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Research Article

## Effects of metakaolin and treated rice husk ash on the compressive strength of concrete

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### Abstract

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Cement industry being source of emission of carbon dioxide (CO<sub>2</sub>) causing global warming; it is necessary to source for alternative binder in concrete. The problem of longer age strength attainment by pozzolanic blended concrete calls for introduction of treated pozzolans. This paper investigates the influence of co-addition Metakaolin (MK) and Treated Rice Husk Ash (TRHA) in the production of concrete. The kaolin was calcined at a temperature of 650°C for 2 hours and thereafter, characterized. Furthermore, the rice husk (RH) was roasted to temperature of 700°C and thereafter, treated with H<sub>2</sub>SO<sub>4</sub>. MK was replaced in varying percentages of 0%, 5%, and 10%. Thereafter, TRHA was added by weight of the cement in varying percentages of 1%, 2%, and 3% to the optimum MK replacement (5%). From the results, it was found that concrete with 5% MK and 2% TRHA had enhanced compressive strength as against the control. Hence, it can be inferred that 5% MK and 2% TRHA concrete could be used in the construction industry. The increase in the strength properties of the concrete could be as a result of additional binding tendency exhibited by MK and TRHA.

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## 1. Introduction

In pursuit of urbanizing the environment, there is need to provide basic amenities amongst are: buildings, roads, adequate and portable water supply system for the citizenry. The rate of development of infrastructures is on the very high side and one of the mostly used construction materials that is very relevant in the highlighted amenities is concrete. Concrete comprises of cement, sand, stone (aggregate) and water. Its fast advancement in this century has made it one of the most widely used construction materials all over the globe [1]. Cement being one of the primary ingredients of concrete is the point of attention most especially the marginal increase in its price and the environmental threat it poses to the atmosphere via emission of CO<sub>2</sub> and other greenhouse gases (GHG). Currently, the interest in Portland cement as a binder in concrete is growing in developing countries with a global production rate of approximately 1.2 billion tons/year. This increase in demand for cement could be complemented for by adding either fully or partly supplementary cementitious materials (SCM) that when in contact to lime released from hydration of cement and in the presence of water exhibit binding property, thus adding more strength to the composites [2, 3].

The call for addition SCM of in concrete technology had resulted into searching for industrial and agricultural wastes that have tendency of exhibiting the binding property.

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In other words, most industrial wastes are source of agricultural as most industrial raw materials are agro-based. The available waste in countries differs which in turn is subject to types of agricultural produce available in their various domain. Agricultural wastes are by-product obtained at the end of processing and consumption of farm produce and are so enormous amongst are: wheat, corn cob, guinea corn, need seed, groundnut shell and rice husk [4]. The rate of cultivation and consumption of rice is very high and consequently, the availability of its husk. The high silica content of the soil had in turn made the silica content of plants to be high and this has positive influence on the pozzolanic tendency of most agricultural products. Hence, enhance its SCM in concrete composite [5].

Pozzolans are either natural or artificial. Natural pozzolans (NP) being sourced from volcanic eruption could either be volcanic ash or volcanic pumice. The artificial ones are obtained as a result of heat treatment of industrial wastes or clay materials and some examples of this class includes: MK, fly ash (FA), silica fume (SF), Rice Husk Ash (RHA), and Ground granulated blast furnace Slag (GGBFS) [6]. The technical benefits of pozzolans in concreting could be attributed to the presence of abundant quantity of alumina or silica which have tendency of enhancing the hydration process of cement and consequently, better strength enhancement with remarkable microstructural development [7]. Although, MK is quite different from other artificial pozzolans as it is sourced from the heat treatment process involving removal of water and phase changing of hydroxyl present in china clay called kaolin [8 ] [9].

Clay minerals are so enormous but kaolin clay distinct itself amongst other due to high level of purity and consequently, the presence of quartz, rutile, pyrite, siderite, feldspar [10]. In addition, MK could be gotten from calcination of paper sludge [11]. It is more of pure clay with some other minerals but not limited to: halloysite, dickite, anauxite and nacrite [8]. The uniqueness of MK as compared to other pozzolans could be affiliated to the following benefits: Dilution effect, filling ability, lubrication of very coarse cement particles and consequently, enhanced acceleration of the hydration of cement [ 9]. Furthermore, MK had performed very satisfactorily compared to other pozzolans in terms of strength development at early stage of curing in particular silica fume. This has been attributed to its enhanced rate of hydration of cement [12].

