

Suitability of Unrefined Metakaolin and Gear inner Wire for the Production of Reactive Powder Concrete

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Received: 4/5/2020

Reviewed: 1/6/2020

Accepted: 19/6/2020

Reactive Powder Concrete (RPC) is an ultra-high strength concrete that is coarse aggregate-free cement-based material. RPC constitutes of cement, Silica Fume (SF), fine sand, quartz sand and steel fibre as its ingredients. SF and steel fibre are relatively expensive in Nigeria due to their non-availability. Also, SF content of RPC is higher compared to conventional concrete. This paper examines the effects of unrefined Metakaolin (MK) as substitute to silica fume and Gear Inner Wire (GIW) as fibre on the properties of RPC. The unrefined MK used in the production of the RPC was in four volume fractions by weight of cement (0% - 30%) at an interval of 10% while GIW with 12mm length at a constant volume of 0.25% by weights of concrete was used. The specimens were subjected to compressive, tensile and flexural strengths tests for up to a curing age of 180 days. Control specimen with 20% SF was also produced and tested. The results show that 20% unrefined MK can be used to produce RPC with 72.5N/mm^2 compressive strength and 0.25% GIW by weight of concrete can be used to produce RPC with tensile strength and flexural strength of 7.1N/mm^2 and 22.3N/mm^2 respectively. It was concluded that 20% unrefined MK can suitably replace SF in RPC while 0.25% GIW can be used to enhance tensile and flexural strengths of the RPC. Therefore, Nigerian unrefined MK and GIW are suitable in RPC production.

Keywords: gear inner wire, reactive powder concrete, silica fume, strength, unrefined Metakaolin

Introduction

The development of an ultra-high strength and ductile concrete called Reactive Powder Concrete (RPC) was first made possible by Richard and Cheyrezy (1995). RPC is produced by the application of a certain number of basic principles relating to the composition, mixing and treatment of the concrete. It was developed through microstructure enhancement techniques (Yazici *et al.*, 2009) and a lot of researches carried out across the globe indicate that RPC is a future concrete material due to its high mechanical properties.

RPC has compressive strength of more than 150N/mm^2 , flexural strength of up to 60N/mm^2 (Richard & Cheyrezy, 1995) and tensile strength of up to 10N/mm^2 (Qureshi *et al.* 2017) using silica fume. However,

Qureshi *et al.* (2017) obtained a compressive strength of 80N/mm^2 , tensile strength of 10N/mm^2 and flexural strength of 20N/mm^2 with local materials available in Pakistan. RPC exhibits varied compressive strength when cured under different conditions.

A compressive strength of between 170N/mm^2 and 202N/mm^2 for heat treated specimens is possible; up to 400N/mm^2 for autoclaving and between 130N/mm^2 to 150N/mm^2 for non-heat treated specimens (Cwirza, *et al.*, 2008; Yazici, *et al.*, 2009; Tam *et al.*, 2010; Yazici, *et al.*, 2010; Maroliya, 2012). However, the amount of silica fume (SF) in conventional RPC is up to 25% (by weight of cement) and it is not readily available in developing countries like Nigeria. Peng *et al.*, (2015) noted that

SF is expensive and increases heat of hydration that causes shrinkage problems in concrete. Additionally, steel fibre used in RPC is also not available in Nigeria. The absence of the two major materials of RPC production is a setback to concrete technology in Nigeria.

Therefore, other mineral admixtures have served as alternative to SF (Kushartomo *et al.*, 2015; Agharde & Bhalchandra, 2015; Yazici *et al.*, 2010; Yazici *et al.*, 2009; Rougeau & Borys, 2004) in the RPC production. A compressive strength of between 62.9 N/mm² to 324N/mm² and a flexural strength of 8.8 N/mm² to 32 N/mm² were obtained using fly ash as partial replacement of SF (Yazici *et al.*, 2008; Yazici *et al.*, 2009; Ding 2010; Demiss, Oyawa & Shitote, 2018). When ground granulated blast furnace slag was used, compressive strength of 128 N/mm² to 250 N/mm² and a flexural strength of between 25.6 N/mm² to 32 N/mm² were obtained (Yazici *et al.*, 2009; Peng *et al.*, 2010; Nguyen *et al.*, 2011). Asteray *et al.* (2017) observed that a compressive strength of 57.3 N/mm² was achieved at 28 days when rice husk ash was used to replace the SF.

