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Influence of Metakaolin and Cements Types on Compressive Strength and Transport Properties of Self-Consolidating Concrete

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***Abstract*—**The self-consolidating concrete (SCC) performance over ordinary concrete is generally related to the ingredients used. The metakaolin can modify various properties of concrete, due to high pozzolanic reactions and also makes a denser microstructure. The objective of this paper is to examine the influence of three types of Portland cement and metakaolin on compressive strength and transport properties of SCC at early ages and up to 90 days. Six concrete mixtures were prepared with three types of different cements and substitution of 15% metakaolin. The results show that the highest value of compressive strength was achieved for Portland Slag Cement (PSC) and without any metakaolin at age of 90 days. Conversely, the lowest level of compressive strength at all ages of conservation was obtained for Pozzolanic Portland Cement (PPC) and containing 15% metakaolin. As can be seen in the results, compressive strength in SCC containing Portland cement type II with metakaolin is higher compared to that relative to SCC without metakaolin from 28 days of age. On the other hand, the samples containing PSC and PPC with metakaolin had a lower compressive strength than the plain samples. Therefore, it can be concluded that metakaolin has a negative effect on the compressive strength of SCC containing PSC and PPC. In addition, results show that metakaolin has enhanced chloride durability of SCCs and reduced capillary water absorption at 28, 90 days.

***Keywords*—**SCC, metakaolin, cement type, compressive strength, chloride diffusion.

I. Introduction

CONCRETE is one of the most widely used infrastructure materials in the world, and usage of concrete continues to grow in construction projects. SCC is a concrete that was developed in Japan in the 1980s [1]. The goal of SCC development was to create a concrete capable of flowing freely through concrete forms that have congested reinforcement. Seismically active regions require large amounts of reinforcement, and it is arduous to effectively vibrate conventional concretes into such forms. SCCs solve this problem by freely flowing into forms and around reinforcement without needing any vibration [2]. On the other hand, different SCC parameters, in fresh or hardened states, can be affected by incorporating supplementary cement material (SCM). In general, two principles should be considered for the use of admixture in self-compacting concrete: The first is the replacement of cement and adjuvant by mineral addition to produce an economical SCC and the second is the improvement of the mechanical properties, increasing durability and also the workability while simultaneously reducing bleeding and segregation. According to Audenaert et al., when the proportion of the different mixtures of SCC are identical but of various cement type, the rate of chloride penetration decreases by cement high strength class or a blast furnace slag cement [3].

The materials based on PPC have a higher porosity than those based on Portland cement type II, whereas the porosimetric distribution shifts to smaller pores. In the long term, the pozolanic reaction of concrete based on PPC results in the formation of a higher amount of CSH [4]. Pastes containing slags or fly ash have a lower coefficient of diffusion; this may be due to the modification of the porous structure of the pastes by formation of CSH, CAH and CAFH, by creating a much finer microstructure [5]. In terms of rheology, fly ash reduces the shear threshold, but the plastic viscosity can be increased or decreased. For example, Sonebi found that the use of fly ash reduces these two parameters in SCC [6]. However, Park et al. found that fly ash had a low impact on the shear threshold and the viscosity of the cement pastes [7]. Fly ash can also reduce segregation and improve stability. According to results presented by Sabet et al., despite the natural zeolite and silica fume, incorporating fly ash into the mixtures reduces the amount of superplasticizer to arrive at the target sagging range [8]. The superplasticizer dose of mixture containing fly ash was 0.2% and 0.5% less than the control concrete mix. The reason can be attributed to the spherical geometry of fly ash particles. They roll easily over other particles and can disperse the agglomeration of cement particles. The spherical shapes reduce friction at the overall paste interface which produces a "ball bearing effect" at the point of contact. Consequently, the fluidity of concrete mixtures containing fly ash is high and less superplasticizer is required to achieve fluidity. The higher resistance in SCC based on fly ash generally concerns the formation of CSH gel. According to Felekoglu et al., a reaction takes place between the vitreous phase of the ash and the portlandite (CH) by the hydration of the cement which leads to the formation of CSH

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gel [9]. Therefore, the compressive strength of mixture containing fly ash increases especially in the long term with the development of the pozzolanic reaction.

