

Full Length Research Paper

Evaluation of mechanical and permeability related properties of self compacting concrete containing metakaolin

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In this paper, mechanical and permeability related properties of Self Compacting Concrete (SCC) containing metakaolin (MK) were evaluated and their interrelationships discussed. For this purpose, different types of mixture were prepared with different amounts of MK and ordinary Portland cement (OPC) was replaced by 5, 10, 15 ... 30% of MK. When increasing the percentage of MK, the mechanical and permeability related properties of SCC considerably improved. The result showed that the SCC sample incorporating the cement with 20% MK showed better workability, mechanical and permeability related properties than that of the normal SCC sample (without MK). One interesting point was observed that the UPV and other mechanical properties were perfectly fitted in the regression analysis. It is used to indicate that the strong interrelationships were presented with each other. In addition, the resistance to chloride ion penetration was directly varied with the $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 / \text{CaO}$ ratio. A good result was obtained for resistance to chloride ion penetration in SCC at the $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 / \text{CaO}$ ratio of 1.1.

Key words: Self compacting concrete, metakaolin, mechanical properties, chloride permeability, interrelationships.

INTRODUCTION

Self compacting concrete (SCC) was first developed in 1988 in Japan due to gradual reduction of skilled labor in construction industry. It can be achieved without segregation and high deformability by three ways that are limiting aggregate content, low water powder ratio and the use of superplasticizer (Hajime and Masahiro, 2003; EFNARC, 2002; Yahia et al., 1999). Nowadays, SCC gains much popularity throughout the world' because of its some interesting structural properties. However, it is not completely accepted due to higher cost, the lack of standard specifications and testing procedures. It is only treated as special concrete (Hemant et al., 2009). The reason for the increase of the cost of SCC production is

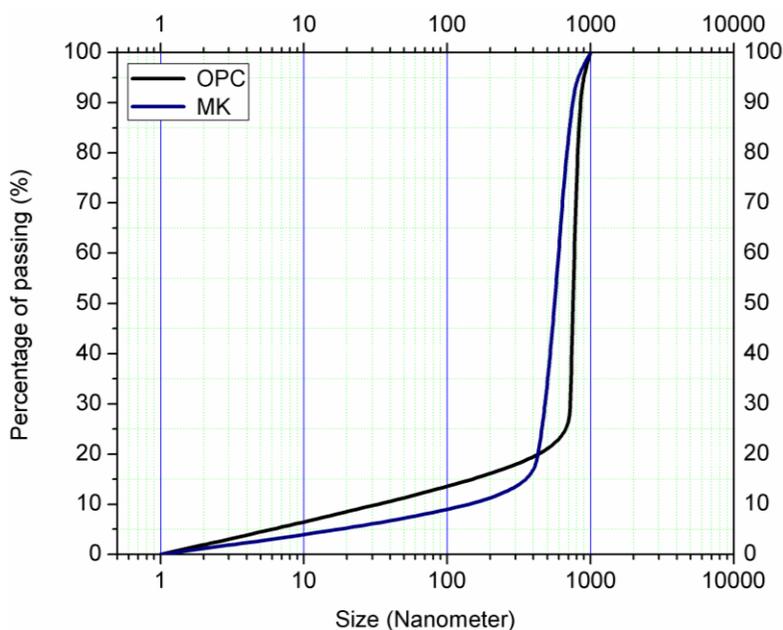
use of higher powder content (cement) and it can also be reduced by use of various mineral admixtures such as rice husk ash, fly ash, metakaolin etc., as partial replacement of cement. The mineral admixtures also improve the structural properties of the SCC (Nor and Hanizam, 2011). It is generally recognized that the incorporation of pozzolanic materials as a partial replacement to OPC in SCC is an effective means for improving the fresh state and harden state properties. This is due to the fact that calcium hydroxide $[\text{Ca}(\text{OH})_2]$ produced by cement hydration reacts with pozzolanic materials like rice husk ash (RHA), metakaolin (MK), fly ash (FA), silica fume etc. and produces additional calcium silicate hydrate (C-S-H) gel. The formation of that gel also can improve the strength and durability of concrete through altering the pore structures of concrete (Luiz et al., 2006).

Metakaolin (MK) is a valuable pozzolanic, and

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Table 1. Sieve analysis and physical properties of fine and coarse aggregate.

Sieve size (mm)	Fine aggregate (% of passing)	Coarse aggregate (% of passing)
20	100	100
12.5	100	90.1
10	100	10.4
4.75	99.9	0.00
2.36	99.1	0.00
1.18	83.1	0.00
0.60	58.3	0.00
0.30	10.0	0.00
0.15	0.70	0.00
Pan	0.00	0.00
Bulk density(kg/m ³)	1752	1640
Specific Gravity (g/m ³)	2.53	2.78
Water absorption (%)	2.01	0.36

**Figure 1.** Particle size distribution of binders.

thermally activated aluminosilicate material obtained by calcining kaolin clay within the temperature range of 700 to 850°C (Sabir et al., 1996; Ambroise et al., 1994). MK is usually added to concrete in amount of 5 to 15% by weight of cement. Using of MK in SCC can increase the mechanical and permeability related properties (Aziz et al., 2005). But when replacing OPC by MK at higher percentage may lead to reduce the workability of fresh concrete mix. This disadvantage can be eliminated by using superplasticizers (SP) or increasing W/B ratio.

