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Effects of incorporating Metakaolin to Evaluate Durability and Mechanical Properties of Concrete

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Abstract :Concrete is known to be the most used construction material worldwide. The environmental and economic aspects of Ordinary Portland Cement (OPC) containing concrete, have led research studies to investigate the possibility of incorporating Supplementary Cementitious Materials (SCMs) in concrete. Metakaolin (MK) is one SCM with high pozzolanic reactivity generated throughout the thermal activation of high purity kaolinite clay at a temperature ranging from 500° C to 800° C. Although many studies have evaluated the effect of MK on mechanical properties of concrete and have reported positive effects, limited articles are considering the effect of MK on durability properties of concrete. Considering the lifetime assessment of concrete structures, the durability of concrete has become of particular interest recently. In the present work, the influences of MK on mechanical and durability properties of concrete mixtures are evaluated. Various experiments such as slump flow test, compressive strength, water permeability, freeze and thaw cycles, rapid chloride penetration and surface resistivity tests were carried out to determine mechanical and durability properties of concretes. Concretes made with the incorporation of MK revealed better mechanical and durability properties compared to control concretes due to combined pozzolanic reactivity and filler effect of MK.

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Introduction

Portland cement is traditionally known to be the most used binder material in concrete. The incremental trend of concrete usage has led to a large volume of Portland cement production annually around the world. Over 4 billion tons of Portland cement was produced in 2015 around the world from which up to 4 billion tons of CO_2 gas was emitted to the air due to the known fact that production of 1 ton Portland cement would result in 0.9 to 1 ton of CO_2 emissions. In addition, the cement industry is one of the primary consumers of natural resources. The usage of SCMs in concrete has increased over the past decade due to the positive effects of pozzolanic reactions. Consumption of calcium hydroxide or CH (An undesirable result of hydration of cement) and taking part in further C-S-H gel production, enhanced resistance against cracking due to denser microstructure. This is because of remarkably lower heat released through the cement hydration process and better mechanical properties due to lower permeability as a result of further C-S-H gel formation. Fly ash, ground granulated blast furnace slag, rice husk ash, and MK are some of the most common SCMs incorporated in concrete manufacture yet [1].

MK is a cement replacement material, formed when [1]pure kaolinite is calcined at high temperatures from 500° C to 800° C. Thermal activation of mineral clay leads to the crystalline structure breakdown (by dehydroxylation) the aforementioned process leads to an aluminosilicate non-crystalline part creation. MK ($Al_2O_3.2SiO_2$) or AS2, is formed by such process [2]. The dehydroxylation breakdown of kaolinite clay, obtained by thermal adjustment or mechanical process in grinding [3]. The level of CH in the concrete paste is reported to decrease significantly by the addition of

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MK. However, the effectiveness of MK mostly depends on the degree of reactivity of such material which in turns depends on thermal activation temperature, and purity of kaolinite used [4]. Today, MK is successfully being used in various types of concrete, such as ultra-high performance concrete, high-performance concrete, self-compacting concrete and fiber reinforced concrete [1].

The use of MK has yielded technical, environmental and economic benefits to the cement and concrete industry. Considering the abundant kaolin mines in Iran, this study investigates the performance of concrete mixtures containing an MK in terms of slump flow test, compressive strength, water penetration, freeze and thaw cycles, rapid chloride permeability test (RCPT) and the surface resistivity at 7, 28, 90 and 180 days.

Literature Review

MK addition to cement mixtures is reported to improve the mechanical properities of concrete. Brooks and Johari [5] reported that the compressive strength of mixture increased with the increase in the MK content. Similar results were also reported by Li and Ding where concrete achieved the highest compressive strength with 10% MK content [6]. In another study, Radonjanin et al. [7] studied the influence of the MK incorporation on compressive strength of mortar. Portland cement was replaced by MK at 10, 20 and 30% levels of replacement (by weight). According to the compressive strength test results, ambient-cured MK mortars containing 10% and 20% MK, at 28 days, yielded 5–6% higher strengths when compared to control mixture. The authors concluded that slight increase in strength of mortars containing MK was due to greater level of agglomertion, occured when MK was being activated thermally. Rezaifar et al. [8] investigated the combined use of crumb rubber and MK in concrete. Crumb rubber was incorporated as aggregate replacement material, while MK was used to replace cement. The authors also used Response Surface Method (RSM) in order to optimize the mix designs. Even though the strength of concrete decreased by the incorporation of crumb rubber, MK notably lowered the compressive strength loss by pozzolanic reactions. Furthermore, the results of water absorption test revealed that use of MK was very effective in decreasing the water absorption. The authors found that the simultaneous usage of crumb rubber by 3.3 vol.% replacement of aggregate and MK by 19.5 vol.% replacement of cement presented the optimized results as the compressive strength was highest and the water absorption was minimized.

The effect of MK addition on durability properties of concrete is also investigated by a number of research studies. Water absorption of mortars containing MK was investigated by Haining et al. [9]. The authors have reported that water absorption of mixture was decreased by the addition of MK, especially at early ages, since the packing density of mixture was optimized because of the extremely fine MK particles. It was also noted that the water absorption of specimens was reduced by increasing the dosage of incorporation to 10% of OPC weight. In addition, the primary products of cement hydration in mixtures were found to be Portlandite and stringier while MK addition decreased the calcium hydroxide content due to the pozzolanic reaction.

The durability of MK Self-Compacting Concrete (SCC) was investigated by Badogiannis et al. [10]. MK was used to replace cement or limestone powder at various levels. A positive influence on the open porosity was observed for both replacement styles, irrespective to the level of replacement. Also, according to the chloride ion penetration test results, replacement of either cement or limestone powder by MK resulted in a reduction of the non-steady state chloride migration coefficient. Gruber et al. [11] reported that the use of 8% and 12% high-reactivity MK (HRM) significantly lowered the chloride ion diffusion coefficient of concrete. Also in another study, Parande et al. [12] deduced that up to 15% replacement of MK in OPC concrete showed to be excellent corrosion resistance property, water absorption and resistivity of concrete.

Experimental Program

Material

ASTM C150 type I Portland cement was used for all of the concrete mixtures [13]. Chemical and physical characteristics of cement are shown in Table 1. The C_3S, C_2S, C_3A and C_4AF contents of the cement by Bogue calculations were 54.6%, 20%, 5.1% and 9.06%, respectively.

To provide high-grade supplementary cementitious materials in terms of pozzolanic activity, the burning conditions such as temperature and the burning time were controlled. A furnace built to burn raw kaolinite clay in Concrete Technology and Durability Research Center at Amirkabir University is shown in Figure 1 [14]. The burning temperature, the oxygen rate, and the burning time are relevant parameters in the production of pozzolanic materials. Thus, two fans have been used to supply the required air for burning. The first fan with a power of 3700 W and 2950 cycles per minute was placed in the lower part of the furnace with a maximum flow ventilation of 745 $\frac{m^3}{h}$. The second fan with a power of 75 W and 2900 cycles per minute was installed on the basic framework of a cylindrical body, which brings in the required air from the internal shells to the furnace via the embedded holes on the mixer rods. The maximum output flow of the second fan is 245 $\frac{m^3}{h}$. The temperature inside the furnace is measured by three thermocouples in three different parts of the furnace [14].