Metakaolin is a pozzolanic material that is produced by the calcination of purified kaolinite clay at high temperatures, ranging from 600 to 900 °C. At these temperatures, the crystalline structure is destroyed and the chemically bound water is expelled. Metakaolin improves the strength and durability of concrete with three main actions: The filling effect, the acceleration of hydration of ordinary Portland cement and the pozzolanic reaction with calcium hydroxide. The main characteristic of metakaolin is its high reactivity with calcium hydroxide, $Ca(OH)_2$, and its ability to accelerate the hydration of cement. Compared to other cementitious additions, such as silica fume or pulverized fly ash, the pozzolanic action of metakaolin is expected to be more significant due to its high concentration of silica and alumina. Specifically, the calcium to silicon (C/S) ratio due to the calcium silica hydrates produced (C-S-H gel) is expected to be higher than the corresponding ratios for silica fumes and pulverized fly ash, which leads to a considerably improved concrete microstructure and ultimate improvement in overall concrete performance in terms of strength and durability. Moreover, as all the other ultrafine pozzolans, metakaolin contributes to filling the pores and voids, but consequently densifies the interface transition zones (ITZ) due to the microfiller effect. The last action refers to the physical action. The addition of metakaolin increases the high-range water reducing adjuvant demand in the SCC mixtures. Also, increasing levels of replacement metakaolins increase the demand for water due to its high chemical activity and high surface area. According to Vejmelkováa et al., SCC with metakaolin requires a greater amount of water [10]. They also concluded that the compressive strength of SCC with metakaolin increased very rapidly during the initial curing period and remained significantly higher. Conversely, the risk of corrosion of the reinforcement induced by diffusion of chloride ions is decreased in a concrete with metakaolin. In reality, the formation of a denser microstructure which is less porous with a discontinuous pores system becomes the criteria for the reduction of the permeability of the chloride ion. Pozzolanic reactions appear to be more capable of developing a discontinuous pore system. The addition of metakaolin makes it possible to increase the compressive strength of concrete and to improve the chemical resistance against acid attack which depends on the characteristics of the acid: pH and concentration. The increase in the mechanical and chemical resistance of the samples containing metakaolin is due to the fineness of the grains to the consistency of the mixtures with metakaolin, in other words, to the refinement of the pores within the cementitious matrix. This paper investigates and compares the effect of three different cements (Portland cement type II, PSC, PPC) and artificial pozzolans such as metakaolin on rheological behavior, compressive strength and transport properties of SCC at early ages and up to 90 days. For this purpose, first, different tests such as slump flow, L-box and sieve, were performed to study the fresh phase of SCC, and then mechanical testing was done on hardened states to evaluate the compressive strength of the different prepared mixtures. Tests concerning the transport phenomena were performed at different ages to evaluate durability properties, including porosity accessible to water, capillary absorption and chloride ion migration in non-steady state by electric field.

II. Experimental Program

A SCC formulation based on Portland cement type II was chosen as control concrete. The same composition was used for the other 2 SCCs where the Portland cement type II was completely replaced by PSC or PPC. In addition, metakaolin was used as an additive replaced with 15% Portland cement type II, PSC and PPC. In total, six SCCs were manufactured with a constant water to binder (W/b) ratio of 0.33 and a constant paste quantity of 600 kg/m^3. The gravel and sand contents of the mixtures were kept constant. Only one type of adjuvant was used during this study, a high range water-reducing admixture (HRWRA) based on chains of modified poly-carboxylate ether (PCE 180). The dosage of superplasticizer is experimentally determined from tests on fresh concrete to obtain a slump flow diameter of 685±25 mm for all SCCs. Table I shows the composition of the 6 SCCs studied and their designations. For all mix designs, the sand used is of granular class 0/4 of alluvial origin having a density of 2.5. In addition, gravel 6/12 from crushing siliceous rocks with a density of 2.58 and a water absorption coefficient of 0.62% is used in this study. Potable water was used for casting all concrete specimens.

