



Influence of various amounts of limestone powder on performance of Portland limestone cement concretes

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ABSTRACT

The benefits of limestone as a partial replacement for Portland Cement (PC) are well established. Economic and environmental advantages by reducing CO₂ emissions are well known. The paper describes the effect of various amounts of limestone on compressive strength, water penetration, sorptivity, electrical resistivity and rapid chloride permeability on concretes produced by using a combination of PC and limestone at 28, 90 and 180 days. The percentages of limestone that replace PC in this research are 0%, 5%, 10%, 15% and 20% by mass. The water/(clinker + limestone) or (w/b) ratios are 0.37, 0.45 and 0.55 having a constant total binder content of 350 kg/m³. Generally, results show that the Portland limestone cement (PLC) concretes having up to 10% limestone provide competitive properties with PC concretes.

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1. Introduction

Concrete is a material used in many civil engineering projects. The majority of the cementitious binder used in concrete is based on Portland cement clinker, the manufacture of which is an energy-intensive process. In addition, it produces a large amount of greenhouse gas emissions, mostly CO₂, resulting from release of CO₂ from limestone in the pyro-processing of clinker. On the other hand, the concrete industry is one of the major consumers of natural resources. In order to reduce energy consumption, CO₂ emission and increase production, cement plants produce blended cements, comprised of supplementary cementitious materials such as slag, natural pozzolan, fly ash and limestone.

Before the 1980s, limestone was assumed as an inert filler. However, the results of recent studies show that calcium carboaluminate hydrates precipitate during the hydration of Portland Limestone Cement (PLC). In addition, there is an interaction between tricalcium silicate (C₃S) and calcium carbonate (CaCO₃); the latter accelerates the hydration of C₃S and modifies the Ca/Si ratio of C–S–H [1]. It is worth noting that limestone does not show pozzolanic properties and, consequently, does not produce C–S–H gel [2]. When limestone is used in cementitious materials, changes in the capillary porosity occur due to several physical effects such as,

dilution effect, filler effect and heterogeneous nucleation [3–5]. The increase in the amount of limestone involves a decrease in the amount of cement and consequently, an increase in the effective w/c ratio. The filler effect implies a modification in the initial porosity of the mix, and it generally produces a decrease in the water required to maintain constant workability. Heterogeneous nucleation occurs because limestone particles act as nucleation sites, increasing the early hydration of cement and, therefore, producing a more disoriented crystallization of CH [6].

Most Portland cement specifications allow the use of limestone up to 5%. Beyond that, Portland limestone cements are categorized based on the percentage of limestone added to the cement. Portland limestone cements consisting of limestone from 5% up to 35% are being produced and used in various countries around the world.

The European Standard (EN 197-1-2000) has permitted up to 5% limestone as a minor additional constituent and also identifies four types of PLC containing 6–20% limestone (types II/A-L and II/A-LL) and 21–35% limestone (types II/B-L and II/B-LL), respectively [3]. The ASTM Standard (C150-04) allows up to 5% of limestone. The calcium carbonate (CaCO₃) content calculated from the calcium oxide content should be at least 70% by mass, so that the requirements of this standard are met [7].

Since 1983, the Canadian standard has allowed the inclusion of up to 5% limestone addition for types 10 and 30 Portland cements. There is a similar trend in Latin American countries such as Argentina, Brazil, and Mexico [6].

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For instance, the Institute of Standards and Industrial Research of Iran (ISIRI 4220-2005) allows the inclusion of 6–20% limestone for PLC [8].

In this paper, the effect of various amounts of limestone on compressive strength, water penetration, sorptivity, rapid chloride permeability test (RCPT) and electrical resistivity in concretes produced by a combination of PC and limestone at 28, 90 and 180 days are investigated.