In this study, the effect of MK on mechanical and permeability related properties of SCC were experimentally evaluated. Also, their interrelationships were discussed. Furthermore, the relationship between

$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ / CaO ratio and the resistance of chloride ion penetration (total current passed in coulombs) on SCC were evaluated.

MATERIALS AND METHODS

Ordinary Portland cement (OPC) conforming to Indian standard code IS 8112-1995 was used. The fine aggregate was natural river sand. The sieve analysis of fine aggregate and coarse aggregate was carried out in accordance with IS 383:1970 code provision. The results of sieve analysis and physical properties of fine and coarse aggregates were tabulated in Table 1. The particle size distributions of binders are shown in Figure 1. Superplasticizers (SP) was used to increase the workability of SCC. For this study, Conplast SP 430

Table 2. Mix proportions of MK blended SCC.

Mix designation	MK	W/C or W/(C+MK)	Quantities (kg/m ³)					
			Water	OPC	MK	SP (2%)	Fine aggregate	Coarse aggregate
SCC-MK0	0	0.55	220	400	0	8	880	800
SCC-MK05	5	0.55	220	380	20	8	880	800
SCC-MK10	10	0.55	220	360	40	8	880	800
SCC-MK15	15	0.55	220	340	60	8	880	800
SCC-MK20	20	0.55	220	320	80	8	880	800
SCC-MK25	25	0.55	220	300	100	8	880	800
SCC-MK30	30	0.55	220	280	120	8	880	800

Table 3. Slump flow, viscosity and passing ability classes with respect to EFNARC.

Slump flow classes (SF)	Slump flow (mm)	Viscosity classes	V-funnel time (s)	Passing ability classes	Passing ability, H ₂ /H ₁ ratio
SF1	550 - 650	VS1	≤ 8	PA1	≥ 0.8 with two rebar
SF2	660 - 750	VS2	9- 25	PA2	≥ 0.8 with three rebar
SF3	760 - 850	-	-	-	-

was used as superplasticiser (conforming to IS: 9103:1999) to improve the performance of SCC. Commercially available MK was used. Mineralogical analysis of MK was carried out by X-ray diffraction analysis. The physical and chemical analysis of OPC and MK was carried out as per IS 4032-1985 and IS 1727-1995 codes.

Mix proportions and casting of SCC specimens

The mix proportion was carried out according to number of trail mixes as well as EFNARC guidelines to produce SCC without segregation and bleeding. In the trail mixes, it can be noted that lower W/C ratio is affected by the workability properties of the SCC with MK and locally available aggregates. For this study, totally seven SCC mixes (MK ranging from 0, 5, 10 30%) were prepared with a water to binder W/(C + MK) ratio of 0.55 and 2% of SP. These mixes were designated as SCC (100% OPC) and SCC-MK05, SCC-MK10, SCC-MK15..... SCC-MK30. The mix proportions are presented in Table 2. For mix preparation, the procedure established by Khayat et al. (2000) was employed to produce SCC with MK. In this way, natural coarse and fine aggregates were homogenized for 30 s at normal mixing speed. Thereafter, adding about half of the mixing water into the mixer while mixing goes on for 1 min. The mixture was rested for 1 min so that the aggregates could absorb the water in the mixer. Then, cement and MK were added and mixed for one more minute. The remaining water and SP were introduced to the wet mixture, while mixing was going on for 3 min. Finally, after 2 min resting, mixing sequence resumed for additional 2 min. This optimum time is necessary to disperse SP and stabilize viscosity. For all mixtures, nine cube specimens of 100 mm size were cast for compressive strength testing, and before compressive strength test, all cube specimens were used for ultrasonic pulse velocity test.

Nine cylindrical specimens of 150 mm diameter and 300 mm height were also cast for determining the splitting tensile strength. Nine cylindrical specimens of 100 mm diameter and 50 mm height were cast for permeability related properties tests. After casting, all the specimens were left in the casting room for 24 h. After 24 h, the

specimens were demoulded and immersed in water curing tank up to the time of testing.

Fresh concrete tests

In the present study, the slump flow, V-funnel times and L-box tests were carried out according to the procedure recommended by EFNARC guidelines. Slump flow test has been proposed to assess filling ability of concrete in the absence of obstructions. According to EFNARC guidelines, there are typically three slump flow classes for a range of applications which are given in Table 3. Slump flow is not a suitable factor to exactly exhibit the fresh characteristic of SCC. But, if the slump flow is kept within a desirable range, it is possible to evaluate the requirements of SCC (Sonebi et al., 2007). SCC containing MK with slump flow values between 660 and 750 mm were proposed in the present study. Viscosity can be assessed by the V-funnel times. On the basis of EFNARC Guidelines, there are two viscosity classes which determined by V-funnel and T50 flow times. In this study, V-funnel times were carried out and the viscosity classes were presented in Table 3. The L-box test is utilized to determine passing ability of SCC when flowing through confined or reinforced areas. The passing ability classifications according to EFNARC were also presented in Table 3.