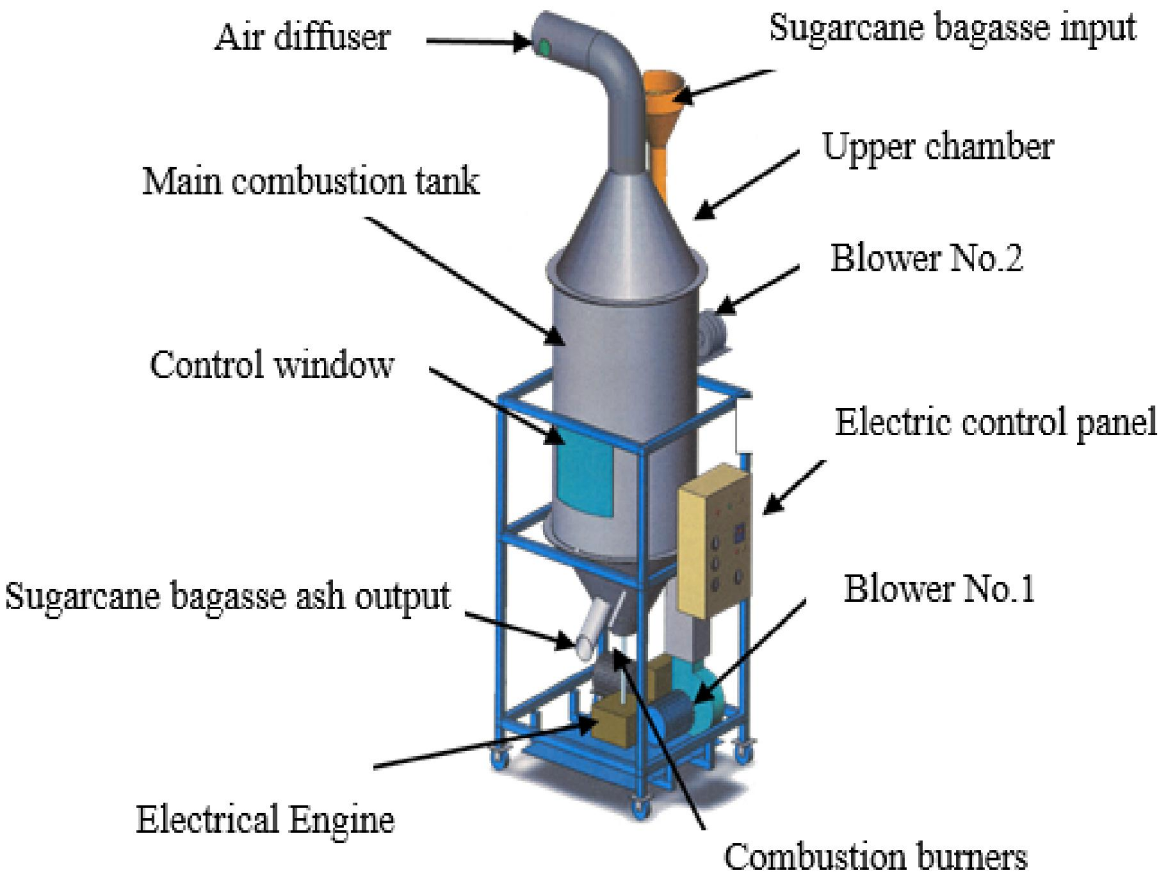
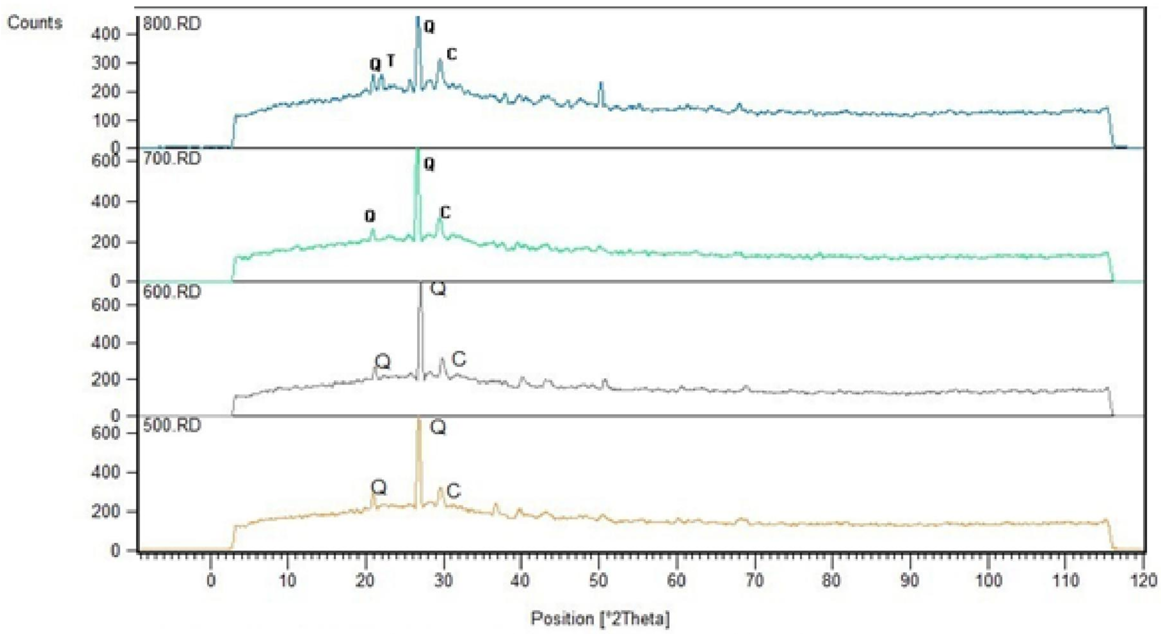


Figure 1 - Schematic image of the furnace used for burning process [14]

To produce MK with suitable pozzolanic activities, different specimens have been burnt in various conditions of time and temperature. According to the previous studies, it was confirmed that burning at the temperature between 500 ˚C and 800 ˚C will produce amorphous silica. In addition to temperature change, the duration of ignition was another variable parameter which was studied. Considering the system performance, it was identified that all specimens in different temperatures complete the burning process in 60 minutes.

