Performance of Gear Inner Wire-Reinforced Reactive Powder Concrete made with Unrefined Metakaolin

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Received: 4/5/2020 Reviewed: 21/5/2020 Accepted: 12/6/2020

Reactive Powder Concrete (RPC) is a new generation of concrete that is coarse aggregate-free cement-based material. RPC is characterized by highly dense matrix, ultra-high strength, excellent durability and economy. The wide application of RPC in special structures across the globe such nuclear power station, etc is becoming a concern due to lack of extensive study on the properties of RPC exposed to elevated temperatures. The research evaluated the effect of unrefined Metakaolin (MK) as substitute to silica fume and Gear Inner Wire (GIW) as fibre on the performance of RPC exposed to elevated temperatures (200°C-800°C). On the other hand, a reference sample produced with 20% silica fume was also tested. RPC specimens produced with 10%, 20% and 30% MK by weight of cement and a constant GIW content of 0.25% by weight of concrete were subjected compressive and absorption properties. Thermal deterioration of the RPC using Ultrasonic Pulse Velocity (UPV) was also assessed. Results show that RPC with 10%MK and 0.25% GIW has better performance in terms of residual strength, water absorption and UPV up to 800°C compared to the control sample with silica fume. To enhance the residual properties of this type of RPC, 10% unrefined MK and 0.25%GIW is required. Therefore, RPC of this type can be used in structures exposed to elevated temperature.

Keywords: Reactive powder concrete, Metakaolin, gear inner wire, elevated temperature, residual strength

Introduction

Reactive powder concrete (RPC) is a new generation of concrete that is self-consolidating and coarse aggregate-free cement-based material. RPC is also referred to ultra-high-performance concrete and was first developed by Richard & Cheyrezy in 1995. It is made up of fine powdered-materials (Yazici, Yardimci, Aydin, & Karabulut, 2009). RPC is used in the construction of many important structures such as long span bridges, sky scrapers, nuclear stations, etc. due to its ultra-strength and durability than the conventional concrete.

RPC has a compressive strength of more than 150N/mm^2, flexural strength of up to 30N/mm^2 (Nematollahi *et al.*,2012) and tensile strength of up to10N/mm^2 (Qureshi et al.,2017) using silica fume as the pozzolan. A compressive strength of 80N/mm^2, tensile strength of 10 N/mm^2 and flexural strength of 20 N/mm^2 was achieved using local materials available in Pakistan (Qureshi et al.2017). But the use of cement in conventional RPC is high and Silica Fume (SF) content is up to 25% by weight of cement. Haghighi, et al. (2007) states that the optimum amounts of silica fume in RPC might be 20% or 25%. Hiremath and Yaragal (2017) used 20% SF as optimum. However, SF is costly and not readily available in some developing countries like Nigeria. Other shortcoming of SF is that it

causes shrinkage due to heat of hydration (Peng *et al.*, 2006).

Using other mineral admixtures other than SF in the production of the RPC seems to be a feasible solution to the problems (Rougeau & Borys, 2004; Yazici, Yardimci, Aydin, and Karabulut 2009; Yazici, Yardimci, Yigiter, Aydin, and Turkel 2010; Agharde & Bhalchandra, 2015). When fly ash was used to replace SF, compressive strength of between 62.9 N/mm^2 to 324N/mm^2 and a flexural strength of 8.8 N/mm^2 to 32 N/mm^2 were obtained (Yazıcı, Yigiter, Karabulut & Baradan 2008; Ding 2010; Demiss, Oyawa & Shitote 2018). A compressive strength of 128 N/mm^2 to 250 N/mm^2 and a flexural strength of between 25.6 N/mm^2 to 32 N/mm^2 were obtained with ground granulated blast furnance slag (Yazici *et al.* 2009; Peng, Hu & Ding 2010; Nguyen, Guang, Klass and Oguzhan 2011). Moreover, Asteray, Oyawa & Shitote (2017) observed that a 28 days compressive strength of 57.3 N/mm^2 was achieved when rice husk ash was used to replace the SF content in RPC. The pozzolan with similar properties to those of silica fume is metakaolin.