Moreover, some materials were used as fibre in the production of mortar and concrete in areas where conventional fibres are not available. The incorporation of waste fibres was investigated (Jalal 2012; Meddah & Bencheikh, 2009). Results of compressive and flexural strengths revealed that the addition of waste metallic fibre up to 1.5% did not affect the compressive strength but higher volume fraction decreased the compressive strength. Pereira de Oliveira & Castro-Gomes (2011) and Foti (2013) studied the effect of waste polyethylene terephthalate (PET) on properties of mortar and concrete respectively. Findings revealed that the incorporation of the PET improves flexural strength and toughness with 1.5% as optimum for desired workability. Research on the use of waste materials like lathe waste, soft drink bottle caps, etc. at 1% of total weight of concrete as fibres was carried out by Murali *et al.*, (2012). There was

increase in compressive strength in the concrete specimens by 41.25% and tensile strength by 40.81%. More recently in Nigeria, Ibrahim *et al.* (2018) used waste gear inner wire as fibre (WGIW) in mortar production. Outcome of the research showed that the fibred mortar sample has higher compressive and tensile strengths at 56 days by 19% and 21.1% respectively than the unfibred and concluded that WGIW at 2% volume fraction could be used as fibre in mortar production.

Therefore, in order to produce RPC in Nigeria, similar, available and alternative material to SF and steel fibre in terms of performance need to be investigated with a view to cutting down importation cost. Metakaolin (MK) has been found to perform similar to SF on the properties of concrete (ACI 232.1R-00) with additional advantage of improving tensile strength and bond strength (Vipat & Kulkarni, 2016) and up to 8%MK enhances tensile strength (Haroon *et al.*, 2017). However, Badogiannis *et al.* (2005) claimed that 10%MK is more favourable. Further savings can be obtained if the refined MK is substituted with the unrefined one (Badogiannis *et al.*, 2005). There are large deposits of kaolinitic clay from which MK is obtained across Nigeria (Ibrahim *et al.*, 2016) and an estimated reserve of about two billion metric tons of kaolin deposit are scattered in different parts of Nigeria (Foraminifera Market Research, 2016; Raw Material Research and Development Council, 2019). This research focused on the production of RPC from unrefined MK as substitute of SF and GIW as steel fibre with a view to establishing their suitability.

Materials and Methods

Materials

Cement

The cement used was Dangote blended cement of 43.5 grades that conformed to the requirements of BS EN 197-1:2011. The chemical composition of the cement is presented in Table 3.

Metakaolin

The Metakaolin (MK) was produced by heating unrefined kaolin at 750°C for 2 hrs

in an electric furnace with the aim of converting the kaolin from crystalline to amorphous form. The kaolin was sourced from a kaolinitic clay deposit situated in Getso, Kano State. Figure 1 shows the MK sample during sieving process and its physical properties and chemical compositions are presented in Table 3.

Silica Fume

Densified silica fume (SF) supplied from Malaysia was used for the experiment. It exists in grey powder form (as shown in Figure 1) and contains silicon oxides and no chloride or other potentially corrosive substance. The chemical composition and physical properties of the SF as per ASTM C618-05 are presented in Table 3.

Fine sand

Naturally occurring river sand with particle sizes of 600 μ m - 150 μ m and absorption of

4% was used as fine aggregate. The sand sample contains majorly of silicon and iron oxides as shown in Table 3.

Gear inner wire

Gear Inner Wires (GIW) main for bicycles brakes were sourced and cut into pieces (as shown in Figure 2) to serve as fibre in the production of the RPC. The GIW used in the research has a length of 12mm, diameter of 0.28mm and aspect ratio of 43. The geometry of the fibre is shown in Table 1.

Superplasticizer

Polycarboxylate ether based super plasticizer (Conplast SP 430) conforming to ASTM C 494 was used to achieve the required consistency of the RPC mixes produced.

