



## Pozzolanic Strength Activity Index of Metakaolin Processed from Kankara Kaolin According to BS EN 450-1

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### ABSTRACT

Locally sourced natural pozzolanas in Nigeria need to be assessed in terms of their suitability for use in cement production because the use of pozzolanas has recently gained importance. In this study Metakaolin as a pozzolan processed from Kankara kaolin was studied for its suitability in cement production using the pozzolanic strength activity index (SAI) approach. The kaolin clay was beneficiated, ball-milled, and calcined at 750 ° C for 2 hours to produce the metakaolin. The SAI test was performed according to BS EN 450-1 with 25 % of cement replaced by the pozzolan to produce the pozzolan mortar and 100 % cement for the control mortar. The metakaolin produced with a sum of  $Al_2O_3$ ,  $SiO_2$ , and  $Fe_2O_3$  of 98 % and the sum of CaO and MgO of 0.75 % respectively satisfies ASTM C618-05 limits of  $\geq 70$  % and  $\leq 5$  %. It was observed that both the pozzolan and control mortar samples recorded increased compressive and flexural strength with curing age with samples containing the pozzolan having higher strength values, also, the flexural strength of all the mortar samples was found to be in the range of 32 % to 47 % of the compressive strength. The SAI test of the metakaolin (pozzolan) recorded an index of  $> 100$  % in compression and  $\geq 89$  % in flexure passing BS EN 450-1 limit of  $\geq 75$  % at 28 days. These findings revealed that pozzolan processed from Kankara kaolin having satisfied the code limits is a good pozzolan and its use as a pozzolan in mortar and concrete production should be encouraged.

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### INTRODUCTION

The production of binary (cement and a pozzolan) and ternary (cement with 2 pozzolans) cements is a global practice and many countries (e.g. Nigeria) have abundant deposits of natural pozzolan capable of being used cement (Pourkhorshidi et al, 2010). ASTM C125 defines a pozzolan as " a siliceous and aluminous material which, in itself, possesses little or no cementitious value but which will, in a finely divided form in the presence of moisture, react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties". It is known that for sustainable construction,

materials with improved mechanical, physical, better durability properties as well as environmental and economic benefits are needed, Martina and Aena (2015), Pozzolans offer such benefits and the use of natural and artificial pozzolans as additives have gained momentum in cement and concrete production, as a result, pozzolans need to be assessed in terms of their suitability and durability for use as cement replacement materials Baki et al (2020). As confirmed by many studies, the pozzolans influence the hydration process leading to a better structure of the hydrated product and hence better structural properties.



Pontes et al, (2013) stated that "Pozzolanicity is a very interesting issue regarding building materials, as a way to enhance mortar and concrete durability". One of the methods used in determining pozzolanicity of these pozzolans when used as a cement replacement material is an indirect test method called the Pozzolanic Strength Activity index (SAI) test, this test method correlates well with direct methods like Frattini test (Nina and Lisbet, 2019) and was reported to be one of the most accurate and reliable methods by Alejandra et al, (2013), the SAI of a pozzolan is a comparison of the compressive strength of mortar with cement replaced at a percentage by the pozzolan to a reference mortar produced with 100 % cement (i.e no pozzolan). A pozzolan would be considered active if the compressive strength of the Pozzolanic Mortar is greater than or equal to 75 % (BS EN 450-1) of the compressive strength of the reference mortar at 28 days, or 85 % at 90 days. Lennart et al (2019) stated that the pozzolanic activity is dependent on the reaction of  $\text{Ca(OH)}_2$  (portlandite) with a powdered reactive  $\text{SiO}_2$  to form more C-S-H bonds which in turn brings about an improvement in the properties of the cementing product.