There is large deposit of kaolinitic clay from which metakaolin is obtained across Nigeria (Ibrahim, Okoli and Dahiru 2016). Study showed that 15% of silica fume has been successfully replaced with commercial metakaolin in the production of RPC, which indicated savings (Smith Gururaj & Siddesh 2015). In normal concrete, unrefined metakaolin has been shown to improve the strength and durability properties of concrete similar to the refined one (Badogiannis & Tsivilis 2009). Therefore, further savings can be realised if the refined metakaolin is replaced with the unrefined one in the production of RPC. Another important ingredient of RPC is steel fibre.

Steel fibre provides an economic approach which minimizes plastic shrinkage cracks, reduces the severity of thermal cracking, impact resistance; increase overall durability and toughness of concrete, etc. (Mehrotra, 2009). The use of fibre in RPC is of great importance especially when the RPC is exposed to elevated temperatures during service. For example, Canbaz (2014) and Ju et al.(2015) noted that spalling starts at 300^{0}C in unfibred RPC whereas fibred specimen shows no spalling. Despite the effect of conventional fibres in enhancing the performance of RPC on fire, these materials are expensive and or not readily available in developing countries like Nigeria. However, some materials have been used as alternative to conventional fibres (Foti, 2013; Ashish & Rinku 2012) and more recently is that of waste GIW as fibre in mortar production (Ibrahim et al.,2018). It was concluded that 2% of the waste GIW was optimum. ACI 544.3R-10, however, states that the volume of steel fibre required in a concrete is between 0.25% and 2%. The use of steel fibre has been shown to improve the performance of RPC at elevated temperatures.

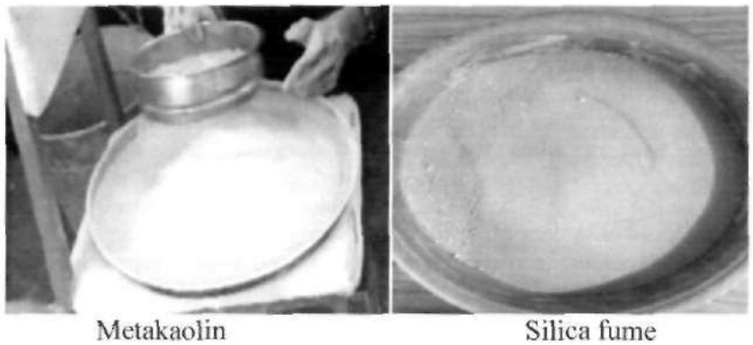
Zheng *et al.* (2013) investigated the influence of steel fibre content on the performance of RPC. Results show that 2% steel fibre can effectively prevent explosive spalling, reduce or delay crack extension and crack propagation, and significantly enhance the compressive and tensile strength of the RPC. Steel microfibers also improved both compressive and flexural performance of RPC at to elevated temperature (Zheng et al.,2012; Yazici *et al.*,2013; Way & Wille 2015; Hiremath & Yaragal, 2018). According to Liu and Huang (2009), the fire performance of RPC is of importance and needs to be investigated prior to the application in building construction.

This paper evaluated the performance of RPC made from locally unrefined metakaolin, reinforced with GIW exposed to elevated temperatures. In order to further reduce the cost of producing the RPC, pressure and heat treatment were not applied. Additionally, using the unrefined MK also may led to reduction in the cost of producing the RPC by eliminating refining and beneficiating process associated with refined MK.

