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EXPERIMENTAL STUDY ON CEMENT MORTAR WITH ILMENITE TAILINGS AND CEMENT KILN DUST

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ABSTRACT

Beneficial use of industrial by product has the potential to provide economic benefit such as preservation of natural resources, and avoid negative environmental impact associated with the acquisition and processing of raw materials. Cement and ilmenite manufacturing is critically important industry in Nigeria and all over the world. As with most large manufacturing industries, by-products material are generated. These industrial by-product and waste materials must be managed responsibly to insure a clean and safe environment. The effect of the utilization of cement kiln dust (CKD) and ilmenite tailings on the compressive strength of mortar were investigated. Series of mortars were prepared with ilmenite tailings at different level of 20%, 40%, 60%, 80%, and 100% to river sand. Conversely, ordinary Portland cement was replaced with CKD at 10%, 15% and 20%. A sample specimens of 50 mm x 50mm x 50 mm mortar cubes were prepared with 0.45 w/c ratio and tested at 1, 3, 7, 14, 21 and 28 days of curing. Laboratory investigations were conducted to evaluate the physical properties in terms of sieve analysis, specific gravity, water absorption, moisture content and setting time of the raw materials. Compressive strength of various mortar cubes were examined and evaluated. The results show that the setting time (initial and final) decrease with increased in percentage of CKD. The compressive strength increases slightly with increasing CKD. The result also showed that there is increased in compressive strength of mortar with the replacement of river sand with ilmenite tailings up to 100% at all the curing period. Considering the test results, CKD can be used up to 20% in replacing ordinary Portland cement and 100% ilmenite tailings in the production of mortar with excellent compressive strength that are above the 17.2 MPa for Type M mortar of ASTM C270 -14a (2014) Standard.

Keywords: Ilmenite tailings, Cement Kiln Dust, Mortar, Setting Time, Water Absorption, Compressive Strength.

INTRODUCTION

Due to continuous increase in industrial globalization and generation of waste, solid waste management has become one of the major global environmental issue. The entrenchment of sustainable environment has become very important in order to preserve valued environmental conditions as well as available resources. As part of measures toward fostering sustainability; the reduction in emissions of greenhouse gasses, the depletion of fossil fuel and waste generation and disposal has become critical issues for consideration. The construction industry through its activities has strong impact on the environment. Although these impacts are both positive and negative, the latter gives an underlying motive for the concern to seek mitigations for environmental problems.
Cement industry is one of the major industries that exert the environment due to its high natural resources and energy requirement and subsequently the emission of significant amounts of carbon dioxide. Nowadays there is global need to use waste material to substitute cement for limiting it environmental impact and converting these waste into useful product for sustainability.
The worldwide utilization of natural sand as fine aggregate in mortar production is very high and several developing countries are having problems with the supply of natural sand so as to meet the increasing demands of civil engineering construction industry. In many countries there is shortage of natural fine aggregate which is appropriate for construction as natural sand which is the most commonly used material. Therefore the shortage of the resources of natural sand is the cause for

sliding the barrier for by-products and tailings material being used as the resource. Re-use of by-product as a partial replacement of fine sand in construction reduce both the exploitation of natural resources and sustainability issues faced by waste. One major industries which among the largest producer of industrial by product is mining industries.

Ilmenite is the primary ore of titanium, a metal needed for making a variety of high-performance alloys. Most of the ilmenite mined worldwide is used to manufacture titanium dioxide, $Ti0_2$, an important pigment, whiting, and polishing abrasive. Worldwide production of ilmenite accounted for about 2 billion metric tons of ilmenite tailings (Bedinger, 2013).

Ilmenite is a titanium iron oxide mineral with the idealized formula $FeTi0_3$ and gained attention due to its semi conductive properties, which might extend its application towards electrical/electronic devices such as solar cells, high temperature integrated circuits, varistors, p-n junction diodes and Schottky barriers (Brown, Navrotsky, Nord, & Banerjee, 1993). Ilmenite tailings are-by-product generated by the titanium slag in the iron industries. Titanium slag, containing 80% of titanium dioxide, is commonly use for the manufacture of white pigment for paint, plastic, paper and fabrics. The industrial process, which consists of transforming an Ilemite ($FeTio_3$) ore, required two stages i.e. purification and reduction; each involve the formation of by product. The purification consist in enriching the ore by eliminating part of the impurities, mostly compose of plagioclase feldspar and sheet silicates (micas, illites and chlorides). This operation require a crushing process, followed by magnetic and gravity separation of the one leads to the formation of by product dust called ilmenite fines or tailings. In many countries huge tailings are produced as by product of metal ore processing and are dump every year in landfills, rivers among other places, and thereby giving rise to huge environmental issue. To fashion mining activities in more eco-friendly manner, it is important to conduct mining operation in a fashion that is more environmentally amiable. In this context, a correct environmental solution to the disposal of a wide range of tailing could be their incorporation into mortar production