In the next step, to determine the crystalline composition in different specimens, the experiment of X-ray diffraction (XRD) test was carried out on the kaoline. So, local kaolin with high kaolinite content (K) was thermally treated by the special furnace at 500, 600, 700, and 800° C in 60 minutes burning time to produce MK. Figure 2 depicts the development procedure of the crystalline peaks of the MK. As shown in Figure 2, the silica of the ash in 800 $°C$ has been crystallized less than at other temperatures and has a higher amorphous degree, so in the temperature of 800 $°C$, the silica of the MK has the highest amorphous degree. Therefore, the MK produced in 800 $°C$ temperature and burning time of 60 minutes was optimal. Diffraction peaks of ettringite and C–S–H phases are observable in samples with MK replacement.



Q: Quartz, Ca: Calcite, Feo: Iron oxide, T: Tridymite

Figure 2 - X-ray diffraction patterns of MK production with different temperature burning

The chemical composition of MK used as SCM and chemical and mineralogical analyses of kaolin are given in Tables 1 and 2, respectively. Local natural sand according to ASTM C33 with the maximum aggregate size of 4.75 mm, and crushed calcareous stone with the maximum aggregate size of 19 mm were used [15]. The coarse aggregates have a specific gravity and water absorption of 2580 kg/m^3 and 1.74%, respectively, and the fine aggregate has a water absorption of 2.3% and a specific gravity of 2560 kg/m^3. Potable water was used for casting and curing of all concrete specimens. The polycarboxylic acid-based superplasticizer (Gelenium-110P) was employed to achieve the desired workability.

Table 1 – Physical and chemical characteristics of cement and metakaolin

	Physical Tests		*Chemical Analysis, %*							
	Specific Gravity	Blaine, (cm^2/g)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
Cement	3.21	3200	21.32	3.83	2.76	62.02	3.44	0.12	0.73	2.98
Metakaolin	2.53	3700	74.3	17.8	0.82	3.38	0.22	0.0	0.39	2.56

Table 2 – Chemical and mineralogical analyses of kaolin

	Chemical Analysis, %								*mineralogical analysis, %*			
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI	Kaolinite	Quartz	Calcite	Other
Kaolin	74.97	17.8	0.81	2.22	0.13	0.05	0.55	7.18	50.73	39.27	6	4

Specimens Preparation

The concrete production was carried out in a mixer of 50 liters' capacity. Group 1 and Group 2 of concrete mixtures were designed at 0.4 and 0.5 water/binder (w/b) ratios, respectively, and had a constant total binder (cement + MK) content of 400 kg/m^3. The percentages of MK that replace OPC in this research are 0%, 10%, 12.5% and 15% by mass of cement that were added to clinker in the laboratory. Details of the mixtures are presented in Table 3. The casting of concrete specimens was conducted in two layers. Each layer was compacted on a vibrating table to ensure proper compaction and to reduce the air voids. After casting, the concrete specimens were covered with a wet towel and cured under laboratory conditions. After 24 h they were demoded and cured in lime-saturated water at 23 ± 2°C to prevent possible leaching of $Ca(OH)_2$ from these specimens.

Table 3 – Mix proportions of concrete

Group	Mix	W/b	Metakaolin (%)	Metakaolin (Kg/m^3)	Cement (Kg/m^3)	Water (Kg/m^3)	Coarse Aggregate (Kg/m^3)	Fine aggregate (Kg/m^3)	Slump (mm)
1	OPC	0.5	0	0	400	200	765	935	90
	MK10	0.5	10	40	360	200	765	935	85
	MK12.5	0.5	12.5	50	350	200	765	935	90
	MK15	0.5	15	60	340	200	765	935	85
2	OPC	0.4	0	0	400	160	810	990	80
	MK10	0.4	10	40	360	160	810	990	75
	MK12.5	0.4	12.5	50	350	160	810	990	80
	MK15	0.4	15	60	340	160	810	990	80

Test Methods

3.3.1. Fresh concrete tests

The slump flow test was conducted in conformity with the standard techniques given by ASTM C143 [16]. The slump flows were kept constant at 70±10 mm. Superplasticizer was used at slight percentages according to the results obtained for the slumps. The slump flow test was carried out to evaluate the capabilities of MK in the slump retention. The density of the mixtures was obtained by weighing the fresh concrete into a standard mold with a specific volume in accordance with ASTM C 138 standard [17].

3.3.2. Compressive Strength

Concrete cubes of 100×100×100 mm dimension were cast for compressive strength. They were tested for compressive strength after 7, 28, 90 and 180 days of water curing. For each age, three specimens were tested, and the mean value of these measurements is reported.

3.3.3. Transport tests

There are different methods to evaluate the absorption of concrete samples with MK. One is testing water absorption based on BS 1881-Part 122 [18]. The cubic specimens were dried at 45 °C for 14 days to reach the constant weight. Then samples were immersed in water and scaled after 0.5, 1, 24, 72 and 168 hours to measure the weight variation. This method would evaluate water absorption that happened in pores. These tiny voids are emptied by oven drying and occupied again with water after the immersion.

In another approach, capillary absorption is measured through the non-saturated concrete specimens. In this method, a sample is in adjacent with a water layer on one lateral and absorbed water evaporation from the other part, a steady flowing regime through capillary absorption is established [19]. The test was performed for measuring of capillary water absorption in accordance with RILEM CPC 11.2, TC 14-CPC for testing capillary absorption of MK specimens. The cubic specimens with 100 mm dimensions were dried in the oven at 45 ± 5 °C. They were immersed in a water bath with 5 mm depth.

3.3.4. Freeze and Thaw

The resistance to freezing/thawing of MK samples was evaluated in accordance with ASTM C 666 [20], in which specimens were subjected to repeated freezing and thawing cycles. Specimens were used to measure the fundamental transverse frequency by using the force resonance method. Samples with dimensions of 100 × 100 × 400 mm were cured under standard conditions and tested for integrity by recording the relative dynamic modulus of elasticity (Eq. 1) every 25 cycles up to 300 cycles.

$$P_c = \frac{n_1^2}{n^2} \times 100$$

P_c: relative dynamic modulus of elasticity, after c cycles of freezing/thawing (%),

n: the fundamental transverse frequency at 0 cycles of freezing/thawing

n_1: the fundamental transverse frequency after c cycles of freezing/thawing

3.3.5. Surface Resistivity (SR)

The electrical resistivity meter was used to measure the surface resistivity (SR) of the specimens. This non-destructive laboratory test method measures the electrical resistivity of water-saturated concrete and provides an indication of its permeability. Electrical resistivity is a function of moisture and electrolyte content of the pores in concrete, which measured based on AC impedance spectrometry using a resistance meter [21]. Experiments have been done with Wenner 4-probe meter. The probe array spacing used was 40 mm. The resistivity measurements were taken at four quaternary longitudinal locations of the specimen. The likely difference and resulting current can be applied to find the electrical resistance. Three readings were obtained from the data logger for each cylinder specimen. The bulk resistivity was calculated as follows (Eq. 2):

$$\rho = \frac{V}{I} \times \left({}^{A}/_{L}\right) = R \times \left({}^{A}/_{L}\right)$$

where ρ is the electrical resistivity (KΩ cm), R is bulk electrical resistance (KΩ), A is a cross-sectional area (cm^2), L is the distance between two electrodes (cm), I is measured current, and V is the voltage.