Materials and methods

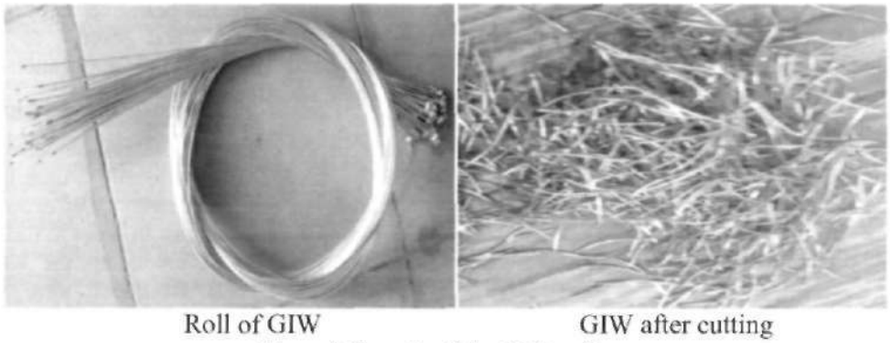
Materials

The cementitious materials used for this research are cement; metakaolin (MK) and densified silica fume (SF). Samples of the MK and SF are shown in Figure 1. The cement is Dangote brand of Portland Limestone. MK was produced by heating unrefined kaolin at 750°C for 2 hours in an electric furnace. This is to ensure complete conversion of kaolin from crystalline to amorphous form as explained in sub-section 3.1.1 and shown in Figure 4. The kaolin was sourced from a kaolinitic clay deposit situated in Getso, Kano State. The silica fume was supplied from Malaysia and GIW was obtained spear parts shops within Kaduna metropolis, Kaduna state. Geometry of the GIW (fibre) is shown in Table 1 and trial test results showed that 0.25% of GIW was optimum for RPC production which is within the 0.25% to 2% range states by ACI 544.3R-10. The chemical composition and physical properties of the cementitious materials are presented in Table 2. Based on the summation of the major oxides (SiO_2 + Al_2O_3 + Fe_2O_3) which is above 70%, the MK can be satisfied as N- Class pozzolan as recommended in by ASTM C 618-05. Polycarboxylate ether based super plasticizer Conplast SP 430 conforming to ASTM C 494 (in the range of 2.8% to 5.0%) was used to achieve the required consistency of the mixes depending on the quantity of the cementitious materials. Naturally occurring river sand with particle sizes of 600μm - 150μm and absorption of 4% was used as fine aggregate.



Metakaolin Silica fume

Figure 1: Cementitious materials



Roll of GIW GIW after cutting

Figure 2: Sample of the GIW as fibre

Table 1: Properties of gear inner wire as fibre

Diameter (mm)	Length (mm)	Aspect ratio (L/D)	Tensile strength (N/mm^2)
0.28	12	43	1623

Mix proportioning

Mix design of the RPC produced evolved from several trials due to the absence of an established design method. The mix proportion used by Richard and Cheyrezy (1995) was used as a basis for the experiment based on trial and error method. The ingredients used in the study (for control mix) were cement, silica fume, fine sand, GIW as fibre, superplasticizer, and water. The specimens were then produced by totally replacing the SF content with MK. The MK was used in different percentages (10%, 20% and 30%) of the weight of cement in order to find the most appropriate quantity to be used in the production of the RPC.

Specimens preparation and curing

Cleaned and oiled moulds were used to prepare the specimen for determining the compressive strengths of RPC with SF as control and MK as specimens. The cementitious materials were dry mixed in a mortar mixer for about one minute at low speed. Premixed water (about 80% of the mixing water) and superplastizer were added into the mixer and the mixing continued for three minutes at medium speed. Fine sand and GIW as fibre were then added into the mixer and mixing continued for four minutes. The remaining mixing water (about 20%) was then added to the mixer and mixed at high speed for another four minutes. Finally, the mixer was then returned to the medium speed and mix for three minutes (Hiremath & Yaragal 2017). The final mix was then poured into a mortar flow table cone to full and allowed to flow under its own weight on flat tray (as shown in Figure 3) and the flow value measured at 270mm±5. The total mixing process took fifteen minutes and flow test was conducted on each batch to ensure flowability of RPC mix. The specimens were cast and kept in moulds at 27 ^{0}C for 24 hours. Specimens were then taken out from the moulds and kept under standard water curing till 28 day of test.

Methods

Fresh property of RPC was determined by the flowability of the fresh mix using the cone of a flow table test as per ASTM C143. The test was carried out by filling the mini-slump cone. The cone was then removed slowly to make fresh mix flow by itself evenly over the flat tray and measure the flow spread diameter. The final spread diameter is taken as an average of four measurements over the top of the spread. The average diameter of the RPC mix indicated the flow value of the RPC. From the prepared mixture of the RPC, the compressive strength test was conducted for cube of size 50mmx50mmx50mm according to BS EN 12390-3:2002 at 28 days. The samples were divided into two. One set was crushed and the other was exposed to elevated temperatures. The elevated temperature test on the specimens was carried out using an automatic electric furnace. Before heating, the specimens were oven dried at 105±5 ^{0}C for 24 hours to minimize the effect of explosive spalling. The specimens were heated to 200, 400, 600 and 800 ^{0}C in the furnace at a heating rate of 2.5^{0}Cmin^{-1}. In order to achieve a thermal steady state, the specimens were maintained for 2 hours at the peak temperature. After heating, the furnace was switched off and the specimens were allowed to cool in the furnace environment for about 2 hours with the furnace door closed. Later, the specimens were removed from the furnace and allowed to cool to ambient temperature in the laboratory environment before testing. The tests conducted are residual and normalized compressive strength, ultrasonic pulse velocity and absorption.