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Figure 1: Cementitious materials

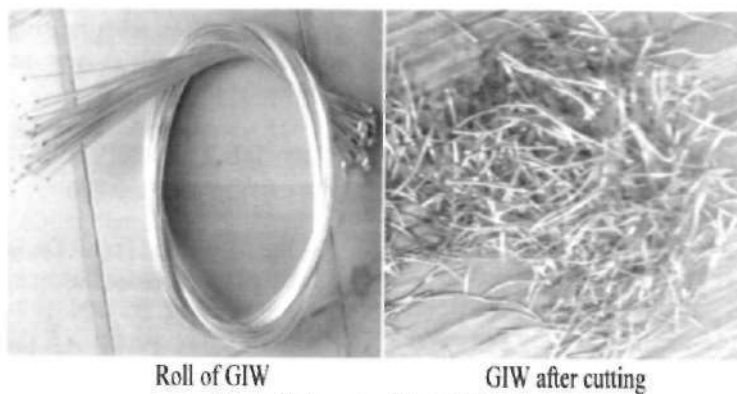


Figure 2: Sample of the GIW as fibre

Table 1: Properties of gear inner wire (GIW) used as Fiber

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

Mix Proportioning

The RPC mix design was evolved from several trials tests as a result of non-established design method. Proportions used by Richard and Cheyrezy (1995) were used as a basis for the trials. The amount of the GIW used in the research was also obtained through trial. The ingredient used in this study for the control mix include cement, silica fume (SF) by weight of cement, fine aggregates, GIW as fibre, superplasticizer, and water. The specimens were then produced by totally replacing the SF content with MK (using 10%, 20% and 30% of the weight of cement). The mix proportions of the RPC specimens are presented in Table 2. Different mixes were labeled for identification, based on the type and content of the pozzolanic material used in their production. For instance, 20SF means, the specimen was produced with 20% silica fume (SF), 10MK is that specimen produced with 10% metakaolin (MK), 20MK and 30MK were specimens produced with 20% and 30% metakaolin (MK) respectively.

Specimens Preparation

To prepare the specimen, the cementitious materials were dry mixed in a mortar mixer for about one minute at low speed of 10 rpm. Premixed water (about 80% of the mixing water) and superplastizer (in the range of 2.8% to 5.0%) depending on the mix were

added into the mixer and the mixing continued for three minutes at medium speed (140 ± 5 rpm). Fine aggregates with 600µm maximum size and GIW as fibre with 12mm length were then added into the mixer and mixing continued for another four minutes. The remaining mixing water (about 20%) was then added to the mixer and mixed at high speed (285 ± 10 rpm) for additional four minutes. Finally, the mixer was then returned to the medium speed (140 ± 5 rpm) and mix for three minutes. This mixing method was adopted from Hiremath and Yaragal (2017). All the fresh mixes had consistency of 270±5 mm. After mixing, the fresh specimens were cast and kept in moulds for 24 hours in the laboratory condition (27 ± 2°C). Cube moulds of 50mmx50mmx50mm, cylindrical moulds of 50mmx100mm and prismatic moulds of 40mmx40mmx160mm were cast for compressive strength, split-tensile strength and flexural strength tests, respectively. Specimens were then taken out from the moulds and cured in water until the testing ages of 7, 14, 28, 90 and 180 days.

Testing Methods

Flowability

The Flowability of the different mixes was tested using a flow table in accordance with ASTM C143. This was conducted by filling a mini-slump cone. The cone was then carefully removed to allow the mix to flow under the influence of gravity. The flow of the mix was obtained by measuring the spread using a measuring tape. Average of four measurements of the spread (fixed at 270±5 mm) was reported for each mix.

Table 2: Mix proportion of RPC specimens

Specimen ID	20SF	10MK	20MK	30MK	20SF	10MK	20M K	30MK
	Non fibred				Fibred			
Cement	1	1	1	1	1	1	1	1
Silica fume	0.20	0	0	0	0.20	0	0	0
Metakaolin	0	0.10	0.20	0.30	0	0.10	0.20	0.30
Sand (150-600µm)	1.1	1.1	1.1	1.1	1.1	1.1	1.1	1.1
Superplasticizer	3.5	2.8	3.8	4.5	3.6	3.2	3.9	5.0
Gear inner wire (GIW)	-	-	-	-	0.02	0.02	0.02	0.02
Water	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3

Note: Cement Content = 900Kg/m³; Fibre Content= % of weight of concrete, SP content=% of binder

Strength properties

Compressive strength, split-tensile strength and flexural strength tests on the specimens were carried out according to BS EN 12390-3:2002, ASTM C496, ASTM C78 respectively. The average of five measurements was reported for each test.

