

The Influence of Ultrafine Filler Materials on Mechanical and Durability Characteristics of Concrete

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ABSTRACT: This paper aims at investigating the influence of partial replacement of Portland cement with ultrafine fillers compared to limestone powder on the durability and mechanical characteristics of concrete. For this purpose, ultrafine calcium carbonate materials with different specific surface areas of 9.7 and 15.1 m²/g and limestone powder (0.72 m²/g) were used. The results indicate that the ultrafine fillers enhanced the durability properties compared to the mixtures containing limestone. For instance, ultrafine fillers provided up to 20% higher concrete surface resistivity and 20% lower rapid chloride migration test (RCMT) coefficient, while limestone mixes showed equal or even lower durability levels compared to the plain mixture with 0% supplementary cementitious materials. The influence of filler materials on the compressive strength was not significant. However, regardless of the surface area, incorporating filler materials at levels up to 5% led to somewhat higher compressive strengths compared to the plain mixture. Generally, it seems that using finer fillers may lead to more durable concrete mixes.

Keywords: Concrete Mixture, Durability, Limestone Powder, Strength, Ultrafine Filler.

INTRODUCTION

In recent years, a considerable research effort has been attracted to the investigation of the influence of supplementary cementitious materials on the characteristics of cement based materials (Ramezaniapour, 2013; Tarighat and Jalaifar, 2014; Dadash

and Ramezaniapour, 2014). In this regard, several mechanisms have been proposed. Among them, some investigations suggest that the supplementary materials have physical effect (filler effect) and could enhance the packing density of cement composites. This effect has been indicated to be more pronounced for the materials with

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finer particles such as silica fume and Nanomaterials (Madani et al., 2012). For instance, in ACI 234 it has been suggested that the reduced permeability and diffusivity of silica fume concrete is a result of pore refinement caused by both pozzolanic reactivity and filler effect of silica fume particles. Several types of supplementary materials are in the form of non-pozzolanic types. This study was thus conducted to investigate the influence of inert filler materials, especially those containing ultrafine particles on the mechanical and durability characteristics of cement composites.

In this study, the limestone powder was investigated as a common inert supplementary cementitious material. The influence of this material on the properties of cement composites has been the subject of several studies. Celik et al. (2014) reported that replacing 15% cement with limestone produced lower strength at early and late ages. Tsivilis et al. (2002) reported that limestone Portland cement concrete had less water demand compared to the Portland cement concrete. Lawrence et al. (2005) suggested that at constant strength, a concrete containing limestone powder could exhibit similar carbonation rate, chloride penetration, and freeze-thaw resistance characteristics to a concrete containing type-1 cement (with or without air entrainment). Vuk et al. (2001) indicated that the influence of limestone addition depended more on clinker type than other properties such as fineness. Meddah et al. (2014) reported that increasing the level of replacing limestone powder with cement led to a reduction in compressive strength, flexural strength, and modulus of elasticity at early and late ages. This drop in strength was more pronounced at replacement levels exceeding 15%.

As limestone powder is a Calcium Carbonate compound, two types of synthetic Calcium Carbonate materials with surface

areas of 9.7 and 15.1 m²/g were investigated in this study. It should be mentioned that very limited studies are available regarding the influence of ultrafine filler materials on the durability and mechanical characteristics of concrete. For instance, Kawashima et al. (2013) reported that ultrafine calcium carbonate material could accelerate the rate of cement hydration, setting time, and strength gain at early ages. The results of another study (Camiletti et al., 2012) indicated that ultrafine calcium carbonate materials may act as filler materials, creating a denser microstructure and at Nano dimensions these materials could accelerate the cement hydration through providing nucleation centers. Qian et al. (2009) studied the penetration of chloride ion of concretes containing fly ash modified with ultrafine calcium carbonate materials. The results showed a higher resistance of concrete against chloride penetration by addition of nano-CaCO₃. The reduction in the penetration of chloride ion, at age of 56 days, was up to 54% for mixes containing nano-CaCO₃ with different fly ash replacement levels.

It is believed that ultrafine materials could significantly enhance the engineering properties of cement based materials. This study was thus focused on the influence of ultrafine calcium carbonate materials on the properties of concrete mixtures. Comparing the influence of ultrafine fillers with conventional filler materials such as limestone powder could provide insights about the influence of the degree of fineness of supplementary materials on the characteristics of cement composites.