(a) During pouring (b) During measuring

Figure 3: Flowability of RPC

Results and Discussion

3.1 Characterization

Tests conducted under characterization include XRD, chemical composition and strength activity of MK, so also the geometry of the GIW.

X-Ray diffraction

Figure 4 shows the XRD pattern of kaolin and calcined kaolin (Metakaolin). It can be observed that the calcined kaolin has reduced peak than the kaolin. The 2θ value (between 10 -30) shows hallow in shape in the calcined kaolin and reduction in intensity which indicates that the material used in the experiment has been transformed from crystalline to amorphous form (Badogiannis *et al.*, 2005; Wang *et al.*, 2005).

Chemical composition of constituent materials of RPC

Table 2 shows the chemical compositions of the cementitious materials of RPC using X-Ray Fluorescence spectrometry instrument (XRF). For metakaolin (a pozzolanic material), the summation of the major oxides ($SiO_2 + Al_2O_3 + Fe_2O_3$) from Table 2 is 88.29% which satisfied the requirement of ASTM C 618-05 for class N pozzolanic materials. Moreover, the proportion of SO_3 (0.18%) is below 4% and loss on ignition (LOI) is 1.80%. Therefore, the MK used for this research satisfied the requirement of ASTM C 618-05 for class N pozzolanic materials.

Moreover, Table 2 shows the strength activity index results of MK. According to the ASTM C311, a material is considered pozzolanic if its Strength Activity Index (SAI) is ≥75%. The results of SAI conducted on MK indicated that the SAI of MK is 87% at 28 days and is above the minimum specified. Therefore, MK used in this research is reactive.

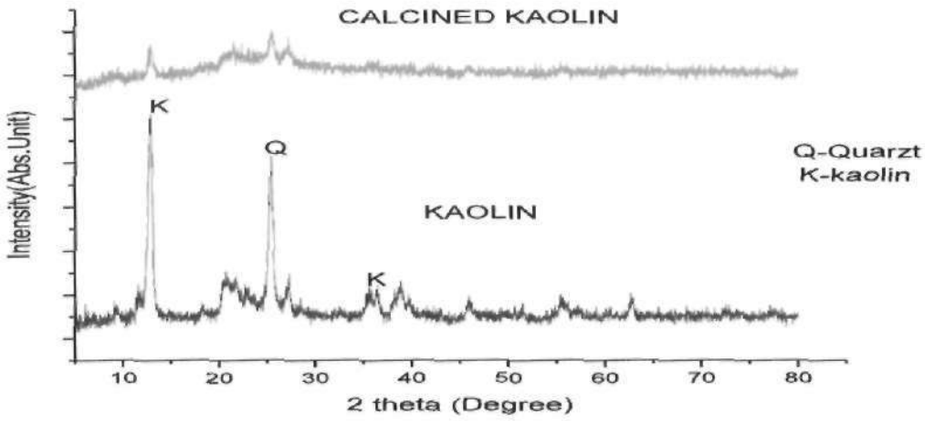


Figure 4: XRD result of Metakaolin

Table 2: Oxide c compositions and physical properties of RPC constituents

Oxide (%)	Sand	Cement	Silica fume	Metakaolin
SiO_2	86.53	17.519	92.00	65.05
Fe_2O_3	2.94	2.768	0.50	2.59
Al_2O_3	1.64	4.74	0.70	20.65
CaO	0.40	71.297	0.50	0.82
CuO	0.00	0	0	0.02
NiO	0.00	0	0.015	0.03
MnO	0.01	0.072	0.128	0.08
Cr_2O_3	0.00	0	0.006	0.03
TiO_2	0.00	0.105	0.071	0.00
MgO	0.60	0	0.50	1.66
SO_3	*0.10*	*0.00*	*0.00*	*0.18*
ZnO	0.00	0.007	0.006	0.01
$SiO_2 + Al_2O_3 + Fe_2O_3$				88.29
LOI	0.84	3.492	3.00	1.80
Physical properties				
Surface area (m^2 /kg)			2 0, 000	509.0
Strength activity index (%)			-	87
Specific gravity			2.21	2.53

Flowability of RPC

The flowing nature of the RPC produced (as a self-compacting concrete) was achieved by the use of superplasticizer. The targeted flow of 270±5mm (as shown in Figure 3) on the MK and silica fume RPC was achieved by the addition of the superplasticizer. It was observed that the superplasticizer (sp) dosage for RPC increased with the increasing MK content (Dinakar *et al.*, 2013) due to its high surface area.