During the manufacture of cement a large quantities or amounts of dust were produced as Cement Kiln Dust (CKD). Generation of CKD is estimated at approximately 30 million tonnes worldwide per year (Dyer, Halliday, & Dhir, 1999). In advance production techniques, it is possible to recycle most of the CKD with clinker making process. Most of the standard specification limited the Alkali quantity in Portland cement less 0.6% in order to prevent the Alkali aggregate reaction (Maslehuddin et al, 2009).

Cement Kiln Dust is an industrial by product formed during manufacturing of Cement and is a waste materials that is traditionally destined for landfills. It is a fine powdery material similar in appearance to Portland cement and created during the production of Cement Clinker. The Dust is a particular mixture of partially calcined and non-reacted row feed, Clinker Dust and ash enriched with Alkali sulphates and other volatiles. These materials are captured by exhaust gases and collected in a particular matter control devices such as cyclones, bag houses and electro-static precipitators. The bulk of this dust to landfilled with a significant environmental impact and financial loss to the local Cement industry in terms of the value of raw materials, processing and energy consumption during preprocessing. Cement Kiln Dust composed of Alkali compound and clinker particles, which can be high percent in lime content (Adaska and Taubert, 2008). Therefore this research is based on using Cement Kiln dust as a partial substitute of Cement in production of mortar.

The depletion of natural resources, increasing greenhouse emission and awareness of the need for sustainable development in terms of safe reuse of waste, the transformation of these waste into valuable materials is emerging as a strong trends in this context and taking into account the growing awareness of the need for protection of health and environment, correct environmental solution to the disposal of wide range of solid waste (Mining tailing) could be its utilization into cement mortar. Mehatlaf (2018) studied on compressive strength properties behavior of Cement mortars with addition of CKD and stated that low addition of CKD has no effect on the compressive strength, but addition of about 20-40% of CKD lead to high decrease in strength. Marku, Dumi, Liço, Dilo, and Çakaj (2012) shows that CKD strength contribution in the Portland cement blend is low. They stated that as the CKD content increase 0-45% the aggregate paste bond is weaker. So the CKD weakens the hardened Cement paste.

Hassan, Abdulkareem and Ahmed, (2013) studied different partial addition of Cement Kiln Dust (CKD) and observed that the addition of CKD lead to decreased in compressive strength and tensile strength for all the specimens of concrete and mortars at different ages. Sharif and Farhan (2011) investigated the effect of Cement Kiln Dust on characterization of Portland cement. The result show that when ordinary Portland cement mixed with 25% of CKD led to increase of 39% and 31% of the amount of water for normal consistency, with increasing weight percentage of CKD led to decrease in initial and final setting time (Hilal and Medulla, 2010).

Maslehuddin et al, (2009) investigate the estimation on the characterization of CKD when mixed with cement concretes. Sample of concrete were prepared with different weight percentage of CKD (0%, 5%, 10% and 15%). They reported that the replacement of OPC with CKD led to decreased in the compressive strength for all specimens, however, they observed that there was no significant reduction in compressive strength at 5% replacement. Similarly, Al-Harthy, Taha, and Al-Maamary (2003) stated that there is no beneficial increase in strength beyond 5% addition of CKD.

Bashter, Makarious, and El-Sayed Abdo (1996) used heavy weight aggregates of different mineral origin (Iimonite, Ilmenite, and siderite) for radiation shielding. They observed that those minerals can be used for radiation barriers or Shielding in concrete. Kansouh (2012) studied the shielding properties of Ilmenite-Limonite concrete, and ordinary normal concrete. The result show that Ilmenite-Limonite concrete is a better reactor of biological shielding than other concrete (serpentine concrete).