MK is obtained by the thermal activation (dehydroxylation) of kaolin clay in a temperature of between 600°C to 850°C for at most 12 hours [13]. The calcination process has the tendency of breaking the crystalline nature of the MK thereby changing it to amorphous phase and consequently, improving on the pozzolanic tendency [14]. The process has the potential of unbounding water molecules in the pores of the clay as well as deformation of the crystalline nature. MK is quite distinct from other forms of pozzolans in that it could be purified to enhance its colour and sizes of the particles thus improving tremendously the reactivity [15]. MK when used as a partial replacement substance for cement in concrete, reacts with calcium hydroxide  $\text{Ca}(\text{OH})_2$ , one of the by-products of hydration of cement and results in additional C-H-S gel which result in increased strength. Naresh [16] concluded 10% Mk replacement of cement enhanced the mechanical properties of concrete when incorporated with silica fume in the production of concrete. Malagavelli [17] opined 10% MK infused in concrete enhanced the splitting tensile, flexural and compressive strength of concrete as compared to the control specimen.

Furthermore, the work of Ramezaniapour [18] showed that MK performed satisfactorily well in terms of compressive strength enhancement and durability. Asides from MK having significant effects in terms of strength enhancement, it surpasses silica fume on the aspect of pull out performance of steel fibers incorporated in cement paste [19]. A parameter in determining the reactivity of pozzolan in concrete is to compute the Strength Activity Index (SAI). According to BS 3892 [20] the value of SAI greater than 0.80 at 28 days of curing

show high pozzolanic tendency. also, ASTM C618 [21] stated that a SAI greater than 0.75 is an indication of reactive NP. The influence of MK on the properties of concrete is not limited to the hardened state as it plays important role in the fresh properties of concrete. In particular, the consistency and setting of the concrete; the high surface area of MK makes the hydration of MK blended cement to require more water and hence, as the percentage of MK increases, the consistency reduces [22], [23]. The incorporation of moderate MK in concrete has good influence on the workability compared to other pozzolans where the addition of superplasticizer is required in order to achieve relatively good consistency [24]. Both the initial and final setting times of metakaolin blended concrete were observed to increase with increase in MK replacement [25].

Rice Husk (RH) has been found to have varying qualities in terms of purity due to the presence of some carbon content and other impurities like oxides of sodium and potassium which in turn could impair the reactivity went burnt to form RHA [26]. The recommended calcination temperature to give a reactive substance should not be more than 700 °C and any temperate above that could result into the transformation of amorphous silica into crystalline thereby leading to an unreactive material [27]. According to Prasad [28] the calcination temperature to give RHA with satisfactory pozzolanic properties should be in the range of 500 to 600°C. The pretreatment of RHA using some acids amongst are: hydrochloric and nitric have been found to significantly enhanced its purification [29]. This research is aimed at investigating the effect of addition of MK and RHA treated with sulfuric acid and base on the properties of concrete. With specific objectives of determining the chemical properties of MK as well as investigating its incorporation and purified RHA on the compressive of concrete

## 2. Materials and Method

In this research, the following materials were used: Ordinary Portland cement (OPC), fine aggregates (River sand), coarse aggregates (granite), MK, TRHA, Super-plasticizer and water. The kaolin clay was obtained from a mining site at Osin-Aremu, Ilorin South Local Government Area with coordinates 8.4519°N and 4.5378°E and the Rice Husk from a rice milling shop at Oja-gboro area in Ilorin, Kwara State. Ordinary Portland cement (OPC), Dangote cement brands of 32.5R which conforms to NIS 444-1[30] was purchased from a vendor in Ilorin, granite conforming to BS 882 [31] with nominal size of 20mm was used as coarse aggregate and river sand also conforming to BS 882 specification was used as fine aggregate in the concrete with the duo sourced from Maleta, Kwara State, Nigeria.