Physical observation

Figure 5 and 6 shows the different RPC samples exposed to 27°C, 200°C, 400°C, 600°C and 800°C. It can be observed that at 200°C there is no colour change (compared to the sample in Figure 5) and visible crack development on various RPC surfaces. Up to 600 °C there is no any visible crack development on the surfaces of the various RPC sample surfaces. However, colour change was observed. At 400°C and 600°C the color changed from grey to light brown. At 800°C the color changes from light grey to light green with very minor micro cracks on the RPC surfaces. The change in color at different temperatures could be attributed to the dissolution and oxidation of iron contained in the fine aggregates (Lee, Choi and Hong (2010). It can be inferred that the presence of the micro cracks at 800 °C could

be due to the presence of the GIW that enhanced the physical performance of the RPC specimens (Zheng *et al.*, 2013; Canbaz, 2014; Ju *et al.*, 2015).

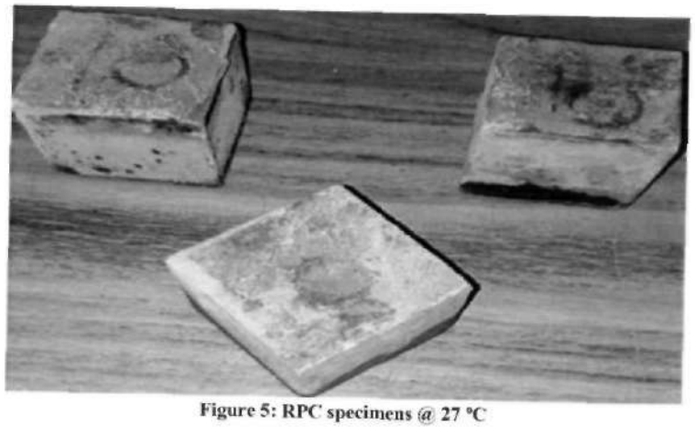


Figure 5: RPC specimens @ 27 °C



Figure 6: RPC sample exposed to elevated temperatures

Residual and Normalized compressive strengths

Unfibred RPC

Figure 7(a & b) shows the residual and normalized compressive strength of the unfibred RPC samples. It can be observed that each temperature range has a different pattern of strength gain or loss. At 200 °C, 20%SF, 10%MK and 30%MK samples show 28%, 45% and 27% higher compressive strength respectively compared to the RPC specimens at 27°C. The increase in the compressive strength of these samples could be due evaporation of free water that strengthened hydrated cement paste at 200°C (Hiremath and Yaragal 2018). At 400 °C, there was an increase in the compressive strength of 10%MK RPC while a decrease in the strength of all other RPC samples. A loss of strength within the range of 7%-43% was observed at 600°C in all samples with no visible micro crack on their surfaces but 10%MK performed better. At 800 °C, there was greater loss of strength and hair-like micro cracks on the surfaces of all the RPC samples. The residual strength at this temperature was between 56% and 65%.

Generally, the strength loss decreases with increase in MK content (10-30%) in the RPC when the temperature was varied between 200 °C to 800°C. This is an indication of better performance of MK in retaining strength at elevated temperature. The benefit of replacing MK with SF was more when 10%MKwas used for unfibred specimens. The deterioration of strength at elevated temperature could be due to the decomposition of calcium hydrate gel that causes severe deterioration in the RPC (Demirel & Kelestemur, 2010). Moreover, the normalized strength of the unfibred RPC followed the same trend as the residual strength. Residual compressive strength is the percentage of the strength retained by concrete with respect to the strength of the unheated sample at 27°C.