3.3.6. Water Penetration

The water penetration test, which is most frequently used to evaluate the permeability of concrete, is the one specified by BS EN-12390-8. In this test, water was applied on one face of the 150 mm concrete cubes specimen under a pressure of 0.5 Mpa. This pressure was maintained constant for a period of 72 hours. After the completion of the test, the specimens were taken out and split open into two halves. The water penetration profile on the concrete surface was marked, afterwards the maximum depth of water penetration in specimens was recorded and considered as an indicator of the water penetration.

3.3.7. Rapid Chloride Permeability Test (RCPT)

The resistance of concrete to chloride ions ingress was assessed by the rapid chloride permeability test (RCPT) at the ages of 7, 28, 90 and 180 days of water curing in conformity with ASTM C-1202. Three specimens of 100 mm in diameter and 50 mm in thickness which had been conditioned according to the standard were subjected to a 60-V potential for six hours. The total charge passed through the concrete specimens was determined and used to evaluate the chloride permeability of each concrete mixture.

RESULT and DISCUSSION

4.1 Superplasticizer (SP) demand

The required SP dosage to achieve the target slump flow of 70±10 mm of all binary mixes is shown in Figure 3. The superplasticizer was used at low percentages according to the results obtained for the slumps. The SP dosage of the group 2 blends was significantly higher than group 1 and the control concrete. The higher the replacement of cement by MK, the more SP was required to achieve the target slump flow, which probably stemmed from a large amount of pores in the frame structure and high surface area of MK and in turn led to increased water demand. A binder having greater surface area requires more water to obtain a given slump flow, so more SP is required to keep the content of water constant.

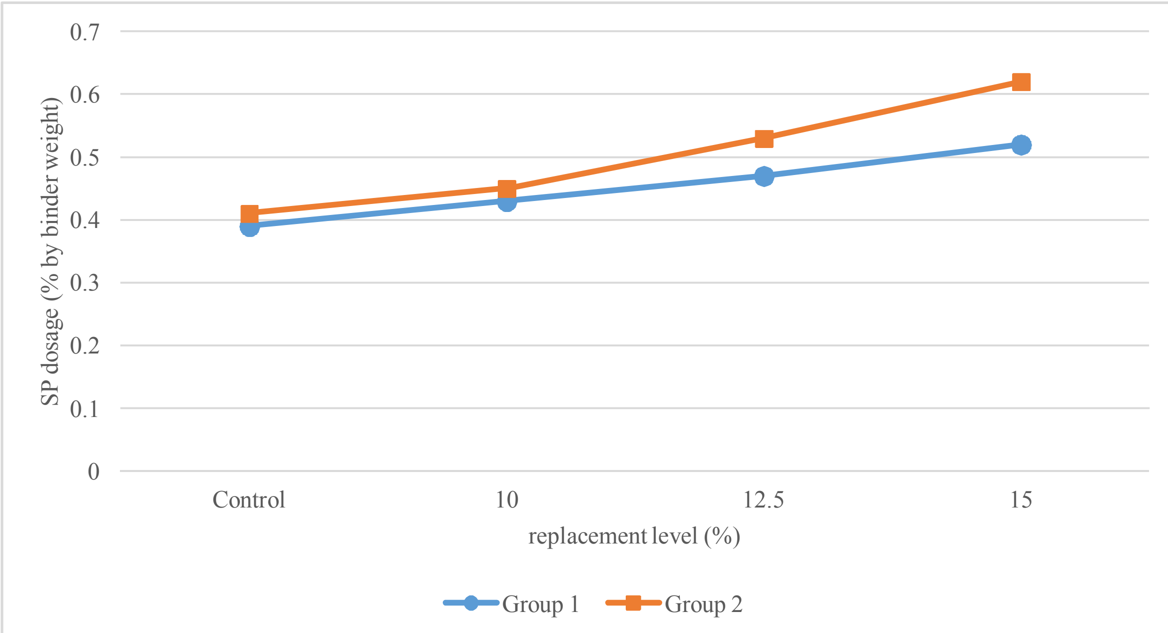
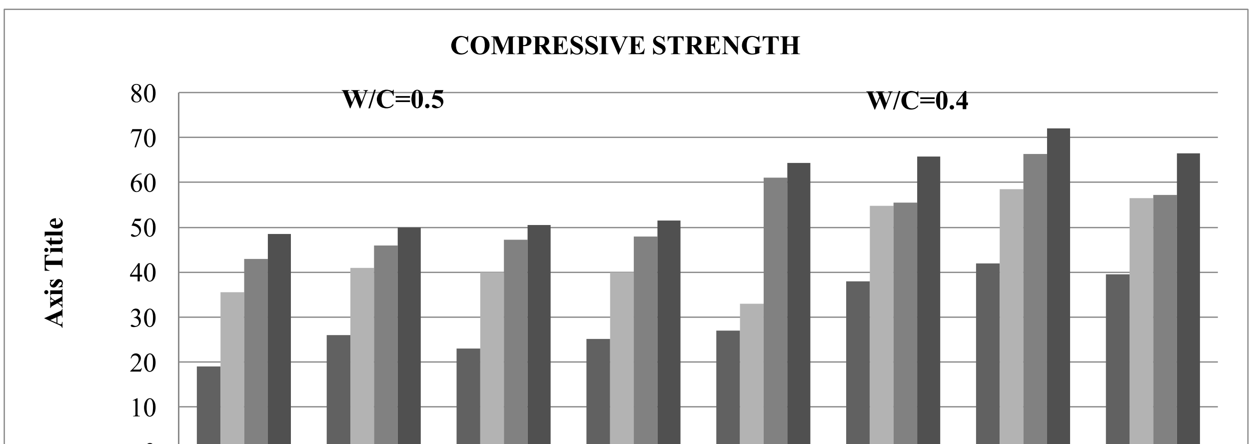


Figure 3 - SP dosage of binary concrete mixes to obtain target fluidity

4.2. Compressive Strength

The compressive strengths of concrete specimens with varying w/b ratios are shown in Figure 4. As expected, the level of compressive strength developed with the period of curing and with decreasing the w/b ratio. According to the literature, the main factors that affect the contribution of MK in the strength are (a) the filling effect, (b) the dilution effect, and (c) the pozzolanic reaction of MK with CH [22].