MATERIALS AND METHODS

Material

In the present study, natural sand with maximum grain size of 4.75 mm and fineness module of 3.64, was used. Two

types of crushed gravel were also utilized including fine and coarse gravel. The grain size of the fine gravel was between 4.75 and 12.7 mm, and the coarser one had aggregates with dimensions from 9.5mm to 19 mm. Water absorption of sand, fine aggregate, and coarse aggregate was 2.9%, 1.96% and 2.19%, respectively and their specific weights were 2.51, 2.57 and 2.56 g/cm³, respectively. The mass proportion of sand: fine aggregates: coarse aggregate was adjusted to 2.42: 1.37: 1. The grading curve of aggregates is presented in Figure 1. The grading curve was the Funk and Dinger grading curve with the exponent of 0.5 according to the following equation.

$$p(d) = \frac{d^q - d_{min}^q}{d_{max}^q - d_{min}^q} \quad (1)$$

where P : is the fraction that can pass through a sieve with opening diameter of d ; d_{max} : is the maximum particle size of aggregates and d_{min} : is minimum particle diameter for gradation curve.

Sealed plastic bags were used to retain moisture in the aggregates. The aggregate moisture contents were obtained by heating the samples on the Hot Plate apparatus

according to the standard test method of ASTM C 566 in order to determine the free water content of the mixtures, considering the SSD moisture. This test method covers the determination of the percentage of evaporable moisture in a sample of aggregate by drying both surface moisture and moisture in the pores of the aggregate.

The cement used was Type-II Cement with a blain surface area of 0.315 m²/g and a specific weight of 3140 kg/m³. The chemical composition of cement is given in Table 1. The limestone filler used in this study was complied with the requirements of EN 197-1 with specific surface area of 0.72 m²/g. The superplasticizer used in this study was a Polycarboxylate ether based superplasticizer.

In the current study, two types of ultrafine calcium carbonates with relatively low and high specific surface areas of 9.7 m²/g (LCC) and 15.1 m²/g (HCC) were used as ultrafine filler materials. The surface areas of supplementary cementitious materials were measured using nitrogen BET method and were significantly different from the natural limestone powder (0.72 m²/g). The properties of these materials are presented in Table 2.

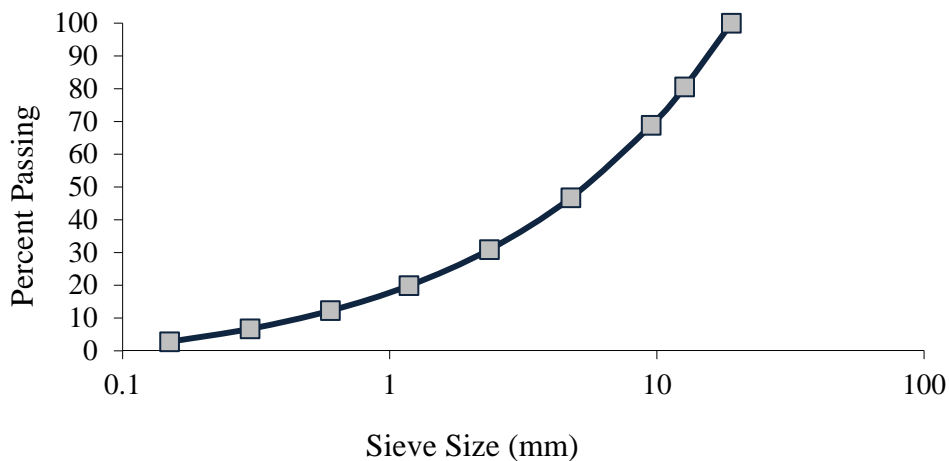


Fig. 1. The grading curve of aggregates

Table 1. Chemical analysis of Type-II Portland cement

Na ₂ O	K ₂ O	SO ₃	MgO	CaO	Al ₂ O ₃	Fe ₂ O ₃	SiO ₂	C ₂ S	C ₃ S	C ₃ A	C ₄ AF
0.28	0.59	2.0	3.57	62.21	4.6	3.39	21.46	24	49	6.2	10.3

Table 2. Physical properties of LCC and HCC

	LCC	HCC
Composition	Calcium carbonate	Calcium carbonate
Appearance	White solid	White solid
Crystalline Structure	Calcite and aragonite	Calcite and aragonite
Density	2700 kg/m ³	2900 kg/m ³
Specific Surface Area	9.7 m ² /g	15.1 m ² /g

Mixture Proportion and Specimen Preparation

The concrete mixtures were made at a constant w/cm ratio of 0.45 and cementitious material content of 400 kg/m³. The replacement levels of cement with each material were 2.5, 5, 10, and 15 percent. Superplasticizer content of the mixtures was adjusted to keep the slump of the mixes within the range of 10±1 cm. The mixture proportions are presented in the table 3. To assure the repeatability and reproducibility of the experiments, the plain mixture was prepared three times: at the start, in the middle, and at the end of the concrete mixtures routine.

In order to determine the compressive strength, cubic specimens with dimension of 100 mm were molded. The concrete was poured in two layers and each layer was compacted with a vibrating table for 30 seconds.