Fibred RPC

Figure 8 (a & b) shows the residual and normalized strength of the fibred RPC samples. It can be observed that each temperature range has a different pattern of strength gain or loss. It was also observed that each temperature range had a particular pattern of strength gain or loss. At 200 °C, 20% SF, 10%MK, 20% MK and 30%MK have shown 10%, 23%, 17% and 32% higher compressive strength compared to the RPC at room temperature respectively. The increase in the compressive strength of these samples could be due evaporation of free water that strengthened hydrated cement paste at 200 °C (Zheng *et al.*, 2013; Hiremath & Yaragal, 2018). The 20%MK has similar residual compressive strength with 20%SF. At 400 °C, there was an increase in the compressive strength of 20%SF, 10%MK and 30%MK in the range of 7-17% with 10%MK recording the highest (17%). However, 20%MK RPC remains relatively the same. A loss of strength was observed at 600°C. The strength loss was within the range of 3-41%. All samples have no any micro crack on their surfaces but 10%MK performed better (with just 3% loss) than all other samples. The 20%SF recorded the highest loss of strength (41%). At 800 °C, there was greater loss of strength and hair-like micro cracks on the surfaces of all the RPC samples. The residual strength at this temperature is 50-69% with 20%SF and 20%MK recording the highest and the least respectively.

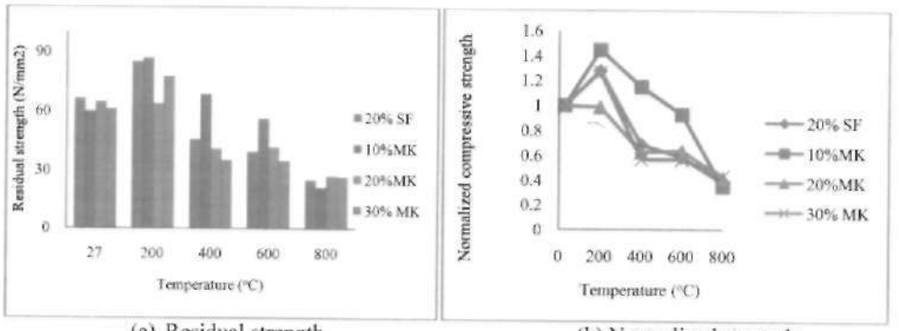
Therefore, it was observed that the strength loss decreases with increase in MK content (10-30%) in the RPC when the temperature was varied between 200°C and 400°C. This is an indication of better performance of MK and GIW in retaining strength at elevated temperature. The benefit of replacing MK with SF was more when 10%MKwas used. The deterioration of strength at elevated temperature (400–800 °C) could be due to the decomposition of calcium hydrate gel that causes severe deterioration in the RPC (Demirel & Kelestemur, 2010; Way & Walle 2015) as well as the presence fibre which prevented proper bonding and may also be regarded as the critical temperature range for the strength loss of fibre concrete (Peng *et al.* 2006) and the bond strength in the concrete matrix degraded significantly

with increasing temperature for straight steel fibres (Abdallah *et al.*,, 2017)

Additionally, the normalized strength of the fibred RPC followed the same trend as the residual strength and it was calculated as the percentage of the strength retained by concrete with respect to the strength of the unheated sample at 27°C.

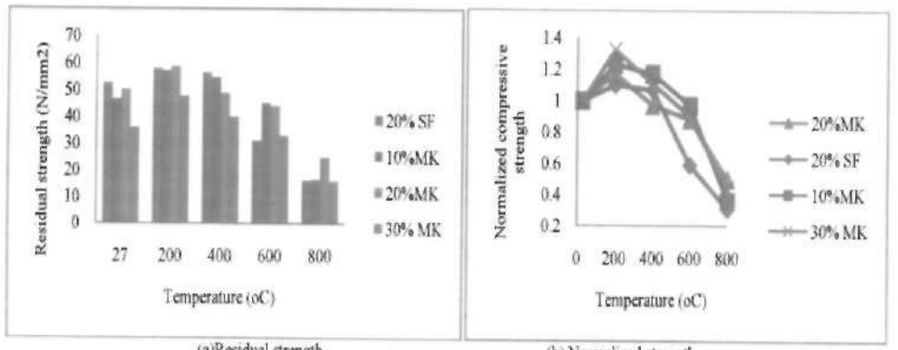
Ultrasonic Pulse Velocity (UPV)

Figure 9 (a & b) depicts the ultrasonic pulse velocity (UPV) of unfibred and fibred RPC made from 20%SF, 10%MK, 20%MK and 30%MK exposed to elevated temperature. It can be seen that the UPV value increased at 200°C as compared to that at ambient temperature. The increase in strength could be due to steam curing caused by hot vapour (Houa *et al.*, 2017). There was loss in UPV value in the range of 1.9-3.8km/s at 400 °C, which indicated decrease with an increasing temperature. Therefore, it can be concluded that at temperature 200°C and 400°C all the RPC samples were good while at 600 °C to 800 °C the RPC samples were in doubtful condition based on an International Atomic Energy Agency, (IAEA, 2010) classification. Generally, the 20%MK seemed to perform better than the 20%SF, 10%MK and 30%MK by having higher UPV values across the temperature.