	OPC	MK10	MK12.5	MK15	OPC	MK10	MK12.5	MK15
7 days	19	26	23	25.1	27	38	42	39.5
28 days	35.5	41	40	40	33	54.75	58.5	56.5
90 days	43	46	47.2	48	61	55.5	66.3	57.25
180 days	48.5	50	50.5	51.5	64.3	65.8	72	66.5

Figure 4 – The effect of metakaolin on the compressive strength at various ages

In general, the MK concretes had higher compressive strengths up to 180 days, at various ages, when compared with the OPC concrete. As a case in point, the strength of the Group 2 MK concrete at the age of 180 days age were higher than that of the control by about 13.4%, 24.1%and 14.6% for MK10, MK12.5and MK15, respectively. The reduction in compressive strength for MK15 compared to MK12.5 was explained as the result of a clinker dilution effect. The dilution effect is a consequence of replacing a part of cement by the same quantity of MK. In MK concrete, the filler effect, the pozzolanic reaction of MK with CH and compounding effect (synergetic effect of mineral admixture) react opposite of the dilution effects [15]. Thus, there is an optimum MK replacement for MK concrete. However, in Group 1, the replacement rate of 15% gives the best result when compared to other replacement levels. This was in a good agreement with the measurements reported by Parande et al. [12].

4.3. Transport tests

For measuring transport performance, the water absorption and capillary absorption of the concrete samples containing MK were measured at different time intervals. The results confirmed that the percentage of water absorption and the height of capillary absorption are reduced by applying the MK. The performance of group 2 was slightly better than group 1 in absorption. The reason is attributed to improvement in the Interfacial Transition Zone (ITZ) of group 1 due to a reduction in total specific pore volumes of concretes. As it is shown in Table 4, increasing the curing time and percentages of MK may lead to a reduction in permeable gel pores due to the great bridging and filler effects.

Table 4 - Results of water absorption and capillary water absorption versus time

		Time (hr)									
	Mix Design ID	0.5	1	3	24	72	168	24	48	72	168
		Water Absorption (%)						Capillary Water Absorption (mm)			
Group 1	OPC	2.55	3.45	4.38	4.76	5.08	5.57	2.5	4.2	5.4	6.0
	MK10	2.11	3.11	3.86	4.42	4.84	5.15	2.3	3.7	5.0	5.7
	MK12.5	1.84	2.71	3.57	4.02	4.32	4.67	2.2	3.3	4.7	5.3
	MK15	1.73	2.65	3.37	4.01	4.37	4.78	2.0	3.2	4.5	5.2
Group 2	OPC	2.42	3.33	4.27	4.61	4.89	5.37	2.7	4.1	5.3	5.9
	MK10	1.97	2.93	3.75	4.27	4.68	5.04	2.3	3.6	4.9	5.5
	MK12.5	1.65	2.59	3.42	3.90	4.20	4.57	2.2	3.3	4.6	5.1
	MK15	1.54	2.50	3.25	3.82	4.25	4.66	1.9	3.1	4.5	5.0

4.4. Freeze and Thaw

Visual observations indicated that honeycomb voids were found on specimens as freeze and thaw cycles continued and these voids caused the mass loss. The weight was recorded for all the specimens during the freeze and thaw operations. One factor to evaluate the damage in concrete specimens subjected to the freeze and thaw cycles was a mass loss ratio. The relationship between the mass loss ratio of different specimens is plotted in Figure 5.

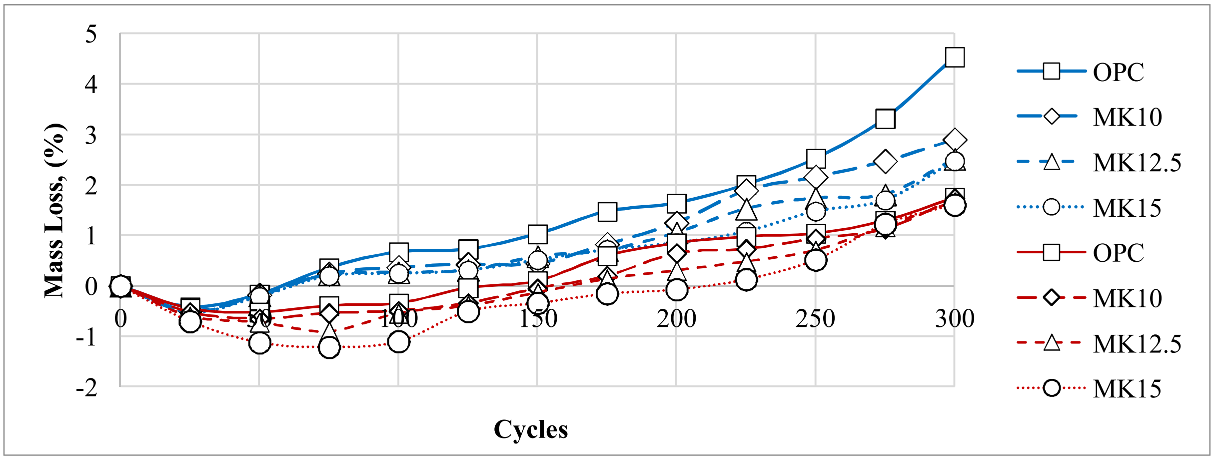


Figure 5 - Mass loss of concrete versus freeze and thaw cycles

As shown in Figure 5, the amount of mass loss was higher in group 1. It indicates that higher water-to-binder-ratio likely increased the freeze and thaw degradation. Lower water-to-binder-ratio (under same and adequate curing condition) decreased the volume of capillary pores and the permeability of the concrete. It prevented more damages, and thus, resulted in better freeze-thaw resistance. Moreover, it is illustrated that the mass loss increased gradually for all the concrete specimens as the freeze-thaw cycles continue. The highest mass loss was 4.51% for a control sample, and the lowest mass loss was 0.62% for 15% MK replacement with 0.4 water-to-binder-ratio. This might be attributed to a denser microstructure in the MK, due to the smaller size of MK particles, which might lead to less damage and less mass loss within the concrete.

Dynamic modulus of the elasticity of the different mixture samples was measured at constant intervals for up to 300 freeze and thaw cycles. As seen in Table 5, the mixtures with MK replacements displayed a slight decrease in the dynamic modulus of elasticity throughout the freeze and thaw test as opposed to what happened with the control specimen. Compared to the initial condition of samples before freeze and thaw cycles, the control sample showed a decrease 37.6% in the dynamic modulus of elasticity at 300th cycles while, the specimen with the 10, 12.5, and 15% MK showed 33.4, 27.1, and 17.4% decrease in the dynamic modulus of elasticity, respectively. These reductions were smaller in Group 2, which had less water-to-cement-ratio. The dynamic modulus of elasticity data is presented in Table 5.

Table 5 - Dynamic modulus of concrete after the particular freeze and thaw cycles (Gpa).