For the electrical resistance, water absorption, and RCMT tests, the cylindrical

specimens with diameter of 10 cm and height of 20 cm were cast in three layers, and each layer was separately vibrated upon placement (Figure 2).

After molding, the samples were kept under wet cloth for 24 hours. Then, they were cured in saturated limewater at 23±1 °C until the time of testing. The electrical resistivity test was applied on 20 cm height concrete cylinders. Afterwards, 5 cm from the top of each cylindrical specimen was cut and removed, then two smaller cylinders with 5cm height were cut and utilized to perform the water absorption test and RCMT, respectively. It should be mentioned that testing of concrete specimens from top and bottom sections of the cylindrical specimens could not provide accurate results. Thus, in the current study, the top and bottom sections were not used to test the durability characteristics. In other words, the cylindrical specimens were obtained from the middle section of the cylindrical specimens.

Table 3. The mixtures proportions

Mixture	Water (kg/m³)	Coarse Gravel (kg/m³)	Fine Gravel (kg/m³)	Sand (kg/m³)	Cement Replacement Material (kg/m³)	Cement (kg/m³)
Plain mixture	180	353.3	486.02	855.13	---	400
2.5 LCC	180	353.2	485.9	854.9	LCC (10)	390
5 LCC	180	353.1	485.8	854.8	LCC (20)	380
10 LCC	180	353.0	485.6	854.4	LCC (40)	360
15 LCC	180	352.9	485.4	854.1	LCC (60)	340
2.5 HCC	180	353.2	486.0	855.0	HCC (10)	390
5 HCC	180	353.2	485.9	854.9	HCC (20)	380
10 HCC	180	353.1	485.8	854.7	HCC (40)	360
15 HCC	180	353.0	485.7	854.6	HCC (60)	340
2.5 LS	180	353.3	486.0	855.1	Limestone (10)	390
5 LS	180	353.3	486.0	855.0	Limestone (20)	380
10 LS	180	353.2	485.9	855.0	Limestone (40)	360
15 LS	180	353.2	485.9	854.9	Limestone (60)	340

The mixing procedure was as follows: First, the dry ingredients including cement, the CaCO_3 material (LLC, HCC or LS) and aggregates were mixed in a 35rpm mixer for 1.5 minutes. Then, water was gradually added to other ingredients within one minute during the mixing. Afterwards, the superplasticizer was added to adjust the workability of concrete during two minutes. Finally, the 35 rpm mixing was continued for another three minutes. It should be mentioned that as the superplasticizer content was not specific, this material was added gradually to the mixture.

Testing Procedure

Concrete cubes of $100 \times 100 \times 100$ mm dimension were cast for compressive strength. They were tested at 7, 28 and 90

days after mixing. The electrical resistivity (surface resistivity) of cylindrical specimens were measured in ohm-meter at the ages 14, 28 and 90 days. The test was performed according to the Wenner method (AASHTO TP 95, 2011). The samples were subjected to the rapid chloride migration test (RCMT) at ages of 28 and 90 days in accordance with the Nordtest method (NT Build 492, 1999). The accelerated migration coefficient of chloride ion was determined in terms of the applied voltage (V), average value of the initial and final temperatures in the Anolyte solution ($^{\circ}\text{C}$), thickness of the specimen (mm), average value of the penetration depths (mm) and test duration (hour). Figure 3 shows the color change in the samples due to silver nitrate spraying.

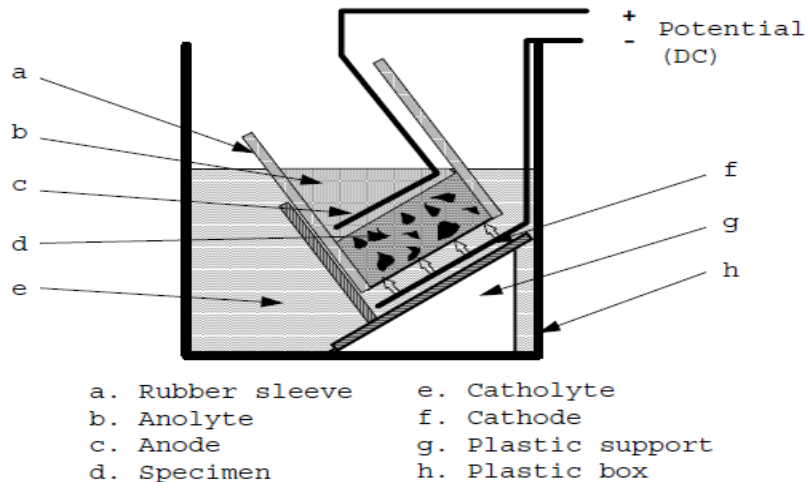


Fig. 2. Schematic arrangement of RCMT (NT Build 492, 1999)