2. Experimental program

2.1. Material

ASTM C 150 type I Portland cement was used in all the concrete mixtures. Chemical and physical characteristics of clinker and limestone are shown in Table 1. The limestone should meet three requirements:

(a) CaCO_3 content greater than 75%; (b) clay content, determined with Methylene blue test (MBA), less than 1.20 g/100 g; and (c) the total organic carbon (TOC) content shall conform to one of the following criteria:

- LL: shall not exceed 0.20% by mass.
- L: shall not exceed 0.50% by mass [9].

The used limestone satisfied all these requirements.

For all mix designs, coarse aggregates were crushed calcareous stone with a maximum size of 19 mm and fine aggregate was natural sand. The coarse aggregates have a specific gravity and a water absorption of 2510 kg/m³ and 1.90%, respectively, and the fine aggregate has a water absorption of 2.75% and a specific gravity of 2570 kg/m³. The gradings of the coarse and fine aggregates according to the BS 882 Standard [10] are presented in Fig. 1. The naphthalene-based superplasticizer, Rheobuild, with a specific gravity of 1200 kg/m³, was employed to achieve the desired workability in specimens having w/b = 0.37.

Table 1
Chemical and physical characteristics of clinker and limestone.

	Clinker	Limestone
<i>Chemical components</i>		
Calcium oxide (CaO) (%)	61.50	54.77
Silicon dioxide (SiO ₂) (%)	21.50	1.47
Magnesium oxide (MgO) (%)	4.80	0.30
Aluminium oxide (Al ₂ O ₃) (%)	3.68	0.22
Ferric oxide (Fe ₂ O ₃) (%)	2.76	0.29
Sulphate oxide (SO ₃) (%)	2.50	0.06
Potassium oxide (K ₂ O) (%)	0.95	0.08
Sodium oxide (Na ₂ O) (%)	0.12	0.05
Titanium oxide (TiO ₂) (%)	0.04	0.05
Phosphorus oxide (P ₂ O ₅) (%)	0.23	0.01
LOI (%)	1.35	42.23
<i>Mineralogical composition^a</i>		
C ₃ S (%)	51	–
C ₂ S (%)	23	–
C ₃ A (%)	5	–
C ₄ AF (%)	8	–
<i>Chemical characteristics</i>		
CaCO ₃ content (%)	–	97.8
Total organic carbon (TOC) (%)	–	0.05
Methylene blue adsorption (MBA) (gr/100 gr)	–	0.07
Moisture content (%)	–	0.37
<i>Physical characteristics</i>		
Blaine (m ² /kg)	320	330
Specific gravity	3.21	2.69

^a According to ASTM C150–04.

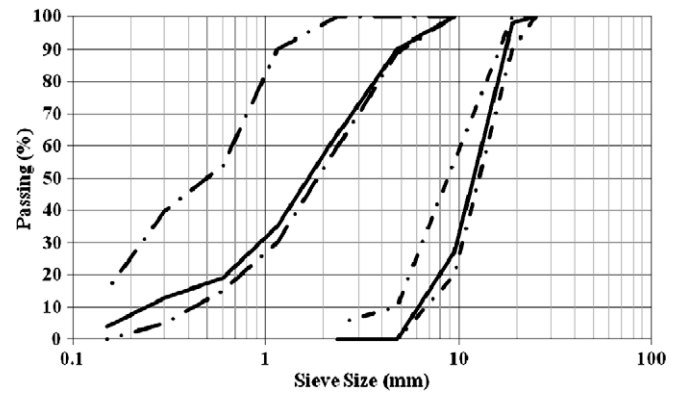


Fig. 1. The grading of the coarse and fine aggregate.

2.2. Mixture proportion

The concrete production was carried out in a mixer of 60 liters capacity. The percentages of limestone that replace PC in this research are 0%, 5%, 10%, 15% and 20% that were added to clinker in the laboratory. All replacements were made by mass. The water: binder (w/b) ratios were 0.37, 0.45 and 0.55 having a constant total binder (cement + limestone) content of 350 kg/m³. Slump was kept constant at 9 ± 2 cm. The mixture proportions for concrete specimens are summarized in Table 2.