Abo-El-Enein et al. (2014) studied Physico-mechanical properties of high performance concrete using different aggregate with silica fume. Their results shows that fine aggregate with Ilmenite and air-cooled slag has higher physical and mechanical properties. They also observed that the utilization of Ilmenite and air-cooled slag produced heavy density concrete that are suitable for shielding gamma rays than conventional concrete.

MATERIALS AND METHODS

CEMENT AND FINE AGGREGATE

Ordinary Portland cement (OPC) of CEM I in conformity with BS EN 197 (2000) standard was obtained from local cement manufacturer in Nigeria. The Cement Kiln Dust (CKD) with the chemical composition shown in Table 1 was collected from Cement Industry located in North-Eastern Nigeria. Dry and clean sand was used to prepare the mortar specimens in this experiment. The physical test data of sand showed that it had: specific gravity = 2.4, relative density = 1.682 g/cm^3, fineness modulus = 2.01 and water absorption rate = 2.2%.

Table 1: Chemical composition of CKD

Chemical composition (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	So_3	Tio_2	P_2O_5	Mn_2O_3	LOI
CKD	11	4.48	1.99	44.45	1.02	0.57	0.08	1.25	0.2	0.18	0.14	33.63

ILMENITE TAILINGS

The ilmenite tailing was obtained from Mining and Germs Industry in North Central part of Nigeria. After collection of the materials various characterization tests were carried out and these include: The particle size distribution of the material was analyzed using a mechanical sieving device according to the process described in BS EN 13139 (2013). The results of particle size distribution of the ilmenite tailing shown in Fig. 2 falls within the fine aggregate medium grading limits (M) specified by BS 882 (1992) which is equivalent to zone II classification of BS 812 - 103.1 (1985). The fineness modulus of ilmenite tailing was found to be 3.50, which is above the range of 2.3 - 3.0 specified by BS EN 13139 (2013) Standard.

Fig. 1: Ilmenite Tailings

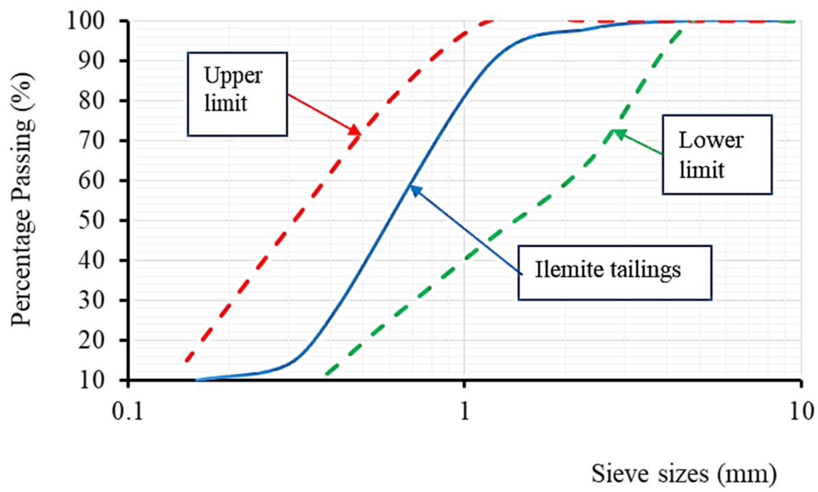

Fig. 2: Particle size distribution of Ilmenite Tailings

MIX DESIGN AND PREPARATION OF MORTAR

In this work, all mortar mixes were prepared by weight batching with a binder-to-fine aggregates ratio of 1:6 using w/c ratios of 0.45. The cement mortar mix were prepared in the Hobart mixer for 2–3 min of mixing.

The Mortar mix were prepared at 0%, 20%, and 40% 60%, 80% and 100% replacement of river sand by ilmenite tailings, while OPC was replaced with CKD at 10%, 15%, and 20% replacement as shown in Table 1. After mixing the mortar, cubes of dimension 50 x 50 x 50 mm were casted for each of the mortar mix and cured in water for 1, 3, 7, 14, 21, and 28 days tested for compressive strength.