Firstly, the kaolin clay was calcined at a temperature of 650 °C for one (1) hour and the RH was burnt at a temperature of 700 °C for a duration of three (3) hours using a furnace in the Department of Mechanical Engineering, Kwara State Polytechnic, Ilorin Kwara State. Thereafter, the sample was taken to the laboratory of the National Agency for Science and Engineering (NASeni), Akure, Ondo State, Nigeria for EDXRF analysis using SKYRAY instrument model EDX3600B. Also, the physical properties of the aggregates were determined and batching of concrete was done by weight with proportioning details as contained in Table 1. A concrete with target strength of 40N/mm<sup>2</sup> (M40) was designed for in accordance with the specification of Council for the Regulation of Engineering in Nigeria (COREN) Concrete Mix Design Manual. Mixing was done manually; the fine aggregate was spread on a non-absorbent surface and thereafter, mixed thoroughly with cement for homogeneity and consequently mixed with coarse aggregates. A water-binder ratio of 0.37 was used in the batching of concrete with the addition of superplasticizer.

Table 1. Quantity of concrete constituent per cubic meter

Concrete	Cement (kg)	MK (kg)	Treated Rice Husk Ash	Water (kg)	Super plasticizer (ml)	Fine Aggregate (kg)	Coarse Aggregate (kg)
Control	27.98	0	0	10.47	150	23.97	44.55
MK 5%	26.58	1.40	0	10.47	150	23.97	44.55
MK 10%	25.18	2.80	0	10.47	150	23.97	44.55
MK 5%+ TRHS 1%	26.3	1.40	0.28	10.47	150	23.97	44.55
MK 5%+ TRHA 2%	26.02	1.40	0.56	10.47	150	23.97	44.55
MK 5%+ TRHA 3%	25.74	1.40	0.84	10.47	150	23.97	44.55

Thereafter, required quantity of water was then added; mixed again thoroughly to achieve the final uniformity as shown in Figure 3. The control experiment was first batched with 0% replacement and thereafter, the cement was replaced with 5% and 10% Metakaolin respectively. To obtain the TRHA, 10 grams of Rice Husk Ash (RHA) samples were stirred in 80 ml sodium hydroxide solutions. RHA was boiled in a covered 250 ml Erlenmeyer flask for 3 hours. The solution was filtered, and the residue was washed with 20 ml boiling water. The filtrate was allowed to cool to room temperature and added  $H_2SO_4$  until pH 2. Thereafter,  $NH_4OH$  was added at room temperature until pH 8.5 was obtained. The filtrate was then dried at 120 °C for 12 hours. The chemical composition of the TRHA was determined and thereafter, added in varying percentages of 1%, 2%, and 3% by weight of the binder to the 5% Metakaolin (being optimum) replacement. As soon as uniformity was achieved, slump test was carried out to determine the properties of the concrete in fresh state. A slump cone of high 300mm with diameters 100mm and 200mm at the top and bottom was placed on a non-absorbent plane surface. The fresh concrete was poured to fill up the cone in three layers with a tamping of 25 strokes for each layer. After filling, excess concrete at the top was carefully scraped using a trowel. Thereafter, the cone was lifted and concrete subsides, the corresponding height of fall was measured to read the slump values. Concrete cubes specimens of size 150mm x 150mm x 150mm were cast and cured in water for 7, 21, 28, and 56days respectively. The compressive strength of the cubes was determined at different ages of curing and the cubes were placed in the machine as shown in Figure 1 and allowed to compressed between the platens of a compression machine with gradual application of load. At a point in time, cracks were observed on the cubes and load readings in (kN) read on the gauge. This was performed three times and the average load was calculated. The compressive strength was computed by finding the ratio of the load to the corresponding area of the cubes. Hence, the Strength Activity Index (SAI) of the specimen was obtained using equation 1 in order to compare the strength of the control specimens relative to the test experiment.

$$SAI = \frac{A}{B} * 100 = \left( \frac{45.86}{30} \right) * 100 = 1.52 \tag{1}$$

Where:

A= Unconfined Compressive Strength Test of pozzolan specimen

B = Unconfined Compressive Strength Control concrete (0% pozzolan) Table 1 Drill pipe dimensions and properties [6]