Metakaolin processed from kaolinite clays at appropriate temperatures (Ambroise et al, 1985) has been reported to react with CH released in the hydration of ordinary Portland cement (OPC), such reaction increases the heat of hydration and produces a better cementitious material compared with when 100 % cement is used. (Frias et al.,

2000). Confirming this is a report by Ali et al, (2019) who reported increased strength properties for samples containing pozzolanas relative to the control after working on improving the durability of recycled concrete aggregate when used in mortar mixtures using binary and ternary blends of cementitious mixtures (Pozzolans)

Because of the mentioned findings, this paper aimed at accessing the Pozzolanic Strength activity of Metakaolin (Pozzolan) Processed from Kankara Kaolin in accordance with BS EN 450-1. The findings will provide information on the suitability of the clay for use as cement replacement material based on the code provisions.

## MATERIALS AND METHODS

### Materials

The following materials were used in the course of this research work:

*Metakaolin (Pozzolan):* This was sourced from Kankara Local Government Katsina State, Kaolin clay deposits. The raw clay has an off white colour and after processing, the metakaolin has a whitish pink colour.

*Fine aggregate:* The fine aggregate used was river sand obtained from a nearby river in Zaria, Kaduna State-Nigeria. The fine aggregate has a specific gravity of 2.7 and an average moisture content of 6.75%  
*Cement:* The cement used was Dangote 3X (CEM II-42.5N) cement which was gotten from a local dealer at the Samaru market, Zaria, Kaduna state, the cement was tested and was found to satisfy the requirements of the relevant codes.

**Table 1:** Properties of the Cement

Property	Observed Value (X)	Code Limits
Consistency (0.5 W/C)	5.5mm	$5 < X < 7$ mm
Initial and final setting times	1hr 26 min-2 hr 46 min	$45 \text{ min} < X < 10$ hrs
Soundness	2 mm	$X < 10$ mm

**Water:** Clean and potable water obtained from the Concrete laboratory of the department of civil engineering, Ahmadu Bello University, Zaria-Nigeria.

## METHODS

*Metakaolin Processing:* This was done at the Chemical Engineering Department laboratory. The kaolin clay was



beneficiated by soaking and filtering to remove impurities and later air and oven-dried. It was ball milled and sieved to finer powdered form (75  $\mu\text{m}$ ). The powdered kaolin clay was then calcined at 750 °C for 2 hours to obtain an amorphous reactive metakaolin.

*X-Ray Fluorescence (XRF):* The oxide composition test of the metakaolin was conducted at the Multi-User Laboratory, Chemistry Department A.B.U Zaria using the Oxford Instruments

*Mortar Production:* This was done at the concrete laboratory of Civil Engineering Department Ahmadu Bello University (A.B.U) Zaria according to BS EN 196-1; the control mortar samples (C1) were produced with 100 % OPC and the Pozzolanic Mortar samples (P1) were

produced with 25% of the cement replaced by the Metakaolin (Table 2) same as Miguel et al (2015) and Martina and Alena (2015). The required quantity of metakaolin, fine aggregate, and cement was measured and dry mixed for 4 minutes, then required quantity of water was gradually added and mixed, after each water addition, a flow test was performed until the water added (W) resulted in a flow property  $\pm 10 \text{ mm}$  of the control mortar flow. The mortar was then cast in prismatic  $40 \times 40 \times 160 \text{ mm}^3$  moulds similar to Miguel et al (2015), then compacted and levelled. It was demoulded from the mould after setting for 24 hours and cured in water at approximately 20 °C for 3, 7, 14, and 28 days when samples were tested for flexural and compressive strength.

**Table 2:** Mortar Mix Proportion

Samples ID	Fine aggregate (g)	Cement (g)	Metakaolin (g)	Water (g)
C1	1350	450	0	225
P1	1350	337.5	112.5	225+W

*Compressive and Flexural Strength Tests:* The compressive and flexural strength tests were respectfully performed according to BS EN 1015-11 (1999) at the concrete laboratory Civil Engineering Department. A.B.U Zaria.