Fig. 3. Color change of concrete sample section after silver nitrate spraying

The water absorption test was conducted as per ASTM C 642 in order to determine the change in resistance towards water penetration in concrete. The cylindrical molds of 100 mm diameter and 50 mm height were prepared and cured for 28 days. After curing, the specimens were oven dried at 110 °C, establishing a mass equilibrium of less than 0.2% between two measurements at 24 h intervals. Then the specimens were immersed in water at approximately 23 °C for 72 h and saturated surface-dry mass was calculated. The absorption after immersion was calculated according the following relationship:

$$\text{Absorption after immersion \%} = \frac{[(B-A)/A] \times 100}{(2)}$$

where *A*: is the mass of the oven dried sample in air and *B*: is the saturated surface-dry mass.

RESULTS AND DISCUSSION

Compressive Strength Test Results

The compressive strength test results at different ages are given in the Figures 4-6. As indicated in the figures, each filler material with content up to 5% caused an

increase in the compressive strength of the concrete mixes. The mixtures containing cement replacement materials with higher surface areas outperformed those with finer particles. In this respect, the maximum strength enhancements were about 8.5%, 5.3% and 3.9% for the mixtures incorporating 5% HCC, LCC and LS mixes, respectively. This increase may be due to occupying the spaces between cement grains and consequently higher packing density of the mixtures (De Larrard, 1999).

At substitution levels higher than 5%, lower compressive strengths were observed which could be due to lower cement content of the mixtures. The strength values of 15 LCC, 10 LS and 15 LS mixes were even slightly lower than the plain mix.

In a general view, the ultrafine fillers at substitution level up to 5% enhanced slightly the compressive strength of concrete. Thus, the surface area of the calcium carbonate seems to be not a significant factor on the mechanical behavior of concrete.

Bederina et al. (2011) also reported that adding limestone fillers improved the mechanical strength of concrete. They also mentioned that the best values of mechanical strength were obtained by using an optimum content of fillers which was about 5%.