Harden concrete tests

The compressive and splitting tensile strength was carried out according to IS 9013-1997 and 5816-1999 at 7, 28 and 90 days. Ultrasonic pulse velocity test and dynamic modulus of elasticity tests were performed at 28 days of water curing as per IS 13311 (part 1)-1992 code and the Poisson's ratio was taken as 0.24 ($\nu = 0.24$ for normal strength of concrete). All the permeability related properties tests were performed after 28 days of water curing. Saturated water absorption values of MK blended SCC specimens were measured as per ASTM C 642. Coefficient of water absorption is suggested as a measure of permeability of water. This is

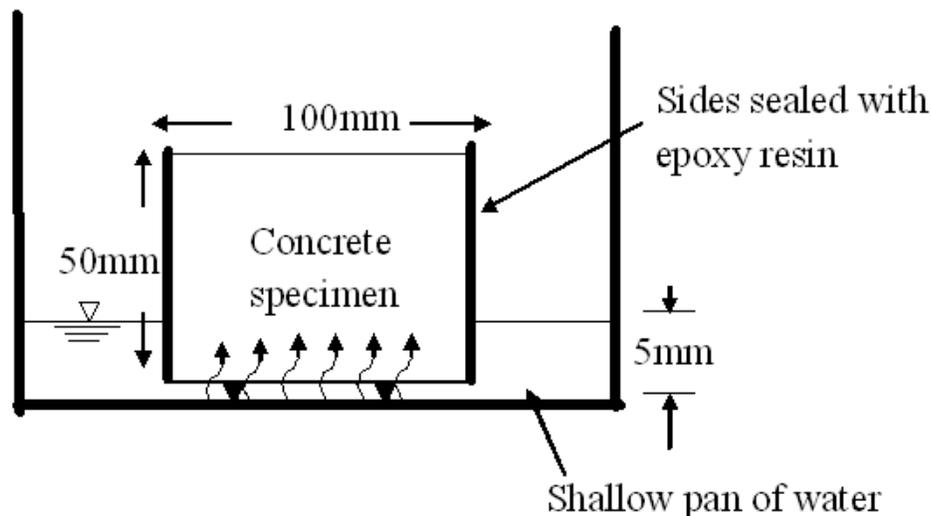


Figure 2. Coefficient of water absorption and sorptivity test setup.

Table 4. Physical properties of OPC and MK.

Material	Bulk density (kg/m ³)		Specific gravity	Fineness passing 45 μ sieve	Specific surface area Blain's (m ² /kg)	Mean grain size (μ m)
	Loose	Dense				
OPC	1.18	1.27	3.13	86	318	23.4
MK	0.50	0.52	2.58	99	2350	3.79

measured by the rate of up take of water by dry concrete in a period of 1 h. The SCC specimens were preconditioned by drying the specimens in an oven at 105°C for 7 days until constant weight was reached and then allowed to cool in a sealed container for 3 days. The sides of the specimen were sealed with transparent epoxy coating so as the water to penetrate the circular cross section. Then, the samples were dipped in the water in a vertical position with one open surface in partially immersed to depth of 5 mm and the other with the laboratory air as shown in Figure 2.

The quantity of water absorbed during the first 60 min was calculated. Coefficient of water absorption values of MK blended SCC specimens after 28 days of water curing were determined by the following expression:

$$K_a = [Q/A]^2 \times [1/t]$$

Where K_a is coefficient of water absorption (m² / s), Q is quantity of water absorbed (m³) by the oven dry specimen in time (t), t is 3600 s and A is total surface area (m²) of SCC specimen through which water penetrates.

Three specimens were used for sorptivity measurement. Measurement of capillary sorption were carried out using specimens pre-conditioned in the hot air oven at about 50°C until constant weight was obtained. Then, the concrete specimens were cooled down to room temperature. As shown in Figure 2, the test specimens were exposed to the water on one face by placing them on a pan. The side face of the specimen was coated with epoxy resin. The water level in the pan was maintained at about 5 mm above the base of the specimen during the experiment. At the suitable time intervals, the specimen was removed from the water, excess water removed by damp paper towel and then the specimen was weighed. It was then replaced in the water and the stopwatch

started again. The gain in mass per unit area over the density of water is plotted versus the square root of the elapsed time. The slope of the line of best fit of these points was taken as the sorptivity value. The sorptivity values of MK blended SCC specimens were evaluated by the following expression:

$$i = S t^{1/2}$$

Where i is the cumulative water absorption per unit area of inflow surface (m³/m²), S is the sorptivity (m/s^{1/2}) and t is the time elapsed(s).

The resistance to chloride ion penetration in terms of total charge passed in coulombs of MK blended SCC specimens were measured as per ASTM C 1202 standard.