	Mix Designs	Freeze and Thaw Cycles											
		25	50	75	100	125	150	175	200	225	250	275	300
Group 1	OPC	38.67	36.25	33.73	32.26	31.45	30.11	29.24	28.04	25.45	24.99	24.55	24.12
	MK10	38.41	37.24	35.26	33.63	32.26	30.62	29.15	28.76	27.11	26.60	26.13	25.56
	MK12.5	39.73	38.49	37.81	35.50	34.31	33.87	32.90	32.77	31.64	30.61	30.04	28.95
	MK15	39.65	39.53	38.01	36.65	36.17	36.38	35.95	34.82	34.08	33.71	33.01	32.74
Group 2	OPC	37.14	38.02	35.27	33.75	31.98	30.78	29.68	27.14	25.55	26.19	25.66	25.36
	MK10	39.49	38.09	36.44	34.87	33.49	32.33	31.60	30.63	29.05	28.73	27.69	27.69
	MK12.5	41.01	40.36	38.75	37.65	36.40	35.38	34.43	33.38	31.91	31.43	31.03	30.70
	MK15	40.46	38.91	37.52	38.00	36.52	35.93	36.39	35.09	35.30	32.99	34.32	34.08

The relative dynamic modulus of elasticity is the ratio of the dynamic modulus at a particular interval, relative to the dynamic modulus at the start of the test. Figure 6 displays the relative dynamic modulus of elasticity data during the freeze and thaw cycles. The obtained data for specimens have been fitted to the function of the number of freeze and thaw cycles. The finest fits for samples have been acquired with a power equation (Eq. 4).

$$E_n/E_0 = k \times n^t \quad (4)$$

where n is the number of freeze-thaw cycles, k and t are the coefficients. Table 6 summarizes all the test data on the dynamic modulus of elasticity and correlation coefficients.

Table 6 - Dynamic modulus of elasticity correlation coefficients

No.	Mixture Design	k	t	R2	The decrease in dynamic modulus of elasticity (%)
Group 1	OPC	2.104	-0.203	0.916	37.62
	MK10	1.940	-0.184	0.921	33.45
	MK12.5	1.563	-0.125	0.915	27.14
	MK15	1.347	-0.080	0.864	17.42
Group 2	OPC	2.019	-0.194	0.915	31.71
	MK10	1.752	-0.155	0.926	29.89
	MK12.5	1.614	-0.130	0.915	25.14
	MK15	1.307	-0.071	0.849	15.78

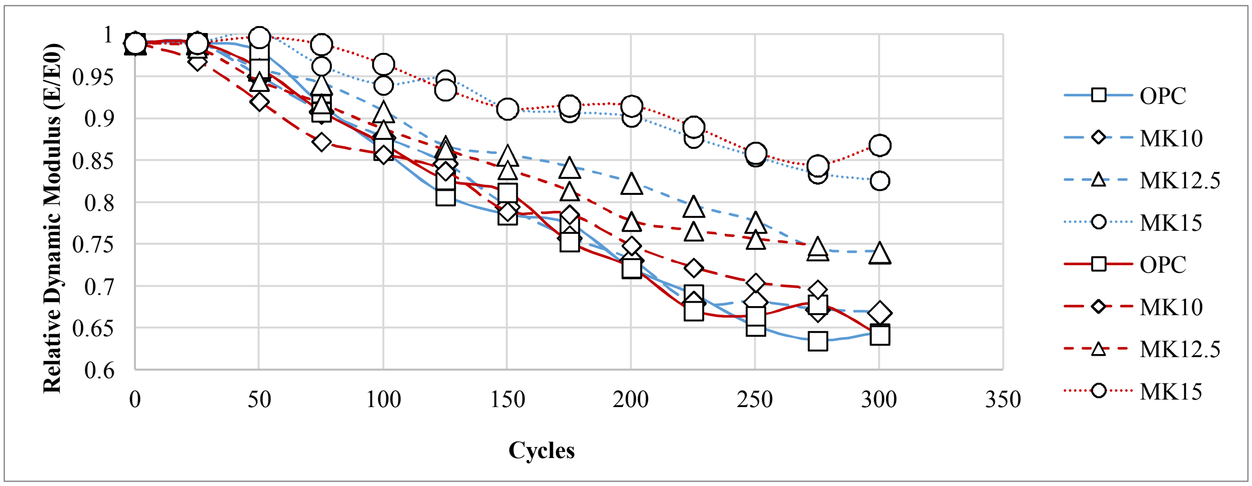
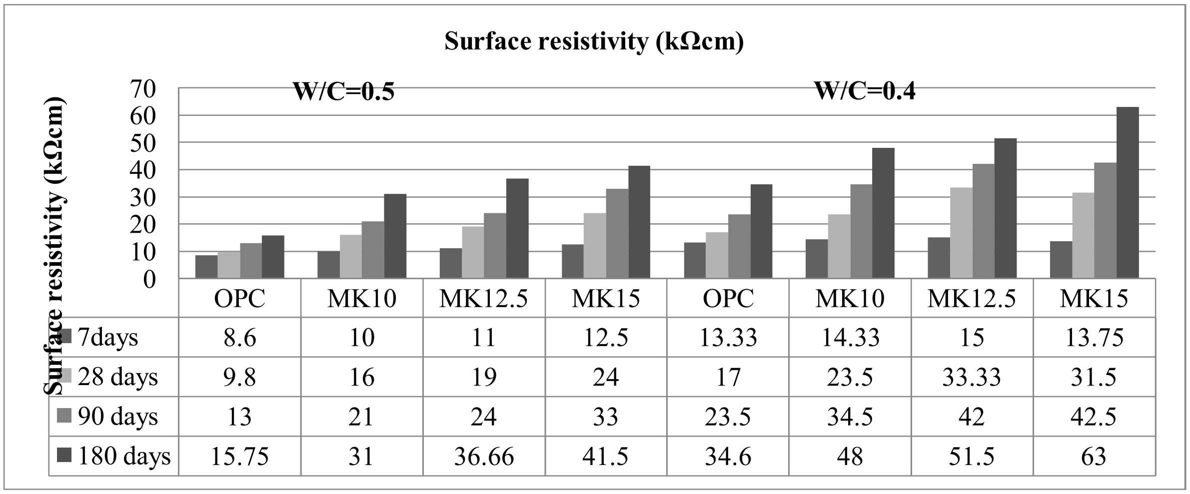


Figure 6 - Evolution of dynamic modulus of elasticity of concrete subjected to freeze and thaw cycles

4.5. Surface Resistivity (SR)

The concrete Surface Resistivity (SR) test is a suitable indicator for concrete penetration and chloride ion permeability. It is a non-destructive, simple, rapid and economical method that can also be used on the site. The electrical resistivity of concrete represents moving ions (such as chloride ions) in pore solution. Concrete resistivity depends both on the microstructure properties of the concrete and the conductivity of the pore solution. The conductivity property of the concrete is predominantly governed by the chemical compositions of the pore solutions; however, it is also affected by the pore structure of the concrete. It can especially be used on concretes when a significant portion of their cementitious chemical reactions have been completed such as those concretes made with silica fume or MK. Results of the electrical resistivity tests, see Figure 7, show that using MK drastically enhances the electrical resistivity compared to the OPC concrete, with the rate being about 2~4 times higher for the 15% MK concrete. In addition, the electrical resistivity increases with decreasing the w/b ratio. The maximum value of electrical resistivity is 63 kΩcm for the MK15 (Group 2) mixture after 180 days, and the minimum is 15.75 kΩcm for the OPC (Group 1) mixture. Parande et al. [12] showed that incorporation of MK (up to 15%) into PC concrete improves the electrical resistivity of concrete.