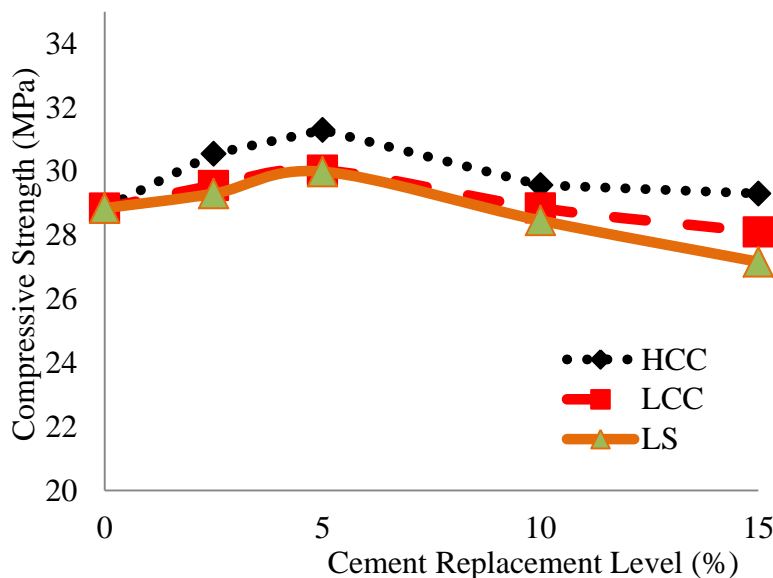


Fig. 4. Compressive strength test results at 7 days

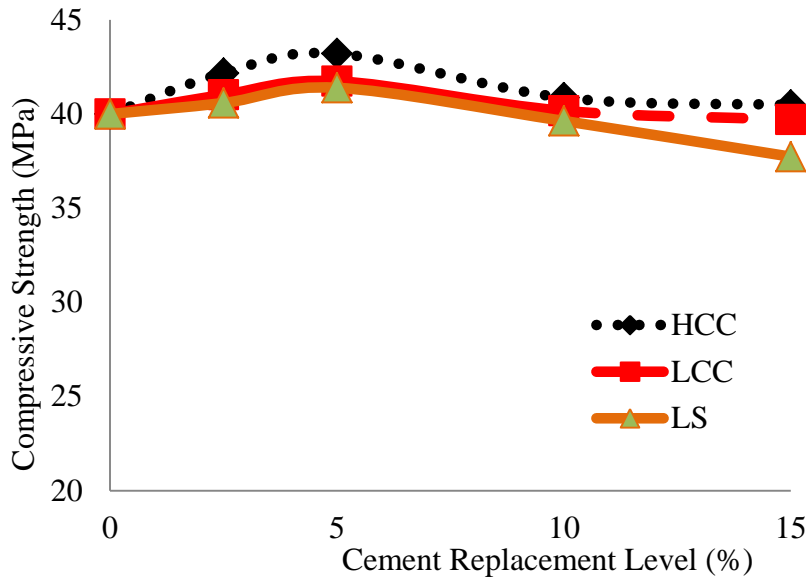


Fig. 5. Compressive strength test results at 28 days

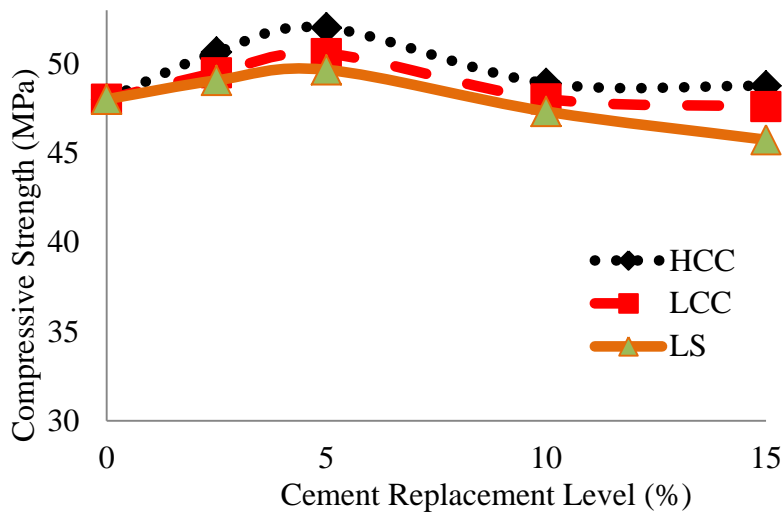


Fig. 6. Compressive strength test results at 90 days

Electrical Resistivity Test Results

Electrical resistivity is a decisive factor in controlling the rate of steel corrosion in concrete (Gjorv, 2014, Dyer 2014). The higher the electrical resistivity of concrete, the lower the rate of corrosion in rebars will be (Madani et al., 2014). The results obtained from the electrical resistivity test (performed according to the Wenner method) are shown in Figures 7-9.

As can be observed in Figure 6, at the age of 14 days, the electrical resistivity of the mixtures was not significantly different from

that of the plain mixture. The surface resistivity of the mixtures containing limestone powder was almost constant compared to the plain mixture. At replacement levels up to 5%, the mixtures containing LCC showed an increase in electrical resistivity of about 10% which this enhancement was diminished at higher substitution levels and similar electrical resistances were obtained. In the mixtures containing HCC, maximum electrical resistivity was obtained at 10% replacement level.