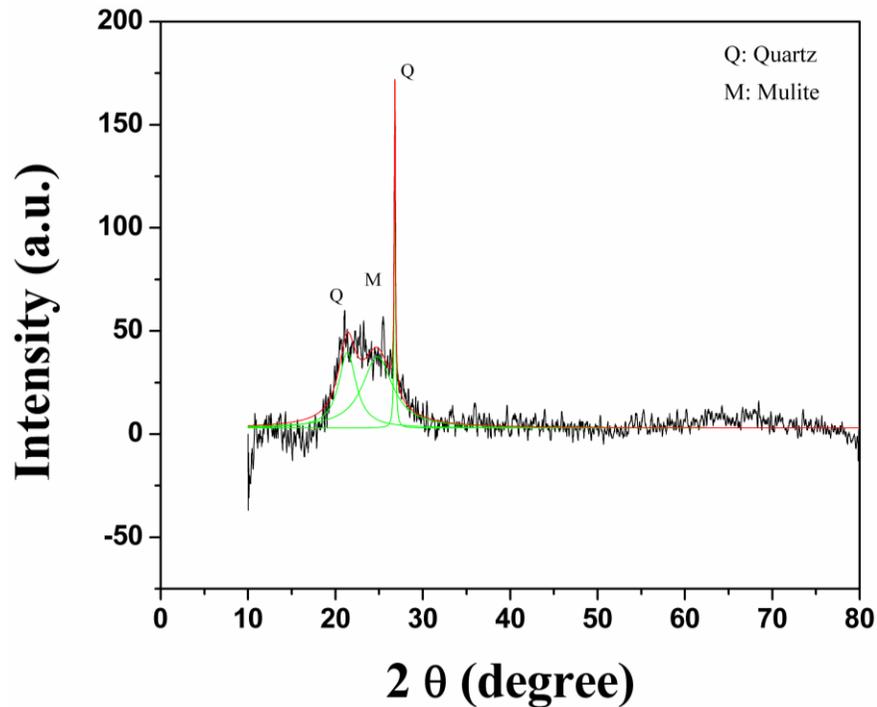
RESULTS AND DISCUSSION

Physical and chemical analyses of OPC and MK

The physical properties of OPC and MK are compared in Table 4. The average specific surface area of MK is 2350 m²/kg and that of OPC is 318 m²/kg. The density, specific gravity and mean grain size of MK are found to be less than those of OPC. The high surface area and fineness of the MK may be used to improve the strength and permeability properties of SCC and change the pore structure of the concrete (Isaia et al., 2003). Chemical composition data for OPC and MK are compared in Table 5. This particular MK consists 51.80% of silica and

Table 5. Chemical composition of OPC and MK (%).

Material	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	Loss on ignition
OPC	20.25	5.04	3.16	63.31	4.20	0.08	0.51	3.08
MK	51.80	43.75	0.82	0.09	0.03	0.07	0.02	0.34

**Figure 3.** X-ray diffraction (XRD) pattern of MK

43.75% of alumina. The loss of ignition value for MK is 0.34, which is less than that of OPC. The XRD pattern of MK used in this study is shown in Figure 3. From other references (Jutice and Kurtis, 2007; Poon et al., 2001), the MK was mainly in amorphous silica form but its slightly deviated from amorphous forms to crystalline form at the angle of 26.8092° and showing high amount of quartz phase in this pozzolan.

Fresh state properties of SCC

The fresh characterizations of SCC containing MK were studied and the results are shown in Figure 4. The slump flow values for different concrete mixes were measured in the range of 660 to 750 mm. According to EFNARC (Table 3), all concrete mixtures under investigation can be categorized as slump flow class 2 (SF2). The concrete mixture at this class of slump flow is suitable for many normal applications such as walls and columns. The flowability of the mixtures was reduced with the higher proportion of MK replacement, as shown in Figure 4. For

instance, at a given SP dosage with W/B ratio of 0.55, the slump flow of SCC-M20 was measured to be 678 mm while this could be decreased to 656 mm when MK introduced to 25%. This could be explained by the higher surface area of the MK particles compared to Portland cement. The V-funnel times of different concrete groups are shown in Figure 4. From this figure, it can be seen that the V-funnel times for all mixes were in the range of 3.9 to 7.9 s regarding the EFNARC guidelines, a V-funnel flow time higher than 25 s did not recommend (Table 3). As shown in Figure 4, the V funnel flow times of all mixtures satisfy this requirement. The results presented in Table 6 also indicated that, the V-funnel time shows a distinct tendency to increase with increasing MK content. The results from the L-box test are also shown in Figure 4. The L-box blocking ratio of SCC containing MK varied from 0.94 to struck stage.

From Figure 4, SCC mixtures containing MK shows satisfactory blocking ratio. Although, in some mixtures, blocking ratio was found to be out of the EFNARC recommendation (Table 3); but it should be noted that Felekoglu et al. (2007) concluded that the blocking ratio