Fig. 1 Compressive strength test of concrete cube

### 3. Results and Discussion

#### 3.1 Chemical Composition of Metakaolin, RHA and TRHA

The result of chemical analysis is as shown in Table 2, the predominant oxide was found to be silicon dioxide and the sum of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> of the metakaolin clay is 71.04% less than 95.22% as obtained in other work [32] but greater than 70%. Hence, the calcined clay can be classified as class N pozzolan [21], [33]. Furthermore, the relative abundance of Sulphate is 0.96% less than 4%, also affirmed the metakaolin belongs to the class N pozzolan [21]. Tables 3 and 4 show the chemical composition of oxides present in both RHA and TRHA. From the tables, the silica content was found to increase with corresponding in both sodium and potassium oxides. These results show an enhancement in purification of the RHA and the result is in agreement when compared with the work of some researchers [34]

Table 2. Results of Chemical Analysis of Metakaolin

Chemical Compounds	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>
Percentage Composition (%)	50.10	19.20	1.74	4.415	4.61	0.96

Table 3. Results of Chemical Analysis of RHA

Chemical Compounds	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O
Percentage Composition	83.62	1.43	0.78	1.24	0.09	0.04	0.08

Table 4. Results of Chemical Analysis of TRHA

Chemical Compounds	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O
Percentage Composition	89.70	1.01	0.29	0.83	0.04	0.02	0.03

### 3.2 Physical Properties of Aggregates

Table 5 indicates the result of the specific gravity and water absorption test carried out on fine aggregate and coarse aggregate used in this research. The range of specific gravity of aggregates specified by ACI Education Bulletin [35] ranges from 2.30 to 2.90. Thus, the results of specific gravity of fine and coarse aggregates are within the acceptable limits for aggregates.

Table 5. Properties of Coarse Aggregate and Fine Aggregate

Property	Fine Aggregate	Coarse aggregate
Specific Gravity	2.56	2.64
Water Absorption (%)	1.10	0.25

### 3.3 Workability

The slump height of the test experiment was found to be greater than the control. This implies that the addition of MK and TRHA increased the consistency of the concrete as compared to the control (0% replacement). It is indicative of higher workability. The 5% MK + 2% TRHA had the greatest height of subsidence and consequently, the most workable proportion see Figure 2.

Table 6. Statistical Analysis of Metakaolin Blended Concrete with TRHA

Metakaolin/ TRHA	N	Mean	Standard Deviation	Standard Error Mean
MK 0%				
MK 5% + 1% TRHA	12	40.2258	4.67782	1.35037
MK 5% + 2% TRHA	12	42.4443	3.83125	1.10599
MK 5% + 3% TRHA	12	31.7112	3.29439	0.95101

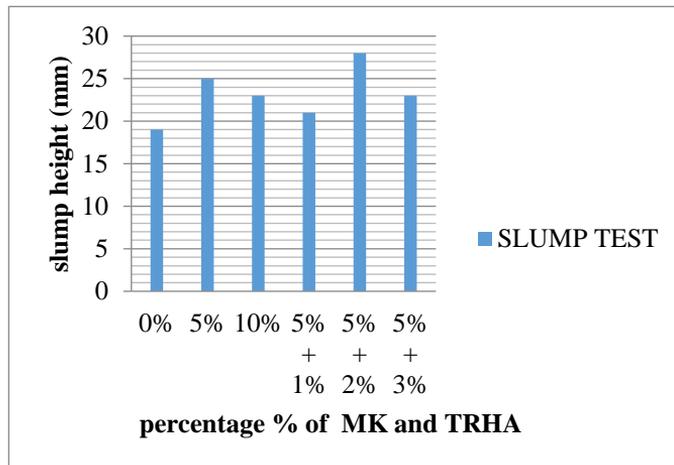


Fig. 2 Graph showing slump height against percentage replacement of MK and TRHA

Table 7. T-Test for Average Compressive Strength of Metakaolin Blended Concrete with TRHA

Metakaolin	T	Degree of Freedom	P-Value	Mean Difference	Correlation
MK 5% + 1% TRHA	20.16	11	0.000	15.05411	0.871
MK 5% + 2% TRHA	24.84	11	0.000	17.19010	0.869
MK 5% + 3% TRHA	5.363	11	0.000	7.13383	0.681