*Strength Activity Index (SAI):* The pozzolanic activity index of the Metakaolin (Pozzolan) was done in accordance with EN 450-1. The same approach was followed by Martina and Alena (2015) and Baki (2020). The Pozzolanic Strength activity index (SAI) is expressed as:

$$SAI = \frac{f_{p1}}{f_{c1}} \times 100 \quad (1)$$

Where  $f_{p1}$  the unconfined strength of the pozzolan is mortar ( $\text{N}/\text{mm}^2$ ), and  $f_{c1}$  is the unconfined strength of the control mortar ( $\text{N}/\text{mm}^2$ )

#### RESULTS AND DISCUSSION:

All mechanical test results (Compressive and flexural strength tests) reported are for averages of three test results

**Table 3:** Oxide Composition of the Metakaolin

Oxide	Concentration (Weight %)	Oxide	Concentration (Weight %)	Oxide	Concentration (Weight %)
Na <sub>2</sub> O	0.082	SO <sub>3</sub>	0.159	Cr <sub>2</sub> O <sub>3</sub>	0.002
MgO	0.487	Cl	0.012	Mn <sub>2</sub> O <sub>3</sub>	0.009
Al <sub>2</sub> O <sub>3</sub>	42.446	K <sub>2</sub> O	0.437	Fe <sub>2</sub> O <sub>3</sub>	0.858
SiO <sub>2</sub>	54.719	CaO	0.263	ZnO	0.004
P <sub>2</sub> O <sub>5</sub>	0.255	TiO <sub>2</sub>	0.263	SrO	0.003

Source: Laboratory work (2018)

Table 3 presents the oxide composition of the pozzolan (Metakaolin), the sum of  $Al_2O_3$ ,  $SiO_2$ , and  $Fe_2O_3$  (98.023%) satisfies ASTM C618-05 requirement of  $\geq 70\%$  and the sum of CaO and MgO (0.75%) also satisfies the code limit of  $\leq 5\%$ . This showed that based on oxide composition, Metakaolin processed from Kankara kaolin can be considered a pozzolan to be used together with OPC as supplementary cementing material.

#### Compressive and Flexural strength Test

Figure 1 presents the compressive results of both the control mortar and the pozzolanic mortar (with 25% cement replaced by metakaolin) against curing age. The results showed a continued strength gain with curing age as a result of continued cement hydration for all samples. It was observed that samples containing the pozzolan have higher compressive strength values compared with the control samples, this might be because of the reaction between the pozzolan that is rich in Si and Al precursors and the CH released during cement hydration (Frías et al., 2000) resulting to better cementitious material (More C-S-H bonds), Miguel et al (2015) also opined that the higher strength is a result of the effect of the finely grounded pozzolan on the microstructure of the hardened paste resulting to improved macro-structure of

the mortar. The 3-day strength for the control sample was  $3.3\text{ N/mm}^2$  while that of the pozzolanic sample was  $3.5\text{ N/mm}^2$ , the strengths increase respectively by 51.5%, and 60% to  $5\text{ N/mm}^2$  and  $5.6\text{ N/mm}^2$  at 28 days for the control and Pozzolanic sample. The 14 and 28 days strength of the control sample showed no noticeable difference while that of the pozzolanic sample increased by  $0.2\text{ N/mm}^2$  which might be because of the presence of the pozzolan (Metakaolin) which makes the continuous formation of C-S-H bonds possible after 14 days similar to Donatelo et al, (2010) who also reported continuous strength gain with curing age only for samples containing metakaolin as a pozzolan. The test results of the flexural strength (figure 2) followed a similar trend of strength increase with the age of curing and with most of the pozzolan mortar having higher flexural strength than the control mortar because of an increased chemical reaction and packing density (Lennart et al, 2019), the pozzolan and control mortar respectively increase in flexural strength from  $1.56\text{ N/mm}^2$  and  $1.17\text{ N/mm}^2$  after 3 days water curing to  $1.95\text{ N/mm}^2$  after 28 days water curing, additionally. This showed that the presence of the metakaolin as a pozzolan does not change the trend strength gain of ordinary Portland cement mortar.