TABLE I
Mixture Proportions of Concrete

Composition (kg/m^3)	SCC II	SCC II + MK	SCC PSC	SCC PSC + MK	SCC PPC	SCC PPC + MK
Gravel 6/12	780	780	780	780	780	780
Sand 0/4	780	780	780	780	780	780
Type II Portland Cement	350	297.5	-	-	-	-
PSC	-	-	350	297.5	-	-
PPC	-	-	-	-	350	297.5
Lime stone powder	250	250	250	250	250	250
Metakaolin	-	52.5	-	52.5	-	52.5
Water	200	200	200	200	200	200
Superplasticizer	3.3	4.9	2.5	3.6	3	4
W/b	0.33	0.33	0.33	0.33	0.33	0.33

III. Results and Discussion

A. Fresh State

In order to evaluate the effects of pozzolan and various cements on the fresh properties of SCC, slump flow and L-box tests were performed according to the procedure recommended by EFNARC Committee and also sieve test was performed according to the procedure recommended by AFGC 2000 [11], [12]. Table II presents the results of the performance tests carried out on the studied SCCs and which characterize their fluidity (slump flow), their filling capacity (L-BOX) and their resistance to segregation (sieve stability).

These results show that the proposed concrete formulations satisfy the specific criteria for qualification of a SCC. The results show that the use of metakaolin reduces the fluidity of the mixture, no doubt due to the large surface area of this admixture. Also, higher contents of superplasticizer were used. In fact, high open porosity and the irregular surface texture of metakaolin, associated with a specific surface area, decreased the available water around particles and resulted in slump reduction. This finding is consistent with that of Paiva et al. [11] who found that using metakaolin decreased the workability of the traditional concrete and SCC. On the other hand, the use of metakaolin seems to improve the resistance of the SCC to the segregation compared to the cements used. Besides, the use of PSC and, to a lesser extent PPC PPC, requires lower contents of superplasticizer.

TABLE II
TEST RESULTS OF FRESH SCCS

Mix name	Slump flow	L-box	Sieve test
	Dia (mm)	H2/H1	Segregation (%)
SCCII	680	0.85	8.6
SCCII+MK	670	0.84	7.3
SCC PSC	710	0.89	9.9
SCC PSC+MK	690	0.87	8.7
SCC PPC	690	0.86	8.2
SCC PPC+MK	660	0.83	6.6

B. Compressive Strength

Compressive strength of HSSCC mixes (fc) was measured on a total of 90 cubes of 100×100 mm at 1, 7, 14, 28 and 90 days of aging after curing in saturated lime solution at room temperature (22 ± 2 °C) in accordance with BS 8110: part1: 1997 [14]. The evolution of the compressive strength (Rc) as a function of time and the nature of the binder is shown in Fig. 1. The results show that the metakaolin used seems to produce lower compressive strengths compared to the control SCC. On the other hand, as of 28 days, Rc of SCCI + MK (39 MPa) exceeds that of the control SCC (38 MPa) and continues its increase. This can be explained by the pozzolanic reactivity of the MK on the one hand, and by its accelerating effect of the hydration of clinker by nucleation effect on the other hand. For SCCs based on PSC and PPC, the low values of compressive strength at young ages can be explained by the slow hydraulicity and pozzolanicity of slag and fly ash. These results confirm the latent property of hydration of cements containing fly ash and slags.

C. Porosity Accessible to Water

The porosity accessible to water is an essential parameter for the characterization of the durability of concrete. The porosity accessible to water test was carried out according to the procedure recommended by the working group and cited in the GranDuBé project (2007) [15]. Fig. 2 shows the porosity values obtained at 28 and 90 days of curing for the six SCCs studied. It can be seen that, the formulation based on Portland cement type II and metakaolin exhibits the lowest porosity values at 28 days as at 90 days. This is certainly due to the filler effect of the fine particles of metakaolin on the one hand, and to the pozzolanic effect of this addition on the other. The use of metakaolin instead of PSC and PPC leads to an increase in porosity to water at 28 and 90 days of cure. These results are in agreement with the measurement of the compressive strength seen previously. Moreover, it can also be observed that the SCC based on PSC shows a porosity greater than the control concrete SCCI and SCCV at 28 and 90 days.