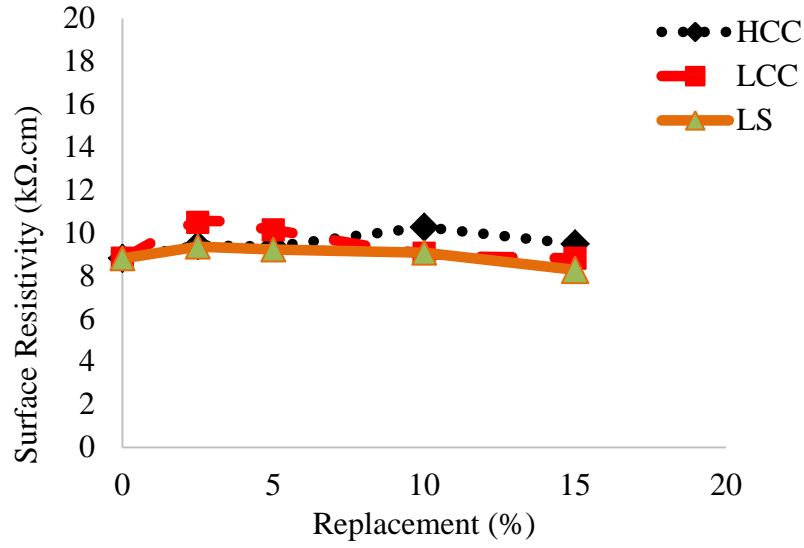


Fig. 7. Electrical resistivity test results at 14 days

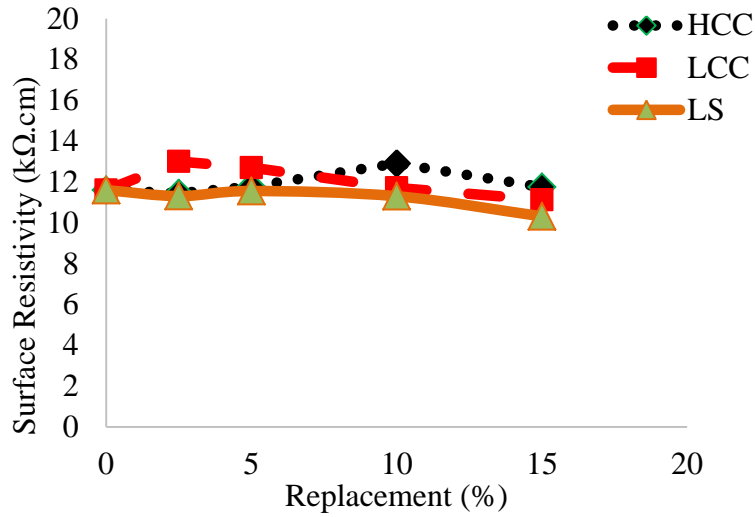


Fig. 8. Electrical resistivity test results at 28 days

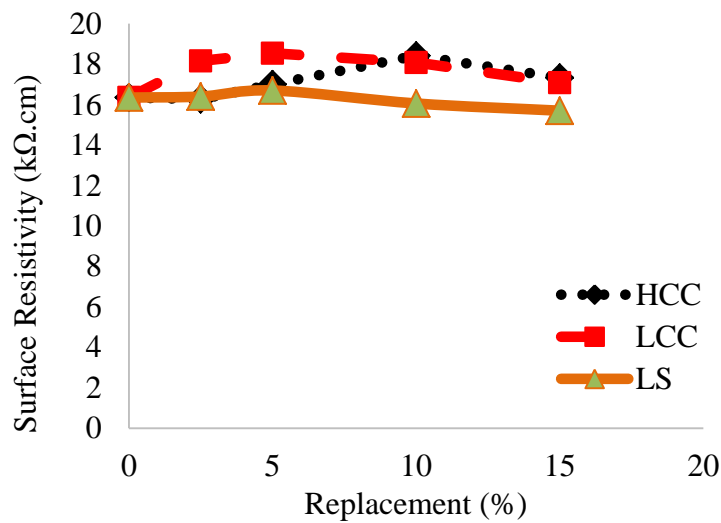


Fig. 9. Electrical resistivity test results at 90 days

At 28 days, the mixture containing limestone powder at different replacement levels had similar electrical resistivity compared to the plain mixture. Upon substitution of 2.5-5 percent LCC, the electrical resistivity was enhanced between 10-15 percent, and as this substitution level increased, the electrical resistivity of the mixture reached that of the plain mixture. At the substitution level of 10%, incorporation of HCC also increased the relative electrical resistivity of the mixture by about 11% compared to the plain mixture. At other substitution levels, the mixtures with HCC had similar electrical resistivity with the plain mixture.

At 90 days, the electrical resistivity of the mixtures containing limestone powder was almost equal to the plain mixture, and the maximum electrical resistivity enhancement of about 3% occurred at a 5% substitution level. A 5% LCC substitution level led to about 16% increase in the electrical resistivity of the mixture. Despite the decreasing trend of the electrical resistivity by increasing the substitution level of LCC, the surface resistivity was still 5% higher compared to the plain mixture. Substitution of cement with HCC also enhanced the electrical resistivity. In this regard, at a substitution level of 10%, a peak with 18% increase was observed in the electrical resistivity, also, at other incorporation levels, the electrical resistivity was still higher than the plain mixture.

Rapid Chloride Migration Test Results

The RCMT coefficients were determined according to the Nordtest method (NT Build 492, 1999) at ages of 28 and 90 days are shown in Figure 10. As can be observed, rapid chloride migration coefficient was reduced in the mixtures containing up to 10% of LCC at both 28 and 90 days. However, at higher substitution level, the

chloride migration coefficient was increased though it was still lower than the plain mixture. The maximum reduction in the RCMT coefficient of LCC mixtures compared to the plain concrete was 16% at 28 days and 17 % at 90 days, which was observed at 10% substitution level.

The HCC mixtures exhibited a relatively similar trend with LCC. At 28 days and substitution level of 5%, a maximum penetration drop of 15% was observed. However, as the incorporation level was increased, chloride penetration was increased and at a 15% substitution, similar migration coefficient compared to the plain mixture was obtained. The same trend was dominant at the age of 90 days, and chloride penetration was decreased by about 20% at 5% substitution.

In the mixtures containing limestone powder, chloride penetration was unchanged at lower substitution percentages (up to 5%) at 28 and 90 days. However, at greater substitution levels, the chloride migration coefficient was increased. At 28 days, the mixtures with 10% and 15% limestone had 8% and 12% higher RCMT coefficient compared to the plain concrete, respectively. Also, at the age of 90 days, RCMT coefficient in the mixture containing 15% LS became 6% higher than the plain mixture.