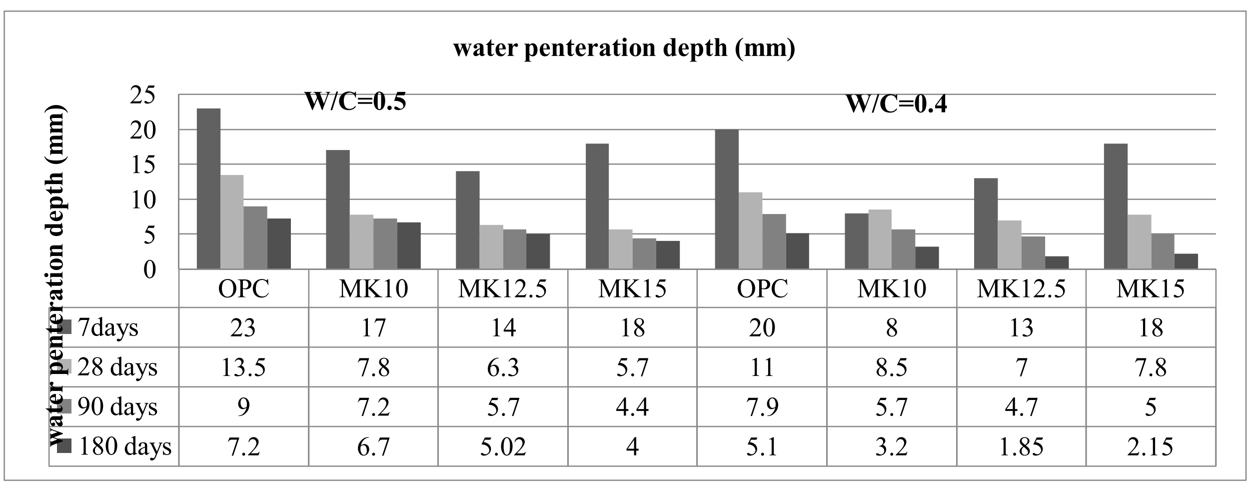


	OPC	MK10	MK12.5	MK15	OPC	MK10	MK12.5	MK15
7days	8.6	10	11	12.5	13.33	14.33	15	13.75
28 days	9.8	16	19	24	17	23.5	33.33	31.5
90 days	13	21	24	33	23.5	34.5	42	42.5
180 days	15.75	31	36.66	41.5	34.6	48	51.5	63

Figure 7 – The effect of metakaolin on the Surface resistivity (kΩcm) at various ages

4.6. Water Penetration Test

Permeability is known to be one of the most important aspects of concrete durability. Concrete with lower permeability shows better resistance against chemical attacks. When water penetrates into the concrete, some soluble salts including chloride ions penetrate into concrete and cause corrosion of reinforcements. Generally, it seems that lower permeability causes higher durability in concretes [23]. Water penetration test was used to evaluate the permeability of concretes and validity of these tests has been approved [24]. Figure 8 shows the results of the water penetration depths in all concrete mixtures. As expected, the lower depth was obtained at 180 days for all concretes, and the MK concretes provided less water penetration depth than OPC concretes. This issue is related to the filler effect, the pozzolanic reaction, and the heterogeneous nucleation. For example, in Group 2, MK12.5 specimens provided a water penetration depth close to 2 mm, while OPC provided 5 mm water penetration depth. The increase of penetration depths for MK15 compared to MK12.5 was explained to be the result of a clinker dilution effect. However, this phenomenon has an insignificant effect on water penetration depth.

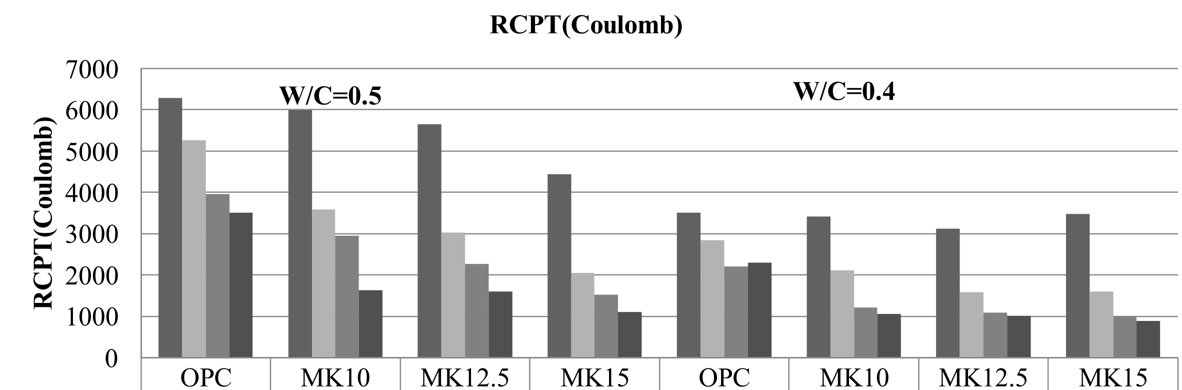


	OPC	MK10	MK12.5	MK15	OPC	MK10	MK12.5	MK15
7days	23	17	14	18	20	8	13	18
28 days	13.5	7.8	6.3	5.7	11	8.5	7	7.8
90 days	9	7.2	5.7	4.4	7.9	5.7	4.7	5
180 days	7.2	6.7	5.02	4	5.1	3.2	1.85	2.15

Figure 8 – The effect of metakaolin on the water penetration depth (mm) at various ages

4.3. Rapid Chloride Permeability Test (RCPT)

The results for chloride penetration, measured in terms of the electric charge passed through the specimens in coulombs, obtained for the age groups of 7, 28, 90 and 180 days are presented in Figure 9. With a continuous moist-curing of up to 180 days and decreasing the w/b ratio, the charge passed through all specimens were reduced. Results show that using MK significantly enhances the resistance to chloride penetration compared with the OPC concrete. Kim et al. [25] reported that all of the mixtures with MK revealed very low levels of permeability. Also, Gruber et al. [11] showed that the use of 8% and 12% high reactivity MK significantly decreased penetration of chloride ion in concretes. The enhancement of the resistance to chloride penetration may be related to the pozzolanic reaction of MK with $Ca(OH)_2$ and reduced electrical conductivity of MK concrete. In Group 1, at the age of 28 days, the OPC concrete specimens showed the highest value of 5266 coulombs while the charge passed through the MK15% concrete was 2052 coulombs. According to ASTM C 1202, when the charge passed through concrete during a 6 h period is below 1000 coulombs, it is categorized as very low chloride permeability. In Group 2, the chloride permeability of the concrete specimens incorporating 12.5% and15% MK was “very low”, while that of the concrete specimens with 0% and 10% MK were “moderate” and “low”, respectively.