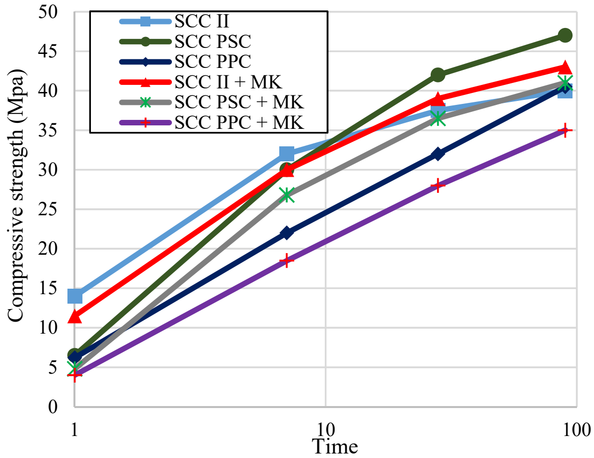

Fig. 1 Evolution of compressive strength, Rc, of SCC mixtures with curing time

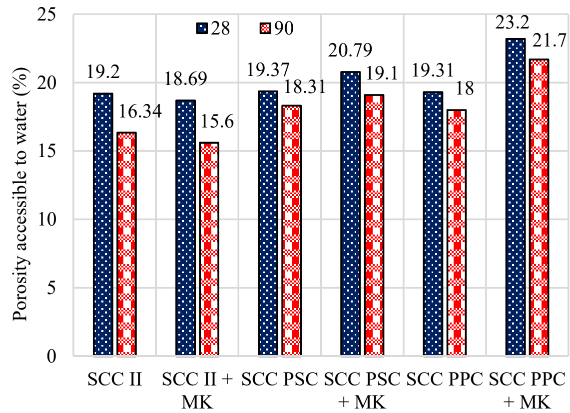

Fig. 2 Porosity accessible to water of the various concretes studied, test carried out after 28 and 90 days of cure

D. Capillary Absorption (Sorptivity)

Capillary water absorption is a term used to describe water ingress into pores of unsaturated concrete due to capillary suction. This test measures the rate of water absorption by capillary suction, brought into contact with water without external hydraulic pressure. In this study, the sorptivity was carried out on cylindrical specimen (Ø 100 × H 50 mm) based on the AFREM procedure. The results are given in Fig. 3. The beneficial effect on reducing capillary absorption compared to the control concrete is observed from 90 days of curing for SCC containing PSC. Moreover, this reduction is more sensitive by incorporating the metakaolin. According to these results, the performance of SCC III + MK in reduction of capillary absorption coefficient in two ages is far better than other mixtures. Nevertheless, at the same ages, the capillary water absorption of Portland cement type II is the highest one. As evident from the results, the capillary water absorption of the SCCs was decreased by incorporating of metakaolin. It was evident that the physical and chemical effects of cement and metakaolin can modify the open channels at the cement paste matrix that leads to a discontinuous pore structure. Besides, it is well known that higher ages can improve the microstructure in interfacial transition zones (ITZs) and result in a considerable decrease in capillary water absorption.