(a) Residual strength (b) Normalized strength

Figure 7: Residual and normalized strength of unfibred RPC



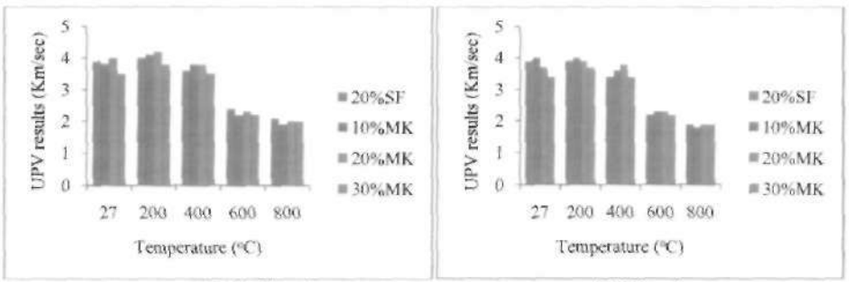
(a)Residual strength (b) Normalized strength

Figure 8: Residual and normalized strength of fibred RPC

Figure 9b shows the ultrasonic pulse velocity (UPV) of fibred RPC made from 20%SF, 10%MK, 20%MK and 30%MK. It can be seen that the UPV value increased at 200 °C as compared to that at ambient temperature (27°C). The increase in strength could be due to steam curing caused by hot vapour (Houa *et al.*, 2017). There was loss in UPV value (3.4) of 20%SF and 30%MK which indicate that these samples are in doubtful condition. However, for 10% MK and 20%MK at 400 °C, their UPV values are still in good condition (IAEA2010). Therefore, the high UPV values could be due to the presence of the WGIW fibre that tends to strengthen the RPC samples. At 600 °C to 800 °C, the RPC were in doubtful conditions.

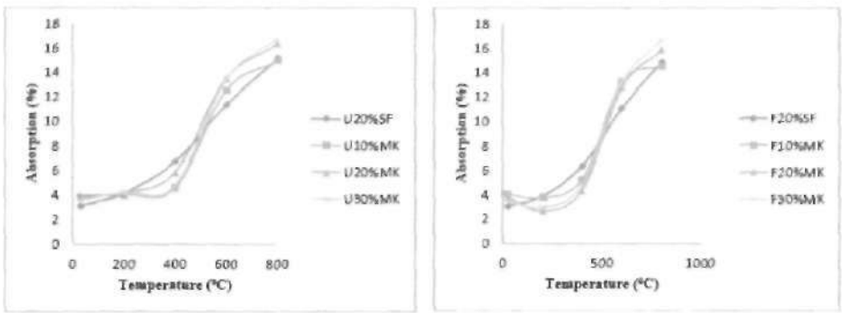
3.6 Water Absorption Capacity (%) of RPC

Figure 10a and 10b show the water absorption capacity of the unfibred and fibred RPC exposed to elevated temperature. At 27 °C, the water absorption of the unfibred samples (20%SF, 10%MK, 20%MK and 30%MK) were 3.1%, 3.8%, 4.0% and 3.5% respectively with 20%SF recoding the least and 20% MK recording the highest absorption. When the samples were exposed to 200 °C, a slight increase in water of between 2.5 to 20% was observed in RPC made with MK, with an even more pronounced increase (32%) in RPC made with SF. Significant increase in the water absorption was noticed in all the samples of the RPC when the temperature was raised between 400 °C to 800 °C. The 20% SF recoding the least (15.1%) and 30%MK recording the highest (16.7%). Therefore, above 200 °C RPC made with MK was observed to absorbed more water than the 20%SF sample. This confirms the claim of Aghabaglou *et al.* (2014).