	OPC	MK10	MK12.5	MK15	OPC	MK10	MK12.5	MK15
7days	6289.5	5983.5	5644	4428.5	3504.5	3406.5	3112	3481
28 days	5266	3578	3017.5	2052.5	2842.5	2115.5	1579	1594
90 days	3956	2953	2268	1524	2195.5	1211.5	1080	998
180 days	3506	1635	1594	1101	2291	1049	1015.5	887.5

Figure 9 – The effect of metakaolin on the rapid chloride ions permeability (Coulomb) at various ages

5. CONCLUSIONS

In this study, the effect of MK as supplementary cementing materials and filling materials on the strength and durability of concretes was investigated.

From the results obtained in this study, the following conclusion can be drawn:

According to slump flow test results, the higher the replacement of cement by MK, the more SP was required to achieve the target slump flow, which probably stemmed from a significant amount of pores in the frame structure. The high surface area of MK and in turn leads to increased water demand.

Concrete incorporating a new MK had higher compressive strength at various ages up to 180 days when compared with the OPC concrete. The level of compressive strength developed with the period of curing and with decreasing the w/b ratio. For the materials in this study at w/b ratio 0.4, the optimum replacement of MK was 12.5%.

MK concretes provided lower water penetration depth.

Lower water-to-binder-ratio decreased the volume of capillary pores and the permeability of the concrete. It prevented more damages, and thus, resulted in better freeze-thaw resistance. Also, the second group of mixtures experienced less mass loss and dynamic elasticity modulus loss.

In RCPT test, Results show that the use of MK significantly enhanced the resistance to chloride penetration when compared with the OPC concrete. This improvement increased with increasing MK content.

Results of the Surface Resistivity (SR) tests show that using MK drastically enhanced the electrical resistivity in comparison with the OPC concrete at about 2~4 times higher for the 15% MK mixture.

REFERENCES

[1] R. Siddique, M. Iqbal Khan, "Supplementary Cementing Materials", Springer- Verlag Berkin Heidelberg, 2011.

[2] Rafat Siddique, Juvas Klaus, "Influence of metakaolin on the properties of mortar and concrete: A review" Applied Clay Science 43 (2009) 392–400.

[3] C. Vizcayno, R. Castello, I. Ranz, B. Calvo, "Some physico-chemical alterations caused by mechanochemical treatments in kaolinites of different structural order", Thermochimica Acta 428 (2005) 173–183.

[4] Kostuch, J.A., Walters, G.V., Jones, T.R., 1993. High Performance Concrete Incorporating Metakaolin — a review. Concrete 2000. University of Dundee, pp. 1799–1811.

[5] Johari, M., Brooks, J., Kabir, S., Rivard, P, "Influence of supplementary cementitious materials on engineering properties of high strength concrete", construction and materials, 25 (2011) 2639-48.

[6] Li Z, Ding Z. Property improvement of Portland cement by incorporating with metakaolin and slag. Cement and Concrete Research 33 (40) (2003), 579–584.

[7] Biljana Ilic´, Vlastimir Radonjanin, Mirjana Malešev, Miodrag Zdujic´, Aleksandra Mitrovic´, "Study on the addition effect of metakaolin and mechanically activated kaolin on cement strength and microstructure under different curing conditions", Construction and Building Materials 133 (2017) 243–252.

[8] Omid Rezaifar, Mohsen Hasanzadeh, Majid Gholhaki, "Concrete made with hybrid blends of crumb rubber and metakaolin: Optimization using Response Surface Method", Construction and Building Materials 123 (2016) 59–68.

[9] GENG Haining, LI Qiu, "Water Absorption and Hydration Products of Metakaolin Modified Mortar", Key Engineering Materials Vol. 726,(2017) pp 505-509.

[10] Efstratios G. Badogiannis, Ioannis P. Sfikas, Dimitra V. Voukia, Konstantinos G. Trezos, Sotirios G. Tsivilis, "Durability of metakaolin Self-Compacting Concrete", Construction and Building Materials 82 (2015) 133–141.

[11] Gruber K.A, Ramlochan T, Boddy A, Hooton R.D, Thomas M.D.A. Increasing concrete durability with high-reactivity metakaolin. Cement and Concrete Composites 23 (6) (2001); 479-484.

[12] Parande AK, Ramesh Babu B, Karthik MA, Kumaar K.K, Palaniswamy N. Study on strength and corrosion performance for steel embedded in metakaolin blended concrete/mortar. Construction and Building Materials 22 (3) (2008); 127–134.

[13] ASTM C150 / C150M-16e1, Standard Specification for Portland Cement, ASTM International, West Conshohocken, PA, 2016.

[14] Ramezanianpour AA ,Mahdi khani M , Ahmadibeni Gh (2009) The Effect of Rice Husk Ash on Mechanical Properties and Durability of Sustainable Concretes, INTERNATIONAL JOURNAL OF CIVIL ENGINEERING 7 (2), 83-91.

[15] ASTM C33 / C33M-16e1, Standard Specification for Concrete Aggregates, ASTM International, West Conshohocken, PA, 2016.

[16] ASTM C143/C143M-15a Standard Test Method for Slump of Hydraulic-Cement Concrete, ASTM International, West Conshohocken, PA, 2015.

[17] ASTM C138/C138M-16a Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete, ASTM International, West Conshohocken, PA, 2016.

[18] BS 1881–116, 1983. Testing concrete. Method for determination of compressive strength of concrete cubes.

[19] D. Perraton, P.C. Aïtcin, A. Carles-Gbergues., Permeability, as seen by the researcher., Y. Malier (Ed.), High Performance Concrete: From Material to Structure, E & FN Spon, London, UK (1994), pp. 186–195

[20] ASTM C666/C666M-15 Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing, ASTM International, West Conshohocken, PA, 2015.

[21] P. Gu, P. Xie, J.J. Beaudoin, R. Brousseau., AC impedance spectroscopy (II): microstructural characterization of hydrating cement–silica fume systems., Cem. Concr. Res., 23 (1992), pp. 157–168

[22] Wild S, Khatib JM, Jones A. Relative strength, pozzolanic activity and cement hydration in

superplasticized metakaolin concrete. Cement and Concrete Research 26 (1996):1537–1544.
[23] Ding Z, Zhang D, Yu R. High strength composite cement. China Building Material Science Technology 1(1999); 14–7.
[24] Krieg W. Rapid chloride permeability testing, a critical review, proceedings of the concrete in hot and aggressive environments (2008); 147-156.
[25] Kim HS, Lee SH, Moon HY. Strength properties and durability aspects of high strength concrete using Korean metakaolin. Construction and Building Materials 21(6) (2007); 1229–1237.

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