture; the concrete became less workable as the metakaolin content increased. The reduction in workability was likely due to its larger surface area which is an indication that metakaolin concrete mixtures require superplasticizer to achieve adequate concrete workability.

Wild et al. (1996) reported that metakaolin addition as supplementary cementitious materials yielded concrete with considerably higher strength over concrete without supplementary cementitious materials (SCMs) and that concrete mixtures containing metakaolin required some dosage of superplasticizer to achieve the required workability of normal consistency and target slump of 76 – 102 mm. The result is also in agreement with the reports of Vikas et al (2012) and Quian and Li (2001). The result from the table showed that the entire samples falls within medium consistency target of 25-100 mm, according to Neville (1990), this mixture can be used in concrete of manually compacted flat slab using crushed aggregate, normally reinforced concrete manually compacted and heavily reinforced sections with vibration.

**Compressive strength test result**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Slump height $H_1$ (mm)</th>
<th>Test 1 (mm)</th>
<th>Test 2 (mm)</th>
<th>Test 3 (mm)</th>
<th>Average Slump $H_2$ (mm)</th>
<th>Slump Value $H_1$-$H_2$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$S_0$</td>
<td>300</td>
<td>207</td>
<td>208</td>
<td>211</td>
<td>209</td>
<td>91</td>
</tr>
<tr>
<td>$S_5$</td>
<td>300</td>
<td>218</td>
<td>219</td>
<td>220</td>
<td>219</td>
<td>81</td>
</tr>
<tr>
<td>$S_{10}$</td>
<td>300</td>
<td>228</td>
<td>227</td>
<td>228</td>
<td>227</td>
<td>73</td>
</tr>
<tr>
<td>$S_{15}$</td>
<td>300</td>
<td>231</td>
<td>229</td>
<td>233</td>
<td>231</td>
<td>69</td>
</tr>
<tr>
<td>$S_{20}$</td>
<td>300</td>
<td>237</td>
<td>236</td>
<td>235</td>
<td>236</td>
<td>64</td>
</tr>
<tr>
<td>$S_{25}$</td>
<td>300</td>
<td>241</td>
<td>240</td>
<td>236</td>
<td>239</td>
<td>61</td>
</tr>
<tr>
<td>$S_{30}$</td>
<td>300</td>
<td>243</td>
<td>242</td>
<td>241</td>
<td>242</td>
<td>58</td>
</tr>
<tr>
<td>$S_{35}$</td>
<td>300</td>
<td>246</td>
<td>245</td>
<td>244</td>
<td>245</td>
<td>54</td>
</tr>
</tbody>
</table>
The compressive strength is the most important test in concrete, (Shetty, 2009). The test determines the strength of the concrete, and the result obtain from this test will help determine the usefulness or other wise of any concrete sample.

The result of the compressive strength test carried out on 150 mm concrete cubes at ages 7, 14, 28 days are shown in Figure 1. An increase in compressive strength of concrete as compared to the control concrete was observed for all metakaolin inclusion specimens ($S_{5}$ to $S_{35}$). Specifically, it was found out from the result of metakaolin inclusion (from 5% and up to 35%) that the compressive strength at 28 days increased in the range of 12.40 % to 36.58 % higher than the control concrete. The compressive strength of sample $S_{20}$ at 28 days was 33.38 N/mm$^2$ compared to 24.44 N/mm$^2$ for $S_{0}$ concrete sample, which is 36.58 % when compared to the control concrete. The result obtained here showed that the maximum strength was achieved at 20 % replacement level at all the tested ages.

The compressive strength result is in agreement with what was reported by Wild et al. (1996), they tested concrete at 5% to 30% metakaolin inclusion produced at 0.45 water-cement ratio from one to ninety days and found that metakaolin produced significant (15 % to 50 %) compressive strength increase in concrete strength compared to control concrete. Meanwhile Justice and Kurtis (2007) reported that increased compressive strength by 15-50% (depending on metakaolin type, w/c and age) as compared to control mixture was estimated for concretes produced with metakaolin.

Wild et al (1996) also reported that there are three basic factors influencing the contribution that metakaolin makes to strength when it partially replaces cement in concrete. These are the filler effect, the acceleration of Portland cement hydration, and the pozzolanic reaction of metakaolin with calcium hydroxide in cement paste. The reduction in strength after 20% replacement can be attributed to the filler effect, this is because the pozzolanic reaction of metakaolin with calcium hydroxide was very effective at lower cement replacement, and hence the high strength recorded at this level.