2.3. Testing procedure and specimen preparation

Concrete test specimens were vibrated over a vibrating table to remove entrapped air. After casting, the concrete specimens were covered with a wet towel for 24 h and cured under laboratory conditions. Then, they were demolded and cured in lime-saturated water at 23 ± 2 °C to prevent possible leaching of Ca (OH)₂ from these specimens.

Concrete cubes of 100 × 100 × 100 mm dimension were cast for compressive strength. They were tested for compressive strength after 3, 7, 28, 90 and 180 days of water curing. The water penetration test, which is most commonly used to evaluate the permeability of concrete, is the one specified by BS EN-12390-8:2000 [11]. In this test, 150 mm concrete cubes, after 28, 90 and 180 days of water curing, were dried under laboratory conditions for 24 h. Water was applied on one face of the specimen under a pressure of 0.5 MPa. This pressure was maintained constant for a period of 72 h. 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 then marked and the maximum depth of water penetration in three specimens was recorded and considered as an indicator of the water penetration.

The sorptivity was measured on 100 mm concrete cubic specimens, which were dried in a 50 °C oven for 14 days. After mass stabilization, the specimens were coated with the epoxy resin on their lateral surfaces only, in order to ensure uniaxial water absorption. The specimen was rested on rods to allow free access of water to the surface and the tap water level was kept no more than 5 mm above the base of the specimen. The masses of the specimens were measured after 0, 3, 6, 24 and 72 h of absorption. The sorptivity coefficient (S) according to BS EN-480-5:1997 [12] was obtained using the following expression:

$$\frac{Q}{A} = c + S\sqrt{t} \quad (1)$$

where Q is the amount of water adsorbed; A is the cross section of specimen that was in contact with water; t is the time (second); c is the constant coefficient; and S is the sorptivity coefficient of the specimen (m/s^{1/2}).

Table 2

Mix proportions of concrete.

Mix	w/binder ^a	Limestone (%)	Concrete composition (kg/m ³)					Superplasticizer (%binder)	Slump (mm)
			Limestone	Cement	Water	Coarse aggregate	Fine aggregate		
A-0	0.37	0	0	350	129.5	973.9	960	1.1	80
A-5	0.37	5	17.5	332.5	129.5	971.6	960	1.1	85
A-10	0.37	10	35	315	129.5	969.6	960	1.1	90
A-15	0.37	15	52.5	297.5	129.5	967.4	960	1.1	85
A-20	0.37	20	70	280	129.5	965.3	960	1.1	80
B-0	0.45	0	0	350	157.5	912.3	950	–	75
B-5	0.45	5	17.5	332.5	157.5	910.2	950	–	80
B-10	0.45	10	35	315	157.5	908.1	950	–	80
B-15	0.45	15	52.5	297.5	157.5	906	950	–	80
B-20	0.45	20	70	280	157.5	903.9	950	–	75
C-0	0.55	0	0	350	192.5	740.8	1050	–	95
C-5	0.55	5	17.5	332.5	192.5	738.7	1050	–	100
C-10	0.55	10	35	315	192.5	736.6	1050	–	100
C-15	0.55	15	52.5	297.5	192.5	734.5	1050	–	95
C-20	0.55	20	70	280	192.5	732.4	1050	–	90

^a Binder = Cement + Limestone.

The electrical resistivity meter was used to measure the surface resistivity at the ages of 28, 90 and 180 days. Saturated cylinders (100 × 200 mm) were used at each test age. The electrical resistivity test for concretes was carried out by the four-point Wenner array probe technique. The probe array spacing used was 40 mm. The resistivity measurements were taken at four quaternary longitudinal locations of the specimen.