### 3.4 Compressive Strength Test

Figure 3 revealed the incorporation of metakaolin at both 5 and 10 % was found to increase compressive strength of concrete as compared to the control with 5% MK being optimum at all curing ages. Figure 4 shows early compressive strength enhancement when TRHA was added to the concrete. The compressive strength of 5% MK and 2% TRHA at 28 days of curing was 45.867 N/mm<sup>2</sup> as compared to the strength of 5% MK at 56 days of curing (43.141N/mm<sup>2</sup>), which implies that strength of 5% MK+ 2% TRHA at 28 days of curing was higher than the strength of 5% MK at 56 days of curing. The result is in agreement as compared to the work of Kwan [26], [29] where acid treated RHA have been found to increase strength. The statistical analysis further explained the result as contained in Tables 6 and 7. Since the significance level of 0.05, a two-tailed test allots half of your alpha to testing the statistical significance in one direction and half of your alpha to testing statistical significance in the other direction. The mean is considered significantly different from  $\bar{x}$  if the test statistic is in the top 2.5% or bottom 2.5% of its probability distribution, resulting in a p-value less than 0.05. because the p-value is Zero ( $< \alpha$  level), you reject the null hypothesis and conclude that there's a statistically significant difference. In addition, the strength of the duo was found to be greater than the control specimen (30.100N/mm<sup>2</sup>). Furthermore, the result of the SAI was found to be 1.52 greater than the minimum value of 0.8 specified by [20].

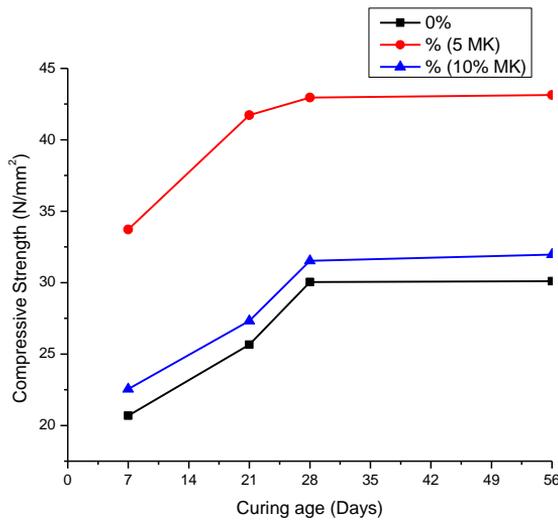


Fig. 3 Graph showing Compressive Strength of Concrete Incorporated with Metakaolin

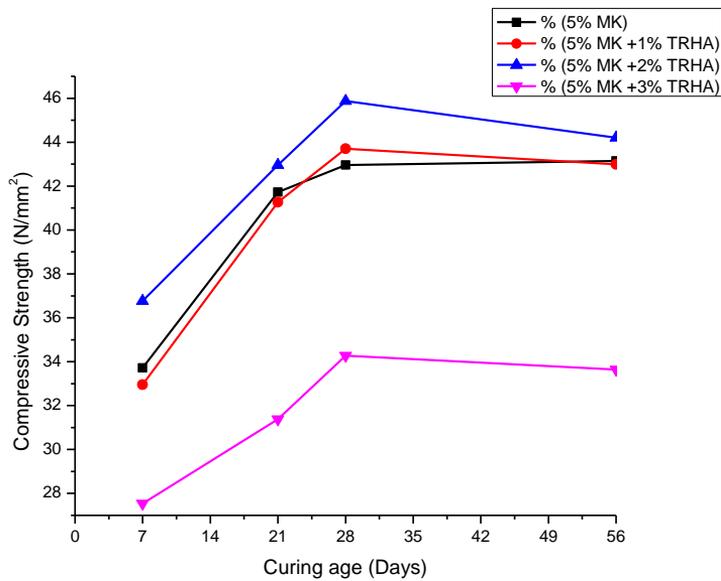


Fig. 4 Compressive Strength of Metakaolin concrete with Treated Rice Husk Ash

## 5. Conclusions

From the research, it could be inferred that:

- The sourced MK having the addition of silica, ferric and Aluminium oxides greater than 70% as specified by ASTM C 618. Hence, the material exhibits supplementary cementitious tendency and classified as Class N pozzolan.

- Also, it can be concluded that the compressive strength of concrete attained its maximum with optimum 5% MK replacement.
- Furthermore, 5% Mk and 2% TRHA gave better compressive strength at shorter age of curing than 5% Mk only with prolonged period of curing with a percentage strength increment of 5.94%. However, the results of compressive strength of both test experiments (5% Mk +2% TRHA) gave strength increment of 34.38% and 30.22% respectively.

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