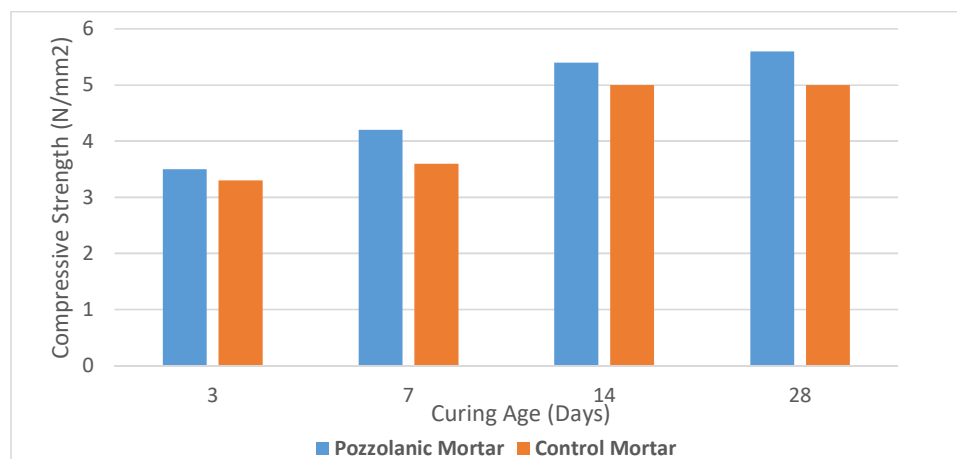
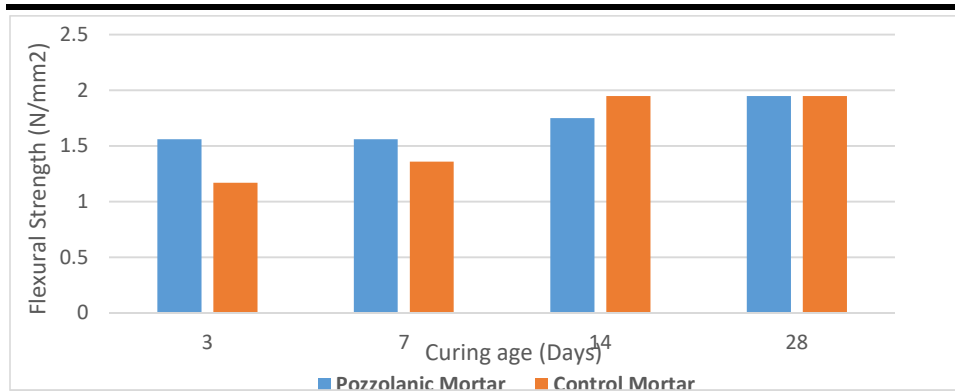


Figure 1: Compressive strength against curing age for the control and Pozzolan Mortar