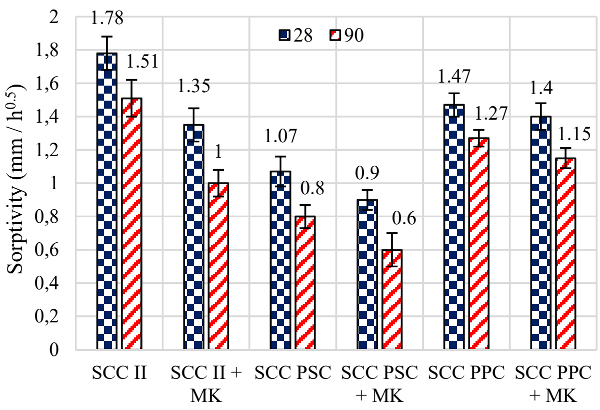

Fig. 3 Results of capillary water absorption test

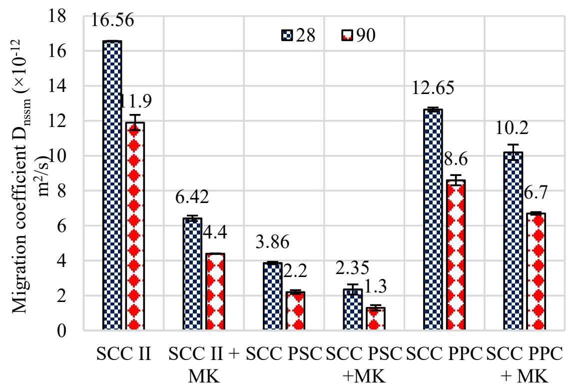

Fig. 4 Migration coefficient of different mixes at 28 and 90 days of aging

E. Migration in Non-Steady-State

After conditioning, the test of migration in non-steady-state was carried out according to NT BUILD 492. Migration coefficients of the different mixes are presented in Fig. 4. This test was appointed for two ages of 28 and 90 days of curing in saturated lime solution on slices of 100×50 mm from cast cylinders. Fig. 5 shows the penetration resistance levels of chloride ions as given in [12]. According to the results illustrated in Fig. 4, the formulations of SCCIII, SCCI+MK, SCCIII+MK showed good resistance against chloride penetration after 28 and 90 days of aging. Moreover, the mixture that contained PPC with 15% of metakaolin also showed good resistance against chloride penetration after 90 days of curing.

- $D < 2\times10^{-12}$ m²/s: Very good resistance against chloride ingress.
- $D < 8\times10^{-12}$ m²/s: Good resistance against chloride ingress.
- $D < 16\times10^{-12}$ m²/s: Moderate resistance against chloride ingress.
- $D > 16\times10^{-12}$ m²/s: Not suitable for aggressive environment.

Fig. 5 Levels of resistance to penetration of chloride ions [12]

The good diffusion was mostly due to the improved pore structure of SCCs. Also, the diffusion results of PSC were less than other cement types, which is due to reduced capillary porosity resulting from continued hydration of the cementing materials. In addition, the migration diffusion results of the SCCs decreased with metakaolin. In other words, in the present study, the lowest level of migration diffusion was achieved for the specimens prepared in the presence of metakaolin, as seen in the results. It suggests that the use of metakaolin improved the quality of SCCs through reduced capillary porosity and densification of their pore structure. As can be seen in SEMs (Fig. 6) at the age of 28 days, the capillary porosity of SCCs decreased with metakaolin content and most of the pores are discontinuous, thus the flow channels for the water movement are reduced. In addition, it can be seen that the microstructure of specimens was improved in the paste matrix. Hence, it was deduced that the metakaolin content improved the durability of SCCs and produced a greater amount of calcium silicate hydrate (C-S-H). The quality of concrete may be evaluated with measuring the passing current by application of 30 V to the concrete sample. This means that the higher the current, the less the resistance of concrete to chloride penetration is. Fig. 7 shows that there is a linear relationship between migration coefficient and initial current. The measurement of initial current seems to be a good durability indicator that can be directly related to the migration coefficient.

Fig. 8 presents a linear relationship between the square root of migration coefficient and chloride penetration depth after 24 h of exposure to migration test. The correlation (R2 = 0.99) suggests that, between these two parameters, the migration coefficient depends more specifically on depth of penetration. Moreover, from NT BUILD 492 standard, varying parameters such as the initial current, the temperature solution and test duration are very effective on the quantity of migration coefficient. Chloride depth penetration after migration test, highlighted by $AgNO_3$ solution, for the different mixtures at age of 28 days is illustrated in Fig. 9.