The rapid chloride permeability test was conducted in accordance with ASTM C-1202 [13] at 28, 90 and 180 days. Two specimens of 100 mm in diameter and 50 mm in thickness conditioned according to the standard were subjected to a 60-V potential for 6 h. The total charge passed through the concrete specimens was determined and used to evaluate the chloride permeability of each concrete mixture.

For scanning electron microscopy studies, selected cement paste samples cured for 3 and 28 days were used. A cement prism was cut into cubes of 10 mm square, one side of which was ground flat. The hydrated samples were flooded with acetone to stop hydration reactions and were, eventually, dried and coated with gold.

3. Results and discussion

As shown in Table 2, slumps in PLC concretes are greater than in PC concretes. It demonstrates that there is a reduction of the water demand in PLC concretes. According to Tsivilis et al. the increase of limestone content to 20% and 35% causes a decrease of the water demand from 26% to 23.5% and 22.8% [14]. Vuk et al. found the addition of 5% limestone decreased water content for the standard consistency of paste by an average of 0.5% in absolute scale [15].

Fig. 2 shows the compressive strength of concrete specimens with varying w/b ratios. As expected, the compressive strength of all concrete specimens increases with the period of curing and decreases with increasing the w/b ratio. In addition, generally, when limestone is increased, compressive strength decreases. However, up to 10% limestone replacement is not significant in this reduction. As a case in point, compressive strength for the C-20 mixture after 180 days was 23% lower than the C-0 mixture.

The maximum compressive strength in concrete specimens at 180 days was 67.0 MPa corresponding to the A-10 mixture and the minimum compressive strength was 32.0 MPa corresponding to the C-20 mixture. The reduction in compressive strength is 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 limestone.

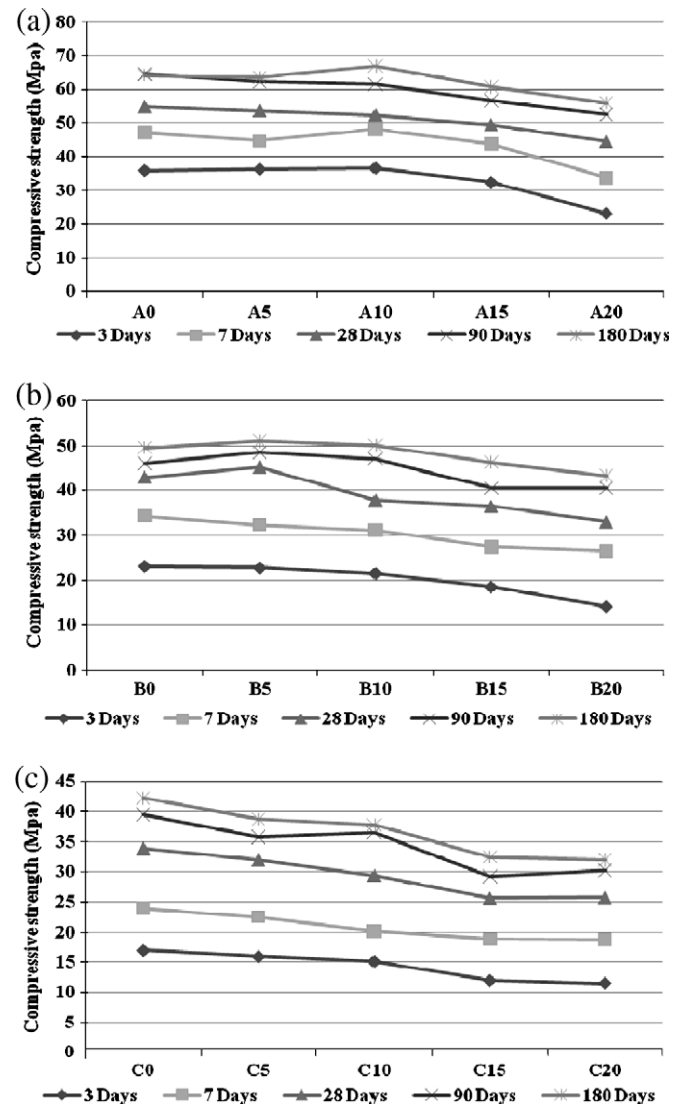


Fig. 2. The effect of limestone on the compressive strength at various ages.