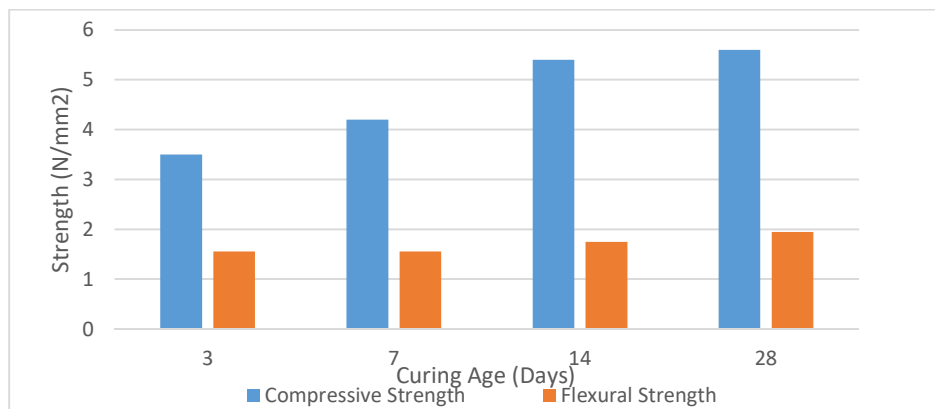


**Figure 2:** Flexural strength against curing age for the control and Pozzolan Mortar

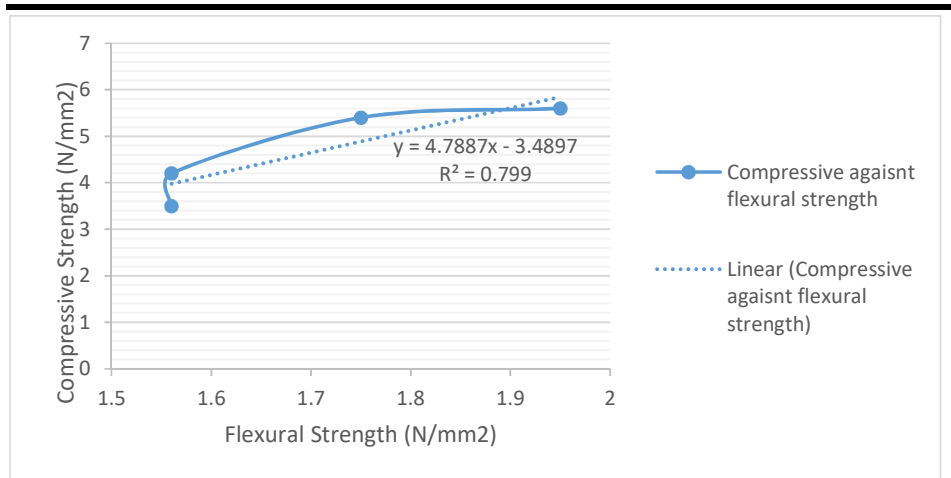
**Relationships between compressive and flexural strength**

Figures 3 and 5 compare the compressive with flexural strength at the curing ages respectively for pozzolan and control mortar while Figures 4 and 6 showed the relationship in the form of a linear regression between the compressive and flexural strength. The pozzolan mortar (Figure 3) with higher compressive and flexural strength compared with control mortar has a ratio of flexural to the compressive strength of 0.45, 0.47, 0.32, and 0.35 respectively after 3, 7, 14, and 28 days of water curing while 0.35, 0.38, 0.39 and 0.39 were respectively observed for the control mortar (Figure 5). Generally, it can be said that the flexural strength of all the mortar samples is in the range of 32 % to 47 % of the compressive strength. Figures 4 and 6 respectively present the linear regression

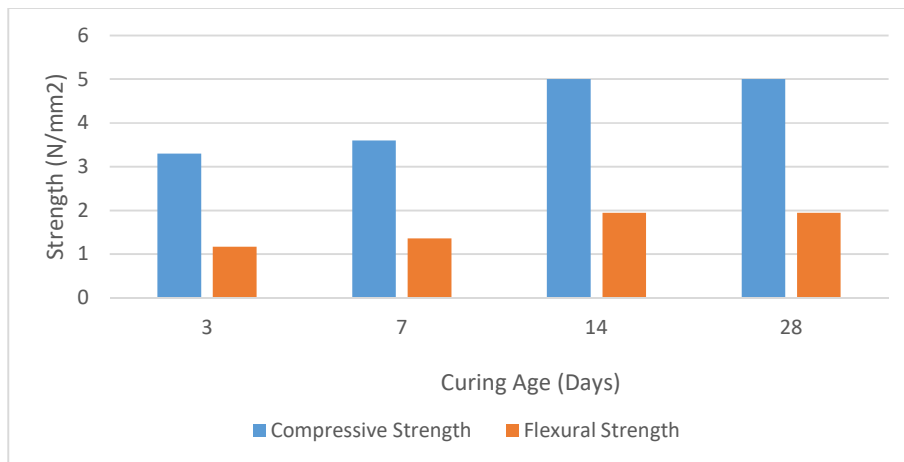
model and the coefficient of linear regression ( $R^2$ ) for the pozzolan mortar and control mortar. An  $R^2$  value of 0.8 was recorded for the pozzolan mortar, and 0.99 for the control mortar, these values portray that with the linear models, 80 % and 99 % of the variation of the compressive strength can be explained by the flexural strength and vice-versa, the observed  $R^2$  values showed a good relationship between the strength values because according to Andrew (2017), smaller  $R^2$  values produce useful predictions. The author further mentioned that with the  $R^2$  approach, the unexplained proportion of the variation can be attributed to factors not considered in the model, e.g. the C-S-H bonds in the pozzolan mortar matrix resulting from the pozzolan and its reaction and the proportion of unreacted pozzolan serving as filler within the matrix.