Results and Discussion

Characterization of Constituent Materials
Tests conducted under characterization include XRD, chemical composition and strength activity of MK, so also the geometry of the Gear Inner Wire.

X-Ray diffraction

Figure 3 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 2 θ value (between 10 -30) also shows hallow in shape. The reduced peak of the calcined kaolin and its hallowed nature indicated that the material used in the experiment has been transformed from crystalline to amorphous form (i.e. Metakaolin). This is similar to the findings of Badogiannis *et al.* (2005) and Wang, Li and Yan (2005).

Chemical Composition

Flowability of RPC

Figure 4 shows the flowing nature of the RPC produced. After series of trials the flow value of the RPC was fixed at 270 \pm 5mm. This value was achieved by the addition of superplasticizer and in accordance with ASTM C143 and ASTM C1611. It was observed that the superplasticizer (sp) dosage for RPC increased (in the range of 2.8% to 5.0%) with the increasing MK content which can be ascribed to its high surface area.

Compressive Strength of RPC

Figure 5(a & b) shows the effect of MK content on the compressive strength of unfibred and fibred RPC at 7, 14, 28, 90 and 180 days respectively. For the two specimens of the RPC, an increase in compressive strength with age can be observed. The compressive strengths obtained at 7, 14, 28, 90 and 180 days for unfibred RPC specimens are higher than the fibred ones. At 7 and 28 days, the compressive strengths of unfibred specimen with 20% MK were comparable to those of the reference (20%SF). At 28 days, the compressive strengths of the unfibred RPC with 10%MK, 20%MK and 30%MK were 90.2%, 97.1% and 92% of the control (66.4 N/mm²) but at 180 days, the compressive strengths of specimens with 10%MK, 20%MK and 30%MK were 93.2%, 96.8% and 87.4% lower of the reference (74.9 N/mm²). It can be observed that the compressive strengths of unfibred specimen with 20% MK at all ages were comparable to those of the reference (20%SF). This result agrees with the findings of Guneyisi *et al.* (2012) and Ding and Li (2002) who observed that MK reacts (in terms of strength) in similar way to SF. Furthermore, MK helps in enhancing the early age and long-term mechanical properties of concrete (Siddique and Klaus, 2009). Hence, 20% seems to be the optimum content of MK in producing unfibred RPC. However, it can be noted from the fibred specimens that the inclusion of the fibre material led to reduction in strength (Jalal, 2012; Meddah & Bencheikh, 2009; Iqbal *et al.*, 2015) and could probably be due to its slippery surface that hindered adequate bond between the fibres and the cement paste. However, results of RPC produced with the 20%MK are superior to what Asteray *et al.* (2017) and Demiss *et al.* (2018) obtained using rice husk ash and fly ash respectively.

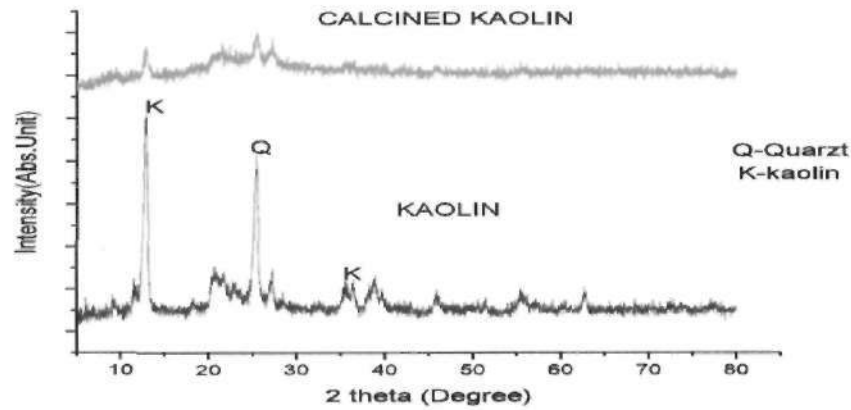


Figure 3: XRD result of Metakaolin

Table 3: 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 ₂ O ₃	2.94	2.768	0.50	2.59
Al ₂ O 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 ₂ O 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 ₂ O ₃ + Fe ₂ O ₃				88.29
LOI	0.84	3.492	3.00	1.80
<i>Physical properties</i>				
Surface area (m ² /kg)			2 0, 000	509.0
Strength activity index (%)			-	87
Specific gravity			2.21	2.53