Table 2: Mixed design for mortar

Percentage Replacement (%)	Sand (g)	Tailings (g)	Cement (g)	10% CKD (g)	Cement (g)	15% CKD (g)	Cement (g)	20% CKD (g)	Water (g) 0.45
0	3888	0	648	0	648	0	648	0	292
20	3110	778	583	65	551	97	518	130	292
40	2333	1555	583	65	551	97	518	130	292
60	1555	2333	583	65	551	97	518	130	292
80	778	3110	583	65	551	97	518	130	292
100	0	3888	583	65	551	97	518	130	292

EXPERIMENTAL WORK

Various experiment were conducted in this research work, these include water absorption and specific gravity, setting time of OPC and CKD, Water Absorption, Moisture Content and Compressive Strength.

INITIAL AND FINAL SETTING TIME

The initial setting time of OPC is important as, in addition to workability, it provides an indication of how long the mixture will remain in plastic condition. It is desirable for mortar to harden and develop strength within a reasonable time after it has been placed. For these reasons, the impact of CKD as a partial replacement of OPC on the initial setting time is very important. The setting reaction takes place by the partial dissolution of the pozzolana in water to produce silica in solution. The dissolved silica then reacts with the calcium hydroxide to form CSH gel (Berry, Hemmings, Zhang, Cornelius and Golden, 1994). The reaction between the CKD and OPC to produce the CSH gel produces a hydraulic setting mortar.

Vicat apparatus was used to estimate initial and final setting time of fresh mortar samples at normal consistency. This test was performed according to BS EN 196 - 3 (2016). Initial and final setting times are given in Table 3.

WATER ABSORPTION AND SPECIFIC GRAVITY

Water Absorption refers to the ability of a material to absorb and retain water. It depends on the material porosity, swelling capacity and receptivity. Water Absorption is characterized by the amount of water to ensure that all the aggregate were transferred.

Pycnometer and drying oven apparatus were used to carry out the test in determining the water absorption and specific gravity of river sand and ilmenite tailings as shown in Fig. 3.

Water absorption test was performed by keeping the samples immersed in water and removing the excess water on the surface of the samples after 24 hours, and measuring the saturated weight. After that, the samples were kept in the oven by maintaining 100±5°C for 24 hours. Oven dry weight of the samples was recorded and the water absorption capacity and Specific gravity were evaluated.

Fig. 3: Pycnometer on a weighing balance

COMPRESSIVE STRENGTH TEST

The preparation of the mortar mixture for the compressive strength was based on the mixed design in Table 1. Mortar cubes of 50 mm x 50 mm x 50 mm were prepared to evaluate the compressive strength of masonry mortars as specified in ASTM C109 (2013). A total of 576 cubes (3 samples from each mix) were prepared and tested at the end of 1, 3, 7, 14, 21 and 28 days of water curing with a temperature of 27 ± 3 °C.

RESULTS AND DISCUSSION

INITIAL AND FINAL SETTING TIME

Initial and final setting time tests were shown to yield different results on plain OPC paste and pastes having CKD. The reactivity of CKD is just like other hydraulic cement depends very much upon the specific surface area or particle size. The OPC cement with finer particles exhibits superior setting time behavior than CKD. This study shows that there is decreased in the initial setting time by raising the CKD level in the cement mixture over that of the plain cement paste.
Table 3 shows the setting time of ordinary Portland cement paste with CKD in various proportion of replacement. The plain cement paste (OPC) has an initial setting time of 90 minutes and the final setting time of 260 minutes. As the CKD replacement increases, the setting time decreases. The lower setting times values in OPC-CKD pastes compared with OPC pastes is considered to be affected from the high amount of lime and alkalis in CKD which accelerate hydration lead to fast setting. The results of initial setting of 20% CKD is 33.3% lower than OPC control (OPC) specimen. Similarly, the results of the final setting time of 20%CKD was 19.2% lower than that of ordinary Portland cement (control). These could be attributed to the presence of CaO in the CKD. Similar finding was reported by Latif, Naganathan, Razak, and Mustapha (2015). Unlike other pozzolanic materials, CKD tends to shorten the final setting time. The rate of retardation of final setting time decreases continuously with increase in CKD content up to 20%.