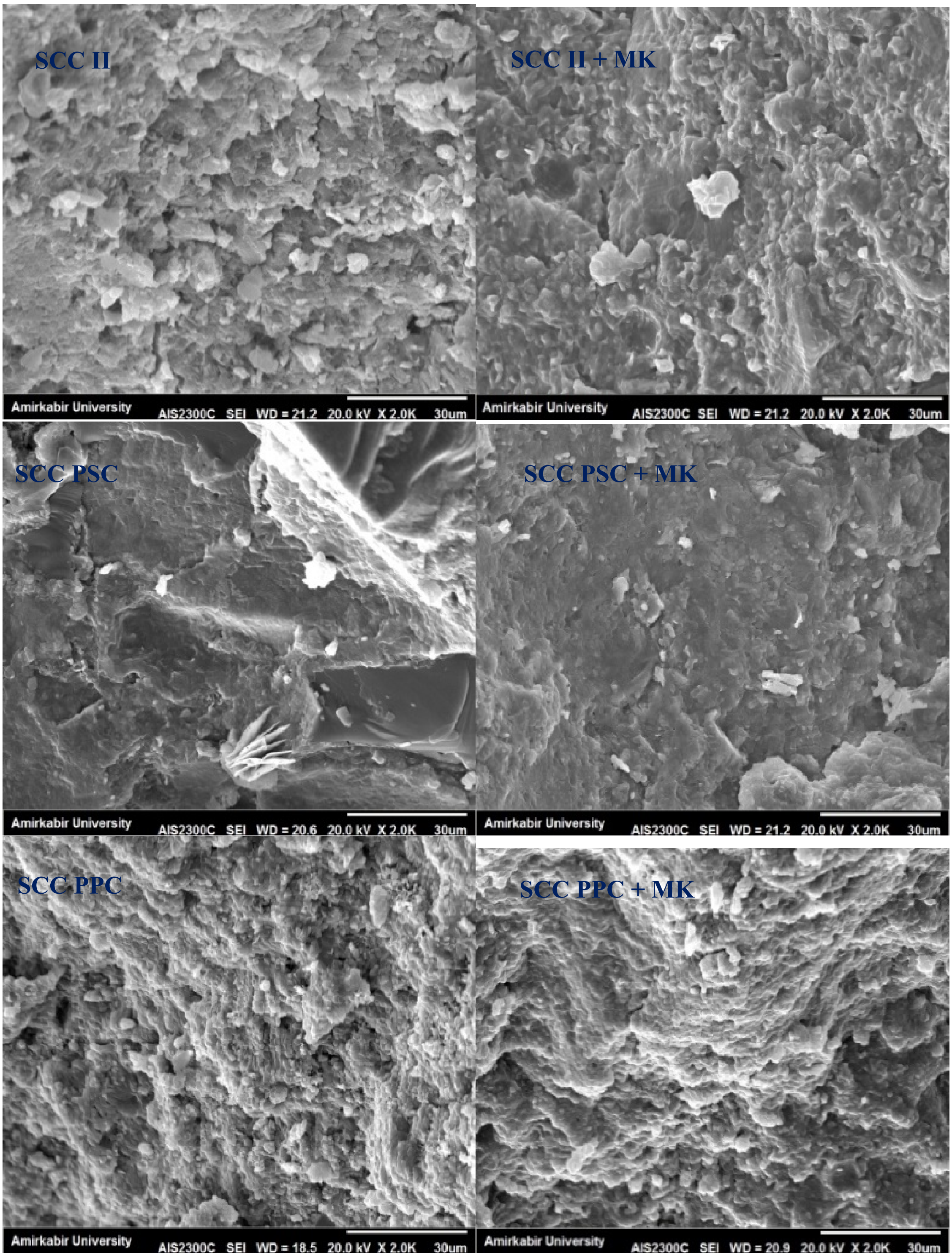

Fig. 6 SEM images of SCCs at age of 90 days

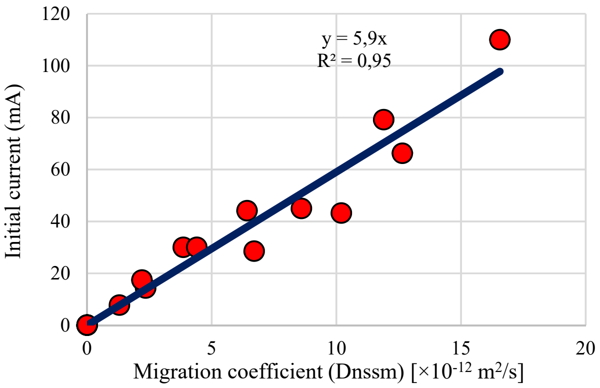

Fig. 7 Evolution of initial current according to migration coefficient of the studied SCCs

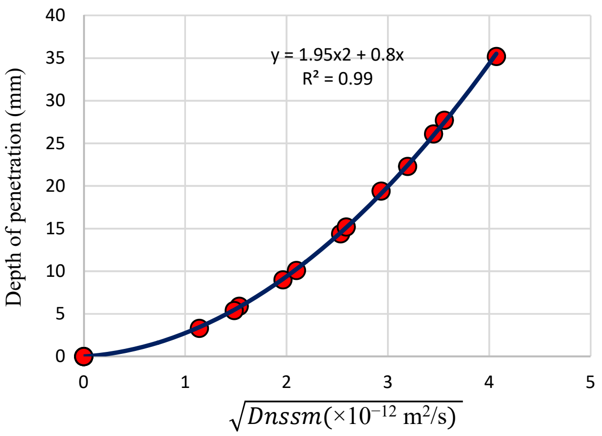

Fig. 8 Evolution of chloride penetration depth according to migration coefficient of the studied SCCs

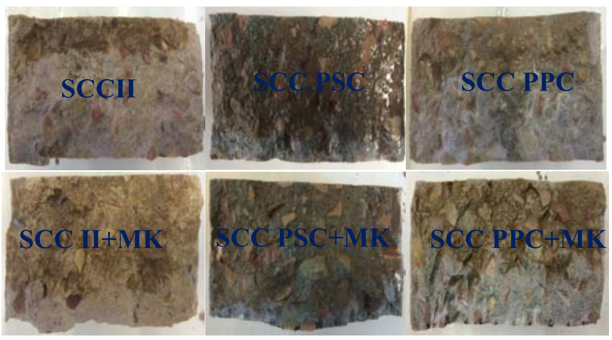

Fig. 9 Chloride penetration depth after migration test in the different studied SCCs at 28 days of aging

IV. Conclusions

Based on the obtained results from this study the following conclusions can be drawn:

1) Metakaolin can have rheological effects on the workability of SCCs. The performance of SCCs mixture decreased according to 15% metakaolin content.
2) Due to improved paste densification resulting from greater hydration products, the compressive strength of SCCs was improved by increasing the curing time. In addition, the highest value of compressive strength was achieved for PSC.
3) The samples containing PSC or PPC with metakaolin had a lower compressive strength than plain samples. Therefore, it can be concluded that metakaolin has a negative effect on the compressive strength in SCC containing PSC or PPC.
4) The replacement of 15% Portland cement type II by metakaolin results in a decrease in the porosity accessible to water. On the other hand, the use of metakaolin instead of PSC and PPC leads to an increase in porosity to water at 28 and 90 days of cure. Moreover, it can also be observed that the SCC based on PSC shows a porosity greater than the control concrete SCCI and SCCV at 28 and 90 days.
5) Capillary water absorption test indicates metakaolin and cement types improve the durability of concrete. This phenomenon indicates that adding 15% of metakaolin had a notable effect on decreasing the conductivity of concrete and on refining the pore structure.
6) The diffusion results of PSC were less than other cement types, which is due to advanced hydration of the cementing materials. In addition, the diffusion results of the SCCs decreased with metakaolin.
7) Taking into account all the results, the use of PSC and PPC as total replacement instead of Portland cement type II and also the partial substitution of various cement with 15% of metakaolin is affordable for all aspects including economic, environmental and durability issues.

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