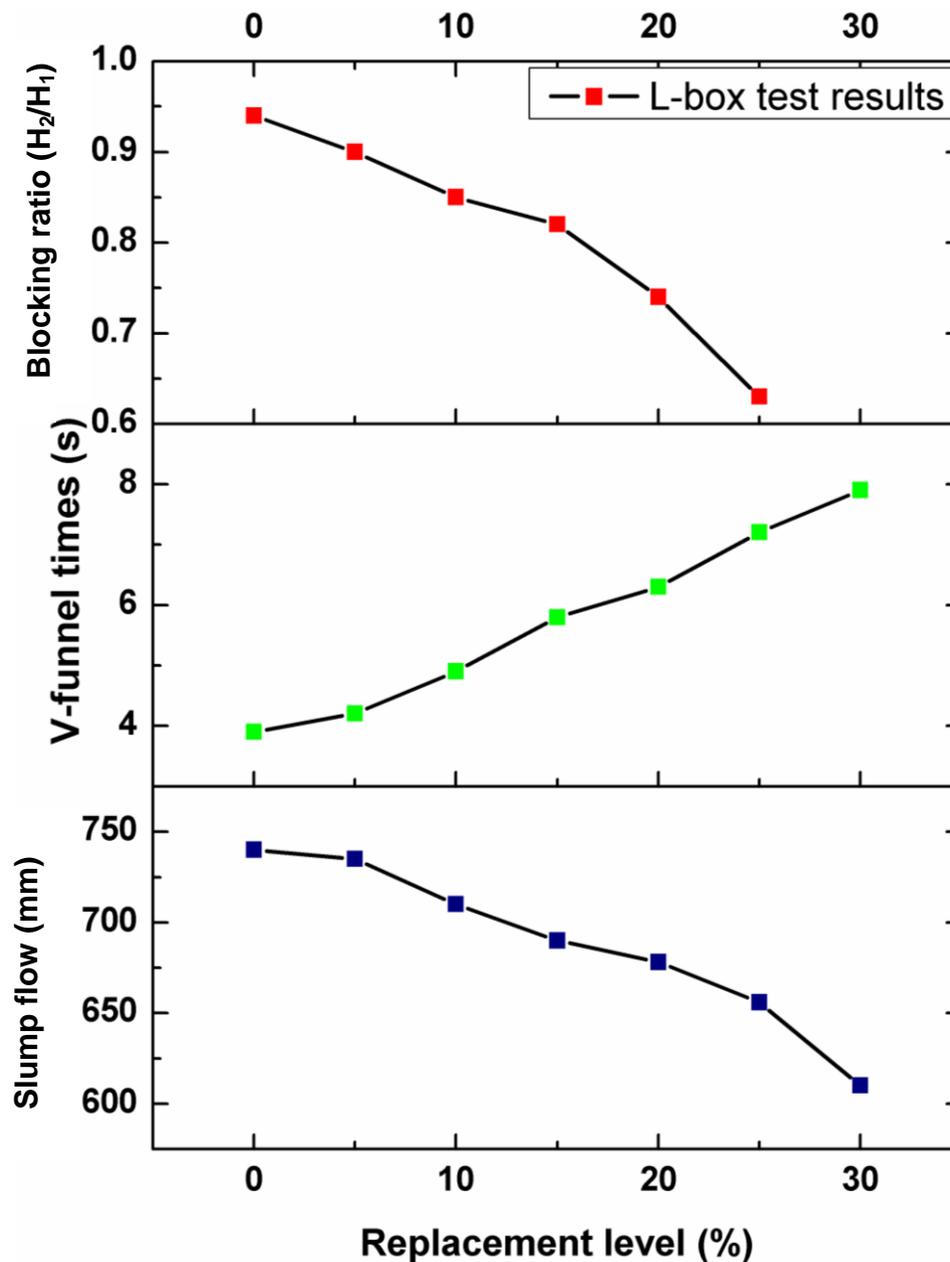


Figure 4. Fresh state properties of MK blended SCC.

Table 6. Regression between other mechanical properties (y) and UPV (x) of MK blended SCC.

Fit		Compressive strength (N/mm^2)	Splitting tensile strength (N/mm^2)	DME (N/mm^2)
Linear ($y = ax + b$)	a	0.020	0.00134	20.576
	b	-48.116	-3.54764	-5.113×10^4
	R^2	0.8616	0.8706	0.9978
Exponential ($y = a - bc^x$)	a	57.494	112.082	4.290×10^5
	b	2.72×10^7	115.825	4.943×10^5
	c	0.996	0.999	0.999
	R^2	0.9254	0.8382	0.9970

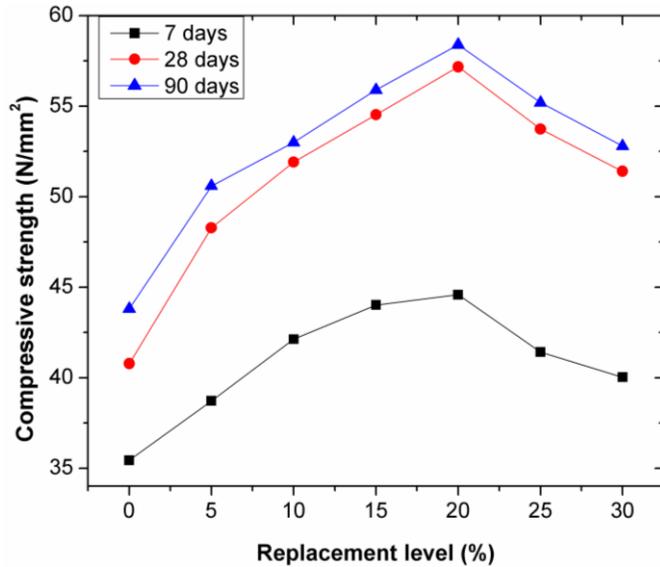


Figure 5. Compressive strength of MK blended SCC.

higher than 0.6 has been accepted for SCC to achieve good filling ability. An overview on the fresh properties of SCC containing MK at constant W/B ratio (0.55) reveals that MK replacement up to 15 to 20%, generally satisfy the fresh-state behavior requirements related to high segregation resistance, deformability, passing and filling abilities.

Mechanical properties of SCC

The compressive strength of MK blended SCC specimen are shown in Figure 5. From the results, SCC with MK shows higher compressive strength than normal SCC (SCC with 0% replacement of cement). Among different MK replacement level, the most remarkable strength development was found to attain for MK replacement at the levels of 15 to 20%. The compressive strength of SCC decreases when the amount of cement replacement by MK is higher than 20%. It may be due to the fact of decrement of workability and CaO/SiO₂ ratio. Figure 6 shows the effect of MK contents on tensile strength. It can be seen that the tensile strength of mixtures increased with increasing of replacement level up to 20%. The maximum increase of tensile strength at 28 days was about 47.05% for SCC-MK20 mix. However, the companion compressive strength improvement at 28 days was 40.22%. This shows that the increase of tensile strength was higher than corresponding compressive strength. In a similar trend to that observed in compressive strength, SCC mixtures containing 15 to 20% MK provided better performance in terms of splitting tensile strength. The modulus of elasticity from the data obtained by non destructive testing of concrete by means of dynamic modulus of elasticity (DME) is somewhat