The optimum substitution level for limestone powder to obtain an acceptable chloride ion penetration was 2.5-10 percent which led to similar or slightly lower chloride migration coefficient compared to the plain mixture. At 15% replacement level, the RCMT coefficient was somewhat higher than the plain mixture at 90 days, so the appropriate level could be 0-15%. Ramezani-pour et al. (2009) also reported a 10% limestone powder substitution as the optimum percentage.

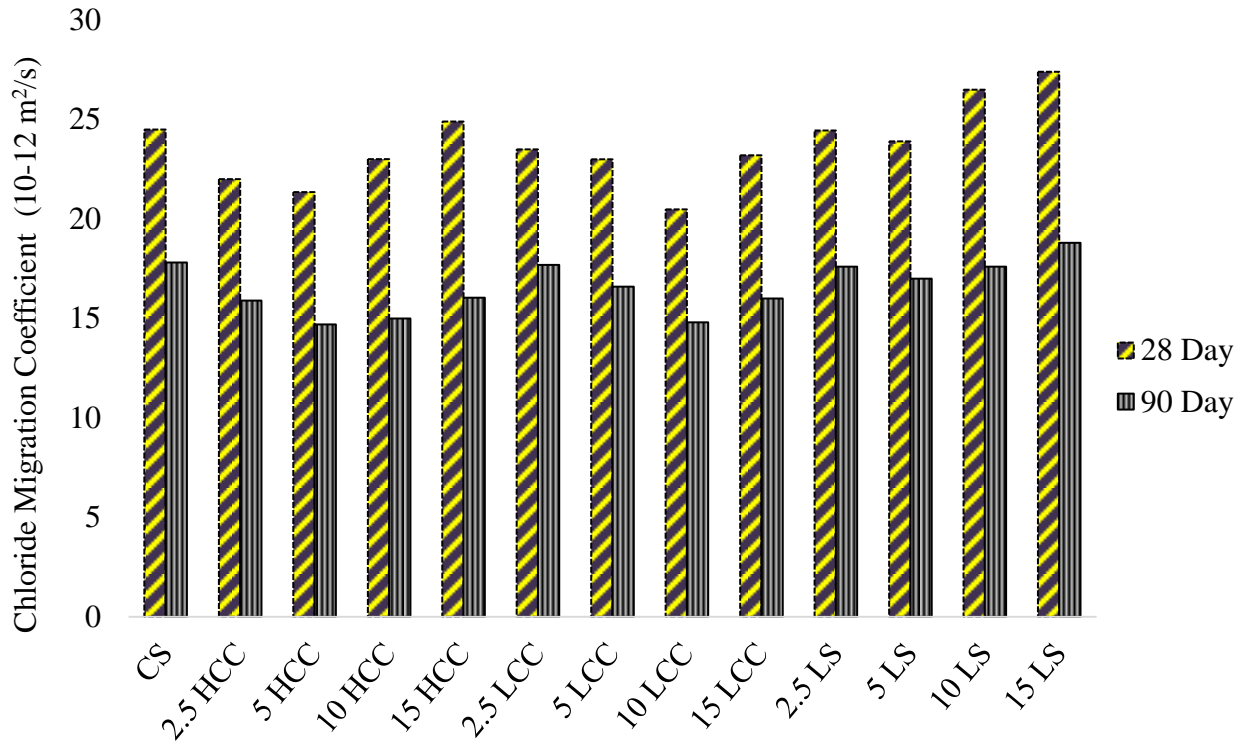


Fig. 10. Rapid chloride migration test results

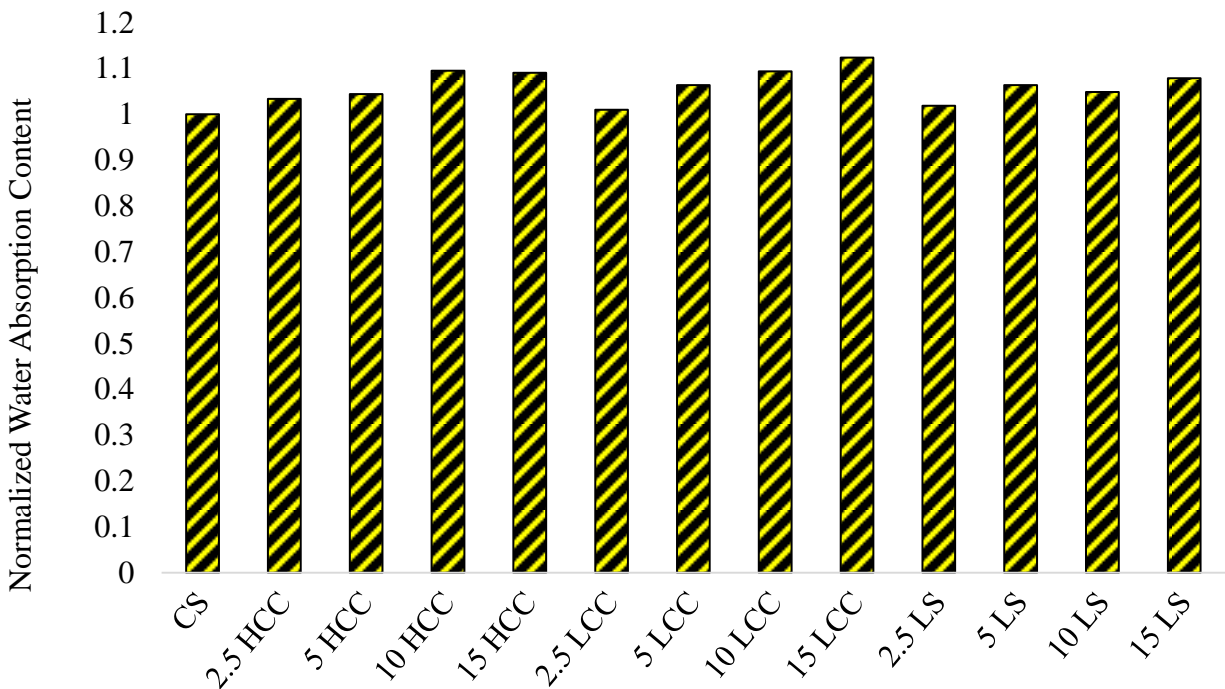


Fig. 11. Water absorption of mixtures

Water Absorption Test Results

In this study, the plain mixture had a water absorption coefficient of about 6.7%

which was lower compared to the mixtures containing supplementary cementitious materials. As shown in Figure 11, the higher

water absorption contents were obtained for the mixtures with higher replacement levels. For instance, the maximum increase in water absorption contents was about 10% for the mixtures containing supplementary materials. The results indicate that increasing the level of cement replacement leads to higher level of water absorption content, however the surface area of the filler materials was not a significant factor.

The durability characteristics are significantly attributed to the microstructure of the pores. In this regard, a tortuous and disconnected microstructure could result in a lower diffusivity and aggressive Ions penetration in concrete. Thus, a concrete with a tortuous porosity may have higher permeable pore volume compared to a concrete with lower permeable voids. The total pore volume of concrete is attributed to the level of cement hydration and water/cementitious materials ratio.

In the current study, the cementitious materials content and free water level were kept constant in all the mixtures. In cement hydration a portion of water is consumed to produce mainly CSH and calcium hydroxide. The mixtures with lower cement content consume lower water in hydration process, thus these mixtures have higher permeable pore volume and water absorption. However, due to ultrafine dimensions of particles of the investigated materials, the mixtures with these materials have improved microstructure. In other word the ultrafine particles, particularly at optimum levels could enhance the tortuously of the microstructure. Consequently, the RCMT coefficient and electrical resistivity are lower for the mixtures with ultrafine particles at optimum levels.