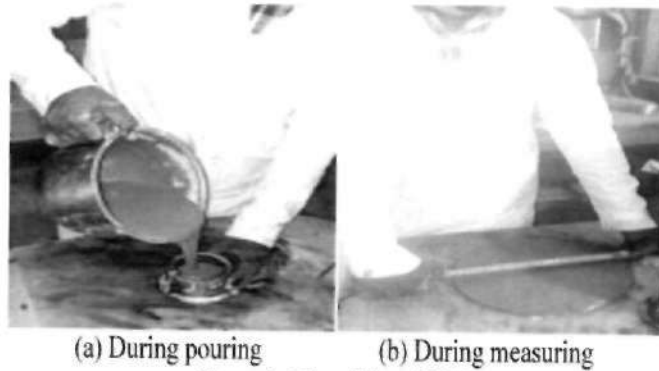


Figure 4: Flowability of RPC

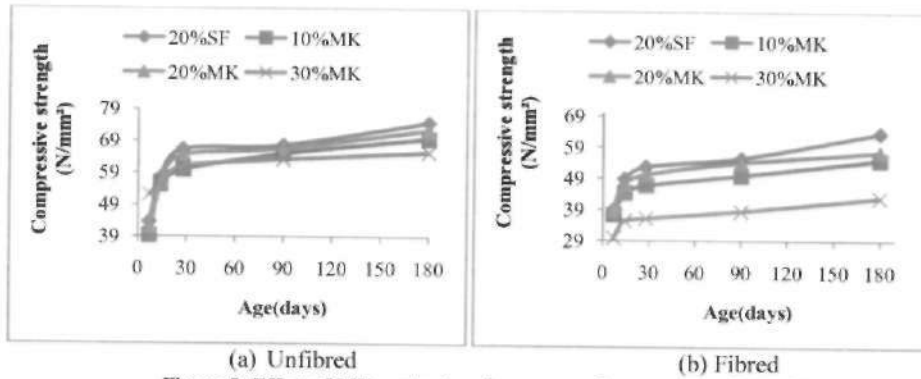


Figure 5: Effect of MK content on the compressive strength of fibred RPC

Tensile Strength

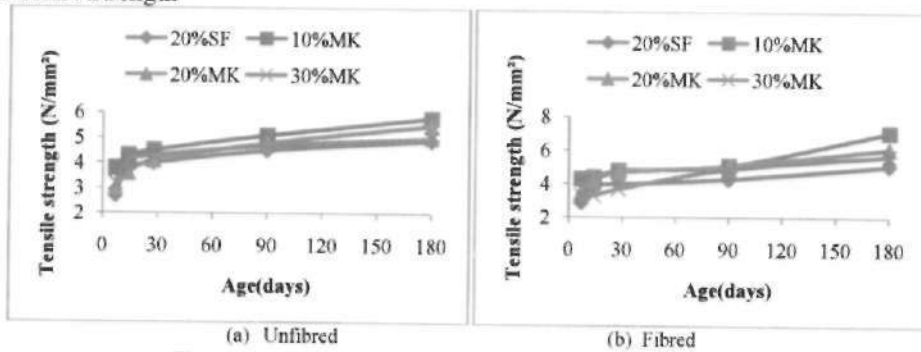


Figure 6: Effect of MK content on the tensile strength of unfibred RPC

Flexural Strength

Figure 6 (a & b) depicts the split tensile strength of unfibred and fibred RPC samples respectively. For the fibred specimens (Figure 6b), it is clear that the inclusion of the fibre (GIW) improves the tensile strengths of the specimens at all ages. Tensile strength improvement reduces with the increasing MK content at all ages. The tensile strengths of the specimens with 20%SF, 10%MK, 20%MK and 30%MK