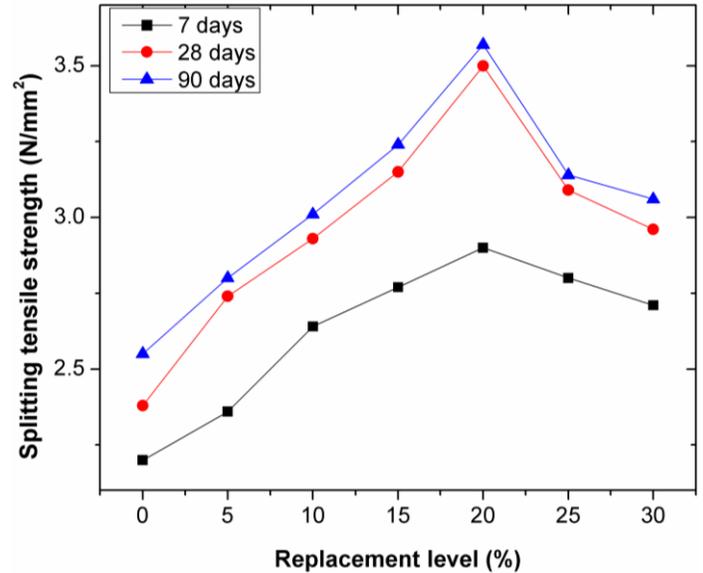


Figure 6. Splitting tensile strength of MK blended SCC.

more important than those determined by static method. This is because the modulus of elasticity as determined by dynamic modulus is unaffected by creep (Akçay, 2004; Shetty, 2002). The UPV is one of the most important non-destructive methods used in the pre determination of concrete properties. UPV values calculated for MK blended SCC specimens is shown in Figure 7. It was observed that in comparison with Normal SCC (100% OPC), the UPV values increases due to the effect of MK. According to IS13311(part 1)-1992 code, the concrete are categorized as “excellent”, “good”, “doubtful”, “poor” and “very poor” for the UPV values of 4500 m/s and above, 3500 to 4500, 3000 to 3500, 2000 to 3000 and 2000 m/s; and below, respectively. Accordingly, all concrete mixtures were found to have good and excellent quality.

Modulus of elasticity of concrete is frequently expressed as a function of compressive strength and it is strongly linked to the coarse aggregate fraction and properties. The elastic properties of concrete are known to be influenced by elastic properties of the constituent materials and nature of the interfacial zone between aggregates and paste. The dynamic modulus of elasticity (DME) values calculated for MK blended SCC's specimens are also shown in Figure 7. It can be seen that the DME increased with increasing percentage of MK up to 20%.

Permeability related properties

The permeability related properties are shown in Figure 8 for MK blended SCC mixes. From the results, water absorption (WA) property of MK blended SCC specimens

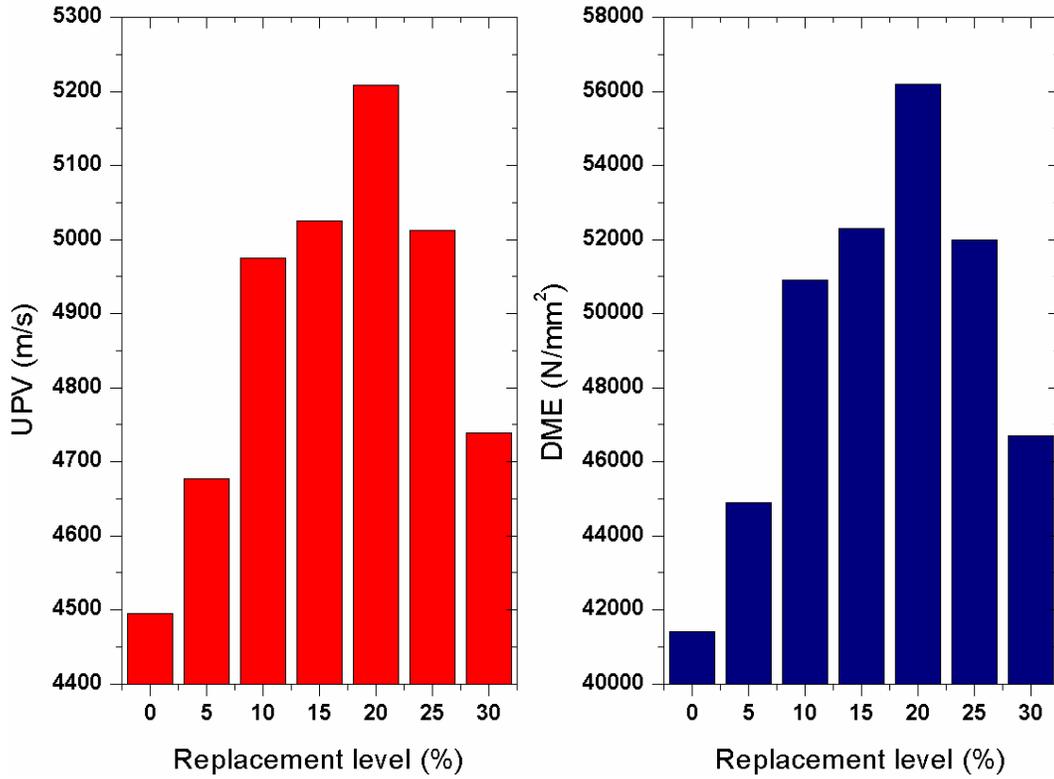


Figure 7. UPV and DME of MK blended SCC.