(a) Unfibred (b) Fibred

Figure 9: Ultrasonic Pulse Velocity Results of RPC



(a) Unfibred **(b) Fibred**

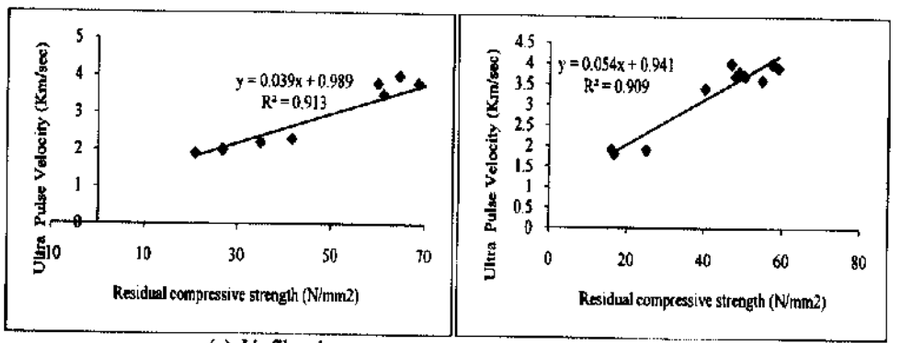
Figure 10: Water absorption capacity of RPC

Relationship between ultra-sonic pulse velocity and residual strength

Figure 11 (a & b) shows the relationship between residual strength and Ultrasonic Pulse Velocity (UPV) for the unfibred and fibred RPC samples made from different percentages of MK. It can be seen that a linear relationship exists between the residual compressive strength and UPV having coefficient of correlation (R^2) value of 0.913 and 0.909 for unfibred and fibred RPC. This means that there is strong linear relationship (excellent reliability) between residual strength and UPV of the RPC samples (Koo & Li 2015). Therefore, UPV can be used to describe the residual compressive strength of RPC made with unrefined MK.

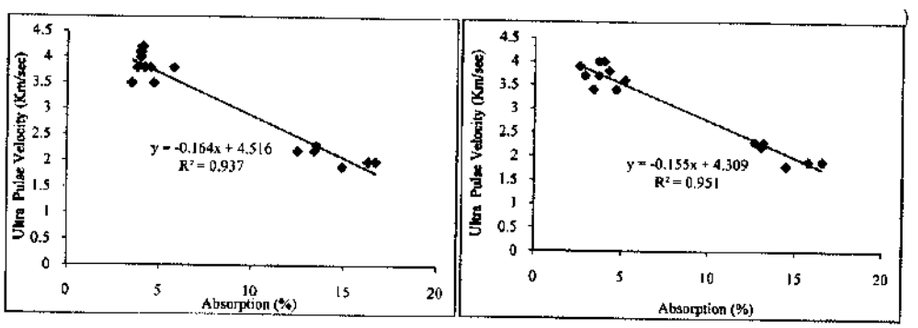
Relationship between ultra-sonic pulse velocity and absorption

Figures 12 (a & b) shows the relationship between absorption and Ultrasonic Pulse Velocity (UPV) for the unfibred and fibred RPC samples made from different percentages of MK. It can be noticed that a linear relationship exists between the absorption and UPV having coefficient of correlation (R^2) value of 0.937 and 0.951 for unfibred and fibred RPC. Therefore, a relationship between absorption and UPV (excellent reliability) of the RPC samples means that UPV can be used to describe the absorption of such samples (Koo & Li 2015).



(a) Unfibred (b) Fibred

Figure 11: Relationship between UPV and Residual compressive strength of RPC



(a) Unfibred (b) Fibred

Figure 12: Relationship between UPV and absorption of RPC

Conclusions

Reactive powder concrete was produced using 10%-30% of unrefined metakaolin as substitute of silica fume and gear inner wire as fibre. XRF, XRD and loss on ignition were used to ascertain the pozzolanic properties of the unrefined MK used. When the RPC specimens produced were exposed to elevated temperatures, sample with 10% unrefined MK and constant 0.25% GIW showed better performance compare to the reference sample (20%SF) in terms of residual strength, absorption and UPV. Moreover, UPV can be used to reliably predict residual compressive strength and water absorption capacity of the RPC. To enhance the residual properties of this type of RPC, 10% unrefined MK and 0.25%GIW is required. Therefore, RPC of this type can be used on structures exposed to elevated temperatures below 800°C and can be easily produced without necessarily the need for pressure and heat treatment.

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