Table 3: Initial and Final Setting Time

Description	**Setting Time**	**CKD**		
	OPC	**10%**	**15%**	**20%**
Initial Time (min)	90	80	70	60
Final time (min)	260	230	220	210

SPECIFIC GRAVITY AND WATER ABSORPTION

The result of specific gravity and water absorption of sand and ilmenite tailings are shown in Table 5. The specific gravity of ilmenite tailings was 4.7, which is higher than the specific gravity of river sand. Caenn, Darley, and Gray (2017) similarly obtained high specific gravity of 5.1 in their research work. Specific gravity of river sand is generally range between 2.6 and 2.7 (Neville, 2011). The high specific gravity of ilmenite tailings could be attributed to the sources of the material which is a waste from ilmenite ore and suspected that the traces particles were accompanied along with the tailings. The water absorption for sand and ilmenite tailings are 4.35% and 2.20% respectively. Ilmenite Tailings absorb more water so it has high absorption rate, this tend to affect the hydration rate of mortar. The water absorption of fine aggregate generally varies in the range of 2 to 6% (Kosmatka, Kerkhoff, & Panarese, 2003). Thus, the water absorption of both sand and ilmenite tailings are within the range specified. The slight higher water absorption of ilmenite tailings compared to river sand could be attributed to prolonged exposure to weathering which might have also contributed to the increased porosity of the tailings (Onuaguluchi & Eren, 2012).

Table 4: Results of specific gravity and water absorption for Sand and Ilmenite Tailings

Aggregate types	**Specific gravity**	**Absorption (%)**
Sand	2.60	2.20
Tailings	4.70	4.35

COMPRESSIVE STRENGTH OF THE MORTAR SPECIMENS

Fig. 4 to 6 shows the behavior of compressive strength with curing time of the hardened cement mortar-specimens containing cement kiln dust and ilmenite tailings. It is observed that the strength development of cementitious blends is influenced by the CKD and the presences of ilmenite tailings. The increase of the amount of CKD from 0 - 20% and replacement of river sand (0-100%) results in the increase of the compressive strengths in all the ages. This could be attributed to the mineralogical and fineness properties of CKD and ilmenite tailings used in the experiments. The test data also revealed that the compressive strength of mortar specimens containing cement kiln dust and ilmenite tailings were higher than the control at all the curing periods. The compressive strength increase slightly with increased of cement kiln dust up to 20% CKD with 100% ilmenite tailings throughout the curing period. The percentage increase of compressive strength relative to control at 28 days for 10%, 15% and 20% CKD and 100% ilmenite tailings are 22%, 27% and 29%, respectively. This observed increase in the compressive strength could be partly attributed to the finer particles of ilmenite tailings which filled the pore and optimized the pore structure. It is equally suspected that the presence of titanium iron mineral in ilmenite tailings might resulted in the positive impact on strength attainment as reported by Mohamed and Paleologos (2018).