The addition of excess metakaolin above the 20% replacement make the chemical reactions to become passive or less reactive, this led to the filler effect at the aggregate paste interface and this encourages paste packaging and the reduction

**Split Tensile Strength Test Result**

The result of the split tensile strength test carried out on the cylinder concrete specimens at curing ages of 28 and 90 days are presented in Figure 2. The increment in tensile strength was similar to that observed in compressive strength test. As the compressive strength increased the tensile strength also increased. The splitting tensile strength for metakaolin inclusion concrete ranged between 2.18 and 3.14 N/mm$^2$ at 28 days higher than 2.15 N/mm$^2$ of the control concrete and 2.66 and 3.28 N/mm$^2$ at 90 days higher than 2.58 N/mm$^2$ of the control concrete. At 20% metakaolin inclusion, the split tensile strength test at 28 and 90 days increased by 27.13% (3.28 N/mm$^2$) and 40.05% (3.14 N/mm$^2$) respectively above the control concrete of 2.15 N/mm$^2$ and 2.58 N/mm$^2$. The maximum development of tensile strength was achieved at 20 % replacement level at the test ages (28 and 90 days) Vikas et al. (2012), reported that the tensile strength of concrete increases systematically with increasing metakaolin replacement level, they reported the tensile increase of 16 % (10 % metakaolin replacement) and 28 % (15 % metakaolin replacement) at 28 days curing age.

**Crack Width Results**

When structural members are subjected to flexural bending, they generally exhibit a series of distributed flexural cracks, even at service load.
These cracks are not harmful and cannot cause damage to the structures unless the crack widths become excessive, Mosley et al. (2007). Excessive wide cracks can be very unsightly and spoil the aesthetics of concrete structures; they can allow the ingress of water and encourage the rapid corrosion of reinforcement with durability failure. In some cases they can reduce the contribution of concrete to the shear strength of a member, (Gilbert 2001). Cracks normally becomes visible to the naked eye when the width is around 0.005 mm. The maximum crack width permitted for a structure at service load is between the ranges of 0.25 mm to 0.38 mm, Mosley et al. (2007) although larger crack width can still be tolerated, but anything above this width may expose the
structures to weather elements that may cause eventual failure of the structure. Table 3 shows both the experimental and theoretical crack width value for all the concrete samples at 28 and 90 days. The experimental crack width at 28 and 90 days generally increases as the amount of metakaolin added to the sample increases, the control sample \( S_0 \) has a crack width of 1.20 mm and 1.10 mm respectively which is the lowest, but the \( S_{35} \) samples have the highest crack width of 2.60 mm and 3.10 mm for 28 and 90 days respectively. It should be noted that these crack widths were measured at failure load. The theoretical crack width that was determined from equation gave an estimated value for the crack width at service load. All the theoretical crack width obtained were below the maximum crack width permitted at service (0.25 – 0.38 mm), hence it can be said that the replacement of cement with metakaolin in concrete may not have much negative effect on the structures crack width during service, although the crack width also increases as more metakaolin were added, but peaked at \( S_{20} \) for both 28 and 90 days and then took a reduction after 20% replacement. It can also be noted that the crack width increases as the curing age of the concrete samples increases.

<table>
<thead>
<tr>
<th>Sample</th>
<th>28 Days Experimental crack width (mm)</th>
<th>90 Days Experimental crack width (mm)</th>
<th>28 Days Theoretical crack width (mm)</th>
<th>90 Days Theoretical Crack width (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( S_0 )</td>
<td>1.20</td>
<td>1.10</td>
<td>0.055</td>
<td>0.060</td>
</tr>
<tr>
<td>( S_5 )</td>
<td>1.40</td>
<td>1.50</td>
<td>0.060</td>
<td>0.065</td>
</tr>
<tr>
<td>( S_{10} )</td>
<td>1.40</td>
<td>1.60</td>
<td>0.065</td>
<td>0.075</td>
</tr>
<tr>
<td>( S_{15} )</td>
<td>1.50</td>
<td>1.60</td>
<td>0.070</td>
<td>0.080</td>
</tr>
<tr>
<td>( S_{20} )</td>
<td>1.60</td>
<td>1.80</td>
<td>0.075</td>
<td>0.085</td>
</tr>
<tr>
<td>( S_{25} )</td>
<td>1.80</td>
<td>2.20</td>
<td>0.070</td>
<td>0.075</td>
</tr>
<tr>
<td>( S_{30} )</td>
<td>2.20</td>
<td>2.60</td>
<td>0.060</td>
<td>0.070</td>
</tr>
<tr>
<td>( S_{35} )</td>
<td>2.60</td>
<td>3.10</td>
<td>0.050</td>
<td>0.060</td>
</tr>
</tbody>
</table>

CONCLUSION

This work has looked at the use of locally available metakaolin as cementitious material. The results achieved from all the experiment carried out showed that what was obtained is comparable to what literature recommended from previous studies. The entire test carried out on concrete mixed with metakaolin gave very encouraging results, cement can be replaced in concrete up to 20%, and this is a good development if the present cost of cement in Nigeria can be put into consideration, and with the abundance of this naturally occurring kaolin clay material in some states of Nigeria, effort should be made by the construction industry to tap into this technology.

REFERENCES


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