CONCLUSIONS

In this study the influence of particle size (specific surface area) of inert cement

replacement materials on the mechanical and durability properties of concrete was investigated. The results indicate that:

- (A) The materials, investigated in this study, had an optimum substitution level of 5% in improving the mechanical characteristics. Thus, it appears that the filler effect of these materials on enhancing the mechanical properties is more pronounced at substitution levels up to 5%. In addition, the surface area of the calcium carbonate fillers materials was not a significant factor on compressive strength test results.
- (B) The electrical resistivity of the mixtures containing limestone powder was nearly equal to that of the plain mixture at ages of 14, 28 and 90 days. Substitution of ultrafine calcium carbonate filler with lower surface area (LCC) at low replacement levels enhanced the electrical resistivity and as this substitution percentage went up, the electrical resistivity of the mixtures reached that of the plain mixture. Addition of HCC also increased the relative resistance of the mixtures at ages of 14, 28 and 90 days up to 18% (compared with the plain mixture).
- (C) Low content of limestone powder did not significantly change the chloride migration coefficient. From this point of view, up to 10% substitution provides a limestone concrete mix competitive to the normal mixes. Up to 10% substitution, LCC and HCC decreased chloride ion migration. However, at greater incorporation levels, the RCMT coefficient was similar to the plain mixture.
- (D) Substitution of cement with LCC, HCC and LS generally increased the water absorption of concrete.
- (E) To sum up, it appears that ultrafine inert materials could enhance the durability characteristics, especially at low

substitution levels compared to those with lower surface areas. However, these materials had not significant influence on the mechanical properties of concrete.

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