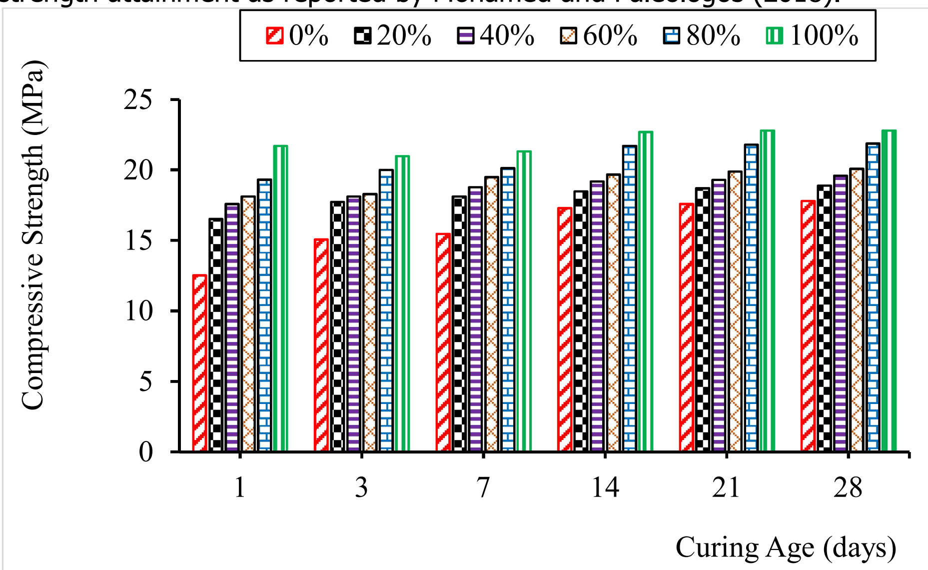

Fig. 4: Results of Compressive strength for 10% CKD

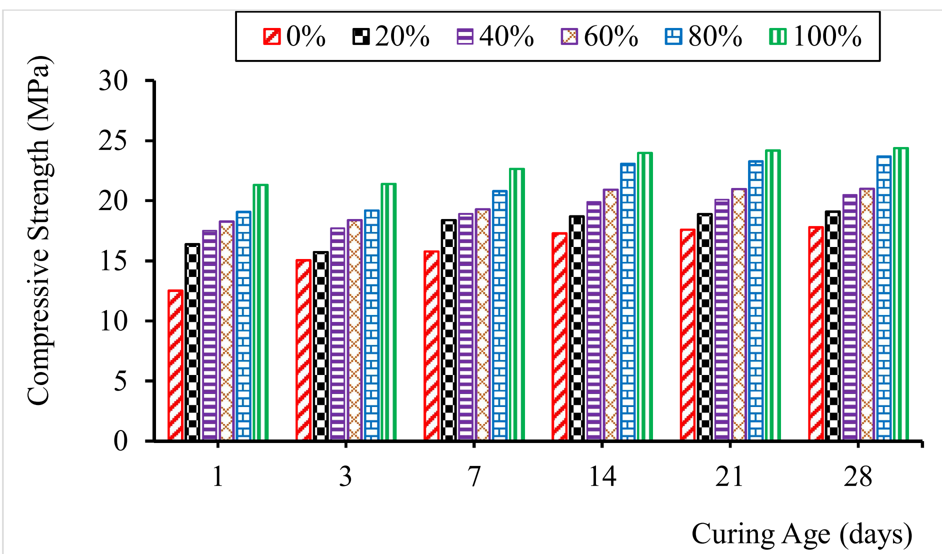

Fig. 5: Results of Compressive strength for 15% CKD

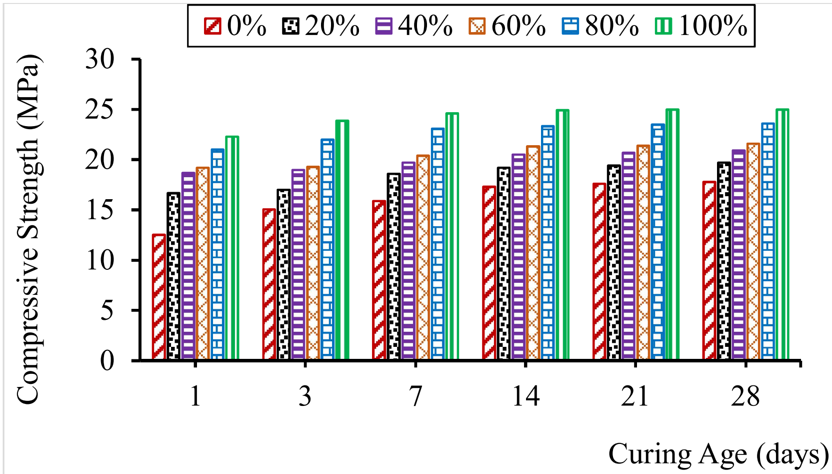

Fig. 6: Results of Compressive strength for 20% CKD

CONCLUSION

Based on the result of the test carried out the following conclusion were made:

1. Ilmenite tailings was found to be well graded and conform with grading for fine aggregates that is suitable for mortar production.
2. Ilmenite tailings absorb more water compared to river sand, however both are within the range specified
3. The results of setting time shows that cement kiln dust has low value of initial and final setting time as compared to OPC.
4. The compressive strength of mortar mixtures of CKD and ilmenite tailings were higher than the control specimen. The compressive strength of mortar improved with replacement of river sand by ilmenite tailings and this might be due to their high content of titanium iron mineral and micro-filling effects.
5. The overall 100% use of ilmenite tailings and CKD up to 20% as partial replacement of cement and sand, respectively has higher compressive strength compared to conventional mortar specimens.

RECOMMENDATIONS

From the findings of this research work, the following are recommendations are proffered;

1. Further research on other mechanical properties (tensile strength, flexural strength and modulus of elasticity) of mortar is imperative.
2. More mechanism study is necessary on ilmenite tailings, CKD materials and mortar specimens in terms of microstructure test.
3. The leaching issue on the use of ilmenite tailings should be examined
4. Mortar durability test as well as alkali silica reactivity (ASR) test should be carried out to determine the effect of ilmenite tailings and CKD on mortar specimens.

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