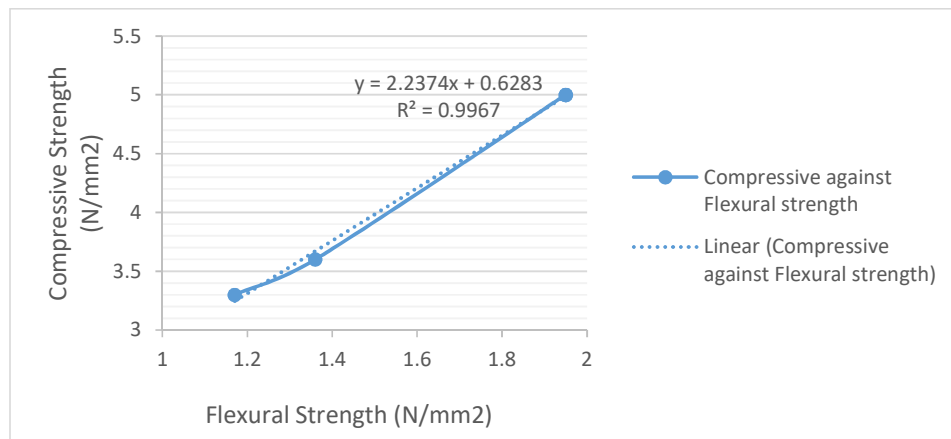
**Figure 3:** Relationships between compressive and flexural strength (Pozzolan Mortar)