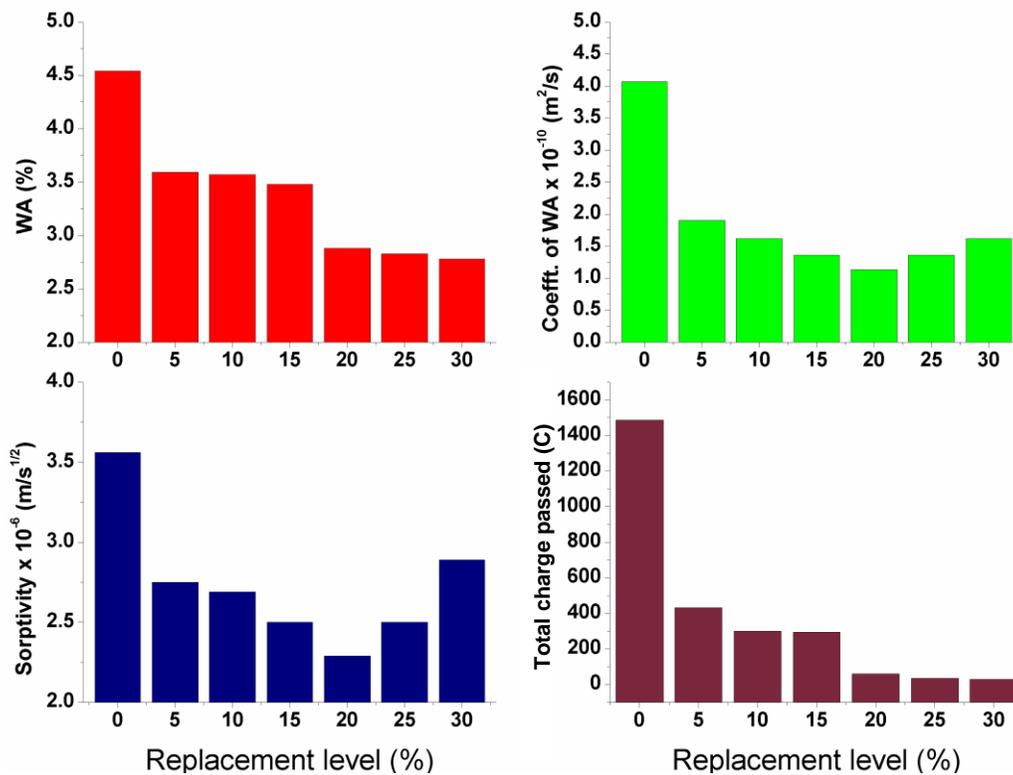


Figure 8. Permeability related properties of MK blended SCC.

Table 7. Regression between other permeability related properties (y) and water absorption (x) of MK blended SCC.

Fit		Coefficient of water absorption (m ² /s)	Sorptivity (m/s ^{1/2})	Chloride penetration (coulombs)
Linear (y = ax + b)	a	1.387x10 ⁻¹⁰	5.053x10 ⁻⁷	779.792
	b	-2.823x10 ⁻¹⁰	1.031x10 ⁻⁶	-2260.747
	R ²	0.7009	0.5089	0.8804
Exponential (y = a - bc ^x)	a	1.021x10 ⁻⁷	3.501x10 ⁻⁴	3.093 x10 ⁵
	b	1.023x10 ⁻⁷	3.491 x10 ⁻⁴	3.116x10 ⁵
	c	0.998	0.998	0.997
	R ²	0.6256	0.3857	0.8498

after 28 of curing are improved with MK content up to 30%. This is due to the fact that MK is finer than OPC and altering the pore structure of concrete. The coefficient of water absorption properties of MK blended SCC is progressively decrease with increase in MK content up to 20%. At 25 and 30% MK, there is an increase in coefficient of water absorption and these values are also lower than that of normal SCC (0% of MK) specimens. The coefficient of water absorption values up to 30% are quite lower confirming that the addition of MK leads to a reduction of permeable voids. The sorptivity progressively decreases with improvement in MK content up to 20%. At 25 and 30% MK, there is an increase in sorptivity and these values are also lower than that of control concrete. This again confirms that MK addition leads to a reduction in pore space. It is also observed from the sorptivity data that 30% MK SCC specimens have shown a 35.67% reduction in sorptivity at 28 days compared to that of SCC without MK.

The total coulombs charge passing through MK blended SCC specimens continuously decrease with increase in MK content up to 30%. Particularly, the total charge passed for 30% MK blended SCC is considerably reduced (more than 90% reduction) at 28 days cured SCC (Okan et al., 2012). Since the total charge passed through the concrete depends on the electrical conductance, the lower loss on ignition value present in MK might have contributed to the significant reduction in the electrical charge passed.

Regression between ultrasonic pulse velocity and other mechanical properties

Evaluation of interrelationship between concrete properties is the important tool to ascertain the concrete behavior through known values. The UPV, as an indicator of structural behavior of concrete choose linear and exponential regression analysis. The linear and exponential regression between UPV and other mechanical properties of MK blended SCC is presented in Table 6. A positive relationship between UPV and other mechanical properties (R² > 90%) has been

confirmed. It is also observed that these mechanical properties properly be correlated with UPV, and it also implies the strong inter relationship.