with 0.25%GIW at 7 days were 2.9 N/mm², 4.3 N/mm², 3.5 N/mm² and 3.3N/mm² while those at 28 days were 4 N/mm², 4.8 N/mm², 4.7 N/mm² and 3.7N/mm² respectively. Moreover, at 90 days, the tensile strength of 10%MK, 20%MK and 30%MK with 0.25%GIW were higher than the reference by 18.6%, 18.6%, and 14% respectively. So also, at 180 days, the tensile strength of 10%MK, 20%MK and 30%MK with 0.25%GIW were higher than the reference

by 39.2%, 19.6% and 11.8%. Hence, up to 30% MK can be used to develop fibred RPC with improved tensile strength. The improvement in the tensile strength of the specimens with MK could be due to the GIW (Ibrahim *et al.*, 2018), filler and pozzolanic effects of MK (Vipat & Kulkarni 2016) that enhance the microstructure of the RPC. As shown in Figure 6a, the tensile strength of unfibred specimens with MK at all ages were also generally higher than that of the control. It is evident from the results that unrefined MK and GIW are suitable in enhancing tensile strength of RPC.

Figure 7 (a & b) demonstrates the flexural strengths of fibred and unfibred RPC. As shown in Figure 7(b), the fibred RPC have higher flexural strengths at all ages compared to the fibred RPC (Figure 7b). For example, at 7 days, the flexural strength of fibred RPC with 20%SF, 10%MK, 20%MK and 30%MK with 0.25%GIW were 14.8, 11.4, 10.44 and 8.6 N/mm² respectively. However, the extent of improvement over curing ages is more pronounced with the specimens made with MK. At 180 days, the 10%MK, 20%MK and 30%MK with 0.25%GIW were higher than the control by 21.9%, 14.2% and 2.7% respectively. In general, 20% MK inclusion with 0.25% GIW outperformed the other specimens at all ages. The improvement in the flexural strength of the specimens with MK could be due to the 0.25% GIW and pozzolanic effects of MK that enhance the microstructure of the RPC. Vipat and

Kulkarni (2016) reported similar findings that MK enhances the properties of concrete through filler and pozzolanic effects. Similar observation was made by Vipat and Kulkarni (2016). Similar findings were also reported by Haroon *et al.* (2017); Demiss *et al.* (2018). Compared to the control, MK improved the flexural strength of unfibred RPC, and the improvement goes along with the increase in MK content. Therefore, unrefined MK and GIW have been found to enhance the flexural strength of RPC.

Conclusion

The unrefined metakaolin and gear inner wires have been found to be suitable in the production of reactive powder concrete (RPC). Unrefined MK of up to 20% by weight of cement can be used to produce RPC with 72.5N/mm² compressive strength and 0.25% GIW by weight of concrete can be used to produce RPC with tensile strength and flexural strength of 7.1N/mm² and 22.3N/mm² respectively. However, GIW has been found to decrease the compressive strength and improves tensile and flexural strengths of the RPC. RPC of this type can easily be produced without necessarily the need for pressure and heat treatment. Moreover, the use of the unrefined MK and GIW can lead to production of cheaper and sustainable RPC by cutting down importation cost of both SF and fibre materials.

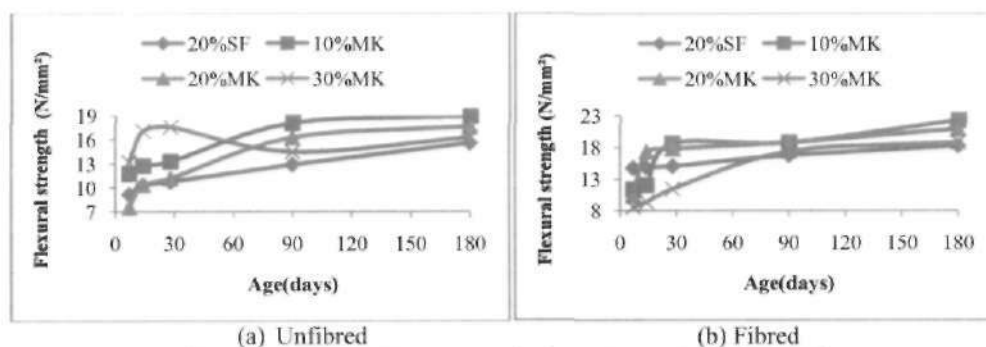


Figure 7: Effect of MK content on the flexural strength of fibred RPC

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