**Figure 4:** Linear regression plot for pozzolan mortar (compressive against flexural strength)



**Figure 5:** Relationships between compressive and flexural strength (Control Mortar)

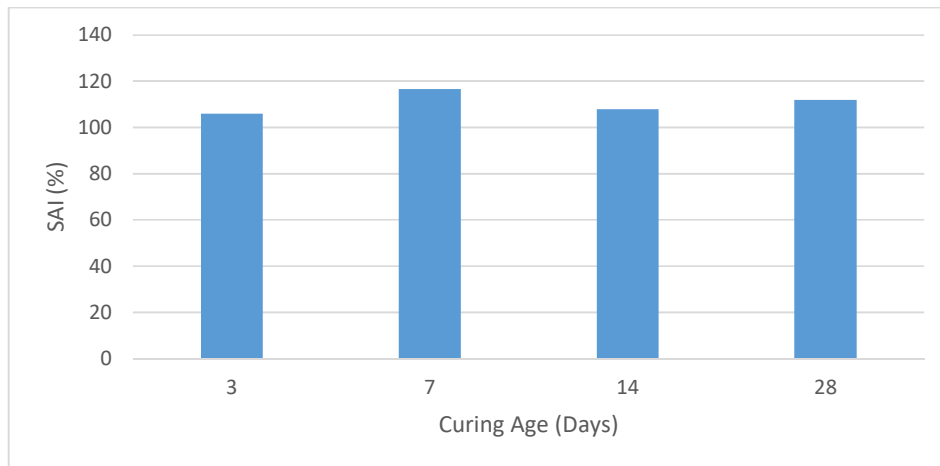


**Figure 6:** Linear regression plot for control mortar (compressive against flexural strength)

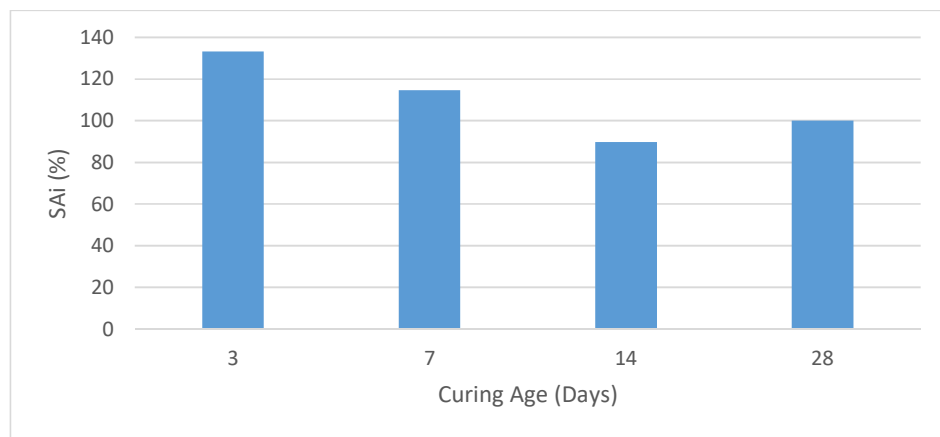
**Pozzolanic Strength Activity Index (SAI)**

The pozzolanic strength activity index of metakaolin (pozzolan) processed from Kankara kaolin considering compressive and flexural strength is presented respectively in figures 7 and 8. BS EN 450-1 requires that a pozzolan can be considered active and should be used with cement as supplementary cementitious material if the activity index is  $\geq 75\%$  at 28 days. From the figures, it can be seen that the SAI values recorded at 28 days for the compressive and flexural strength are 112% and 100% respectively and SAI values recorded at 3, 7, and 14 days are all greater than 100% except at 14 days in flexure which has 90%. These

values are similar to Miguel et al (2015) values of  $> 100\%$  for a finely grounded pozzolan (Silica Fume). At 7 days, the SAI Considering compressive strength (Figure 7) was the highest (117%), this concurs with Donatello et al (2010) whose highest metakaolin pozzolanic test results were recorded at 7 days, also Pontes et al (2013) and Alejandra et al, (2013) both recorded the highest SAI value with metakaolin as a pozzolan. All of the results satisfy the BS EN 450-1 limit, and it can be said that Kankara kaolin beneficiated, calcined at  $750^\circ\text{C}$  for 2 hours, and appropriately ground to a required powdered form ( $75\ \mu\text{m}$ ) produce an active pozzolan (Metakaolin) in accordance with the code.



**Figure 7:** Pozzolanic Strength activity index considering compressive strength



**Figure 8:** Pozzolanic Strength activity Index Considering Flexural Strength



## CONCLUSION

After analysis of the results, it was found that, Kankara kaolin calcined at 750 ° C for 2 hours produces a pozzolan (metakaolin) with a composition of oxides that satisfies ASTM C618-05 requirements for natural pozzolans since the sum of  $Al_2O_3$ ,  $SiO_2$ , and  $Fe_2O_3$  is  $\geq 70\%$  and the sum of CaO and MgO is  $\leq 5\%$ .

Both the pozzolan and control mortar samples recorded increased strength with age with samples containing the pozzolan having higher strength. In addition, the flexural strengths of the pozzolan and control mortar samples are in the range of 32 % to 47 % of the compressive strength values. The pozzolan with a strength activity index of  $> 100\%$  in compression and  $\geq 89\%$  in flexure passes BS EN 450-1 limit of  $\geq 75\%$ . The pozzolan processed from Kankara kaolin having satisfied ASTM C618-05 and BS EN 450-1 limits is a good pozzolan and its use in mortar and concrete production should be encouraged.

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