Regression between water absorption and other permeability related parameters

The interrelationship of permeability related properties is an important tool in correlating mix formulations with long term durability parameters. Water absorption property, as an indicator of transport properties was chosen and the inter relationship was determined with linear and exponential regression. The correlation results of MK blended SCC mixes are summarized in Table 7. It shows that a good correlation (R² > 90% with chloride penetration and R² > 50% with sorptivity) was obtained between water absorption and other permeability related properties. It all implies the beneficial use of MK in SCC as partial cement replacement material.

Relationship between SiO₂ + Al₂O₃ + Fe₂O₃ / CaO ratio and total charge passed

To ascertain the type and nature of interdependence among the total charge passed (chloride penetration) and (SiO₂ + Al₂O₃ + Fe₂O₃) / CaO ratio, the total charge passed is linearly correlated with (SiO₂ + Al₂O₃ + Fe₂O₃) / CaO ratio. Figure 9 shows the relationship between chemical composition and total charge passed derived from RCPT of MK blended SCC. The result showed that this relationship was not perfectly correlated with each other (correlation value, R² = 49.9 %), but in the correlation analysis, the relationship is perfectly fitted (R² = 82.46 %) only when the normal SCC (100% OPC) mix is not considered. This correlation improvement clearly indicated that the MK acted as a highly reactive pozzolanic material in SCC.

Conclusion

Based on the experimental studies presented in this

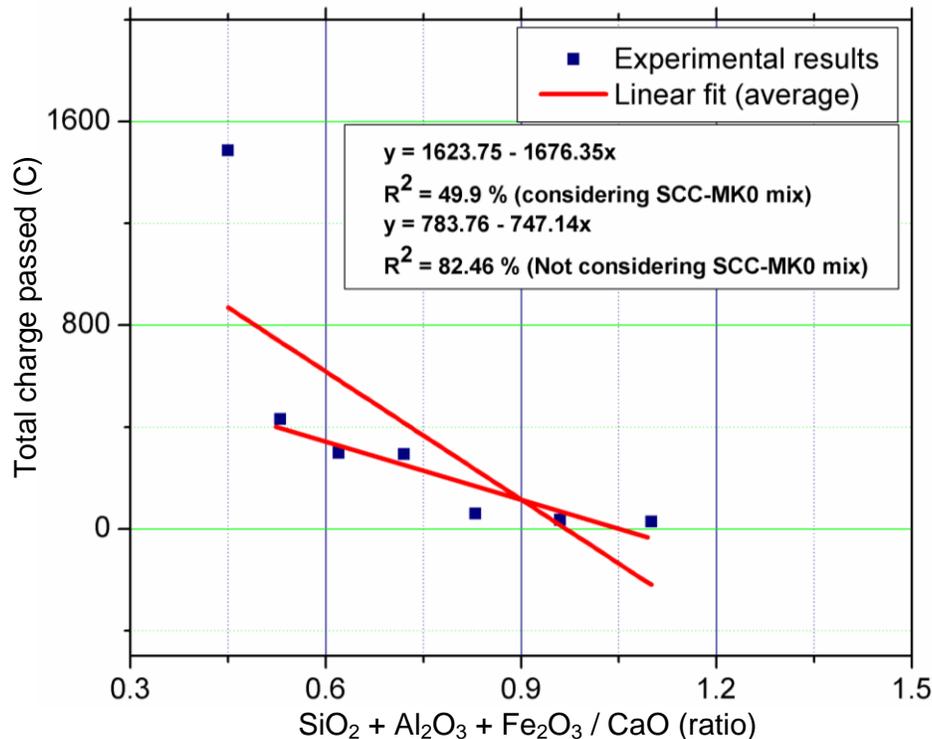


Figure 9. Linear relationship between $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 / \text{CaO}$ ratio and total charge passed (resistance to chloride penetration).

paper, the following conclusions may be drawn:

- 1) Metakaolin may be used as an efficient pozzolanic material with amorphous silica content (51.8%) and a relatively very low loss of ignition value (0.34).
- 2) As high as 20% by weight of OPC can be replaced with MK without any adverse effect on mechanical and permeability related properties.
- 3) All the mixtures had satisfactory self-compacting properties in the fresh state except SCC-MK30 mix.
- 4) Replacement with 30% of MK leads to substantial improvement in strength and permeability properties of blended SCC when compared to that of unblended SCC (0% MK), namely:
 - a) About 26.07% increased in compressive strength.
 - b) About 5.45% increased in ultrasonic pulse velocity.
 - c) About 12.80% increased in dynamic modulus of elasticity.
 - d) About 38.76% decreased in water permeability.
 - e) About 98.10% decreased in chloride permeability, but the workability was not favorable when the replacement level exceeds 20%.
- 5) From the regression analysis, the strong interrelationships of MK blended SCC mixes as follows:
 - a) UPV and compressive strength ($R^2 > 90\%$)
 - b) UPV and splitting tensile strength ($R^2 > 85\%$)
 - c) UPV and DME ($R^2 > 99\%$)
 - d) Water absorption and chloride penetration ($R^2 > 85\%$).

e) The $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 / \text{CaO}$ ratio and total charge passed (chloride penetration) of MK blended SCC performance is inferior ($R^2 = 49.9\%$), but in the correlation analysis, the relationship is perfectly fitted ($R^2 = 82.46\%$) only when the normal SCC (100% OPC) mix is not considered. This correlation improvement clearly indicated that the MK acted as a highly reactive pozzolanic material in SCC.

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