In Portland limestone cements, the filler effect and heterogeneous nucleation react opposite of the dilution effects. For this reason, there is an optimum limestone replacement for Portland

limestone cements. For lower values, properties of PLC are better than PC and vice versa.

Dhir et al. showed that at equal w/c ratios, with equal cement and water contents, the strength of concrete mixes was found to decrease with increasing the limestone content, although differences between PLC concrete containing 15% limestone and PC concrete were minimal [16].

Table 3 summarizes the results of the water penetration depths in all concrete mixtures. It is observed from this table that for all concrete mixtures, the water penetration depth increases with increasing the w/b ratio. The maximum depth of water penetration is 21 mm for the C-20 mixture after 28 days and the minimum is 8 mm for the A-10 mixture. After 180 days, the maximum depth of water penetration is 14.5 mm for the C-20 mixture and the minimum is 2.5 mm for the A-10 mixture.

The results indicate that PLC concretes containing up to 10% limestone generally have lower depths of water penetration than PC concretes and PLC concretes containing more than 10% limestone show greater depths. For example, after 180 days for C series specimens, the depth of water penetration increases from 20% to 45% for concretes containing 15% and 20% of limestone, respectively.

The decrease and increase of penetration depths are related to the filler effect, heterogeneous nucleation and the dilution effect respectively. The results of several studies [17–19] show similar trends.

Water transportation in cementitious materials depends on capillary porosity, its connectivity and the pore structure (tortuosity and constriction or disconnection); these parameters are directly related to the w/b ratio and the progress of cement hydration.

Irassar [6] reported for Portland cement pastes with a w/c of 0.40, 0.45 and 0.50, capillary porosity becomes disconnected when the hydration degree is approximately 0.72, 0.82 and 0.93, respectively. On the other hand, capillary disconnection is very difficult for w/c higher than 0.60, and impossible for w/c of 0.70.

However, for reduction of capillary porosity in Portland limestone cement paste, a lower w/b is needed with increasing the limestone replacement. This issue is related to the dilution effect.

For example, to obtain the capillary porosity threshold in pastes made with PLC fully hydrated ($\alpha = 1$) and w/b ratio of 0.50, 0.45 and 0.40, the maximum percentage of limestone addition will be 5%, 15% and 25% respectively. In other words, capillary disconnection of cement paste containing 5%, 10%, 20% and 35% limestone will be obtained by using a w/b of approximately 0.50, 0.48, 0.43 and 0.34, respectively [6].

Table 4 shows the influence of the w/b ratio on the sorptivity of concretes containing different amounts of limestone at the age of 28, 90 and 180 days. It is clear that the w/b ratio reduction decreases the sorptivity. In all three series (A–C), sorptivity

decreases with the period of curing and increases with limestone replacement. Also, there is no significant difference between the sorptivity of specimens containing 0% and 5% limestone. For example, after 180 days, the sorptivity coefficient for the B-20 mixture was 1.6 times as large as that for the B-0 mixture but this parameter for the B-5 mixture was only 1.02 times as large as that for the B-0 mixture.

Ghrici et al. [20] showed that incorporation of 15% limestone into PC diminishes the sorptivity of concrete by 2% and 9% at 28 and 90 days of age, respectively, for the w/b ratio of 0.6. Also,

Table 4

The effect of limestone on the sorptivity coefficient (10^{-6}) ($\text{m/s}^{0.5}$) at various ages.

Mix	28 days	90 days	180 days
A-0	7.3	5.53	4.84
A-5	7.72	5.59	4.91
A-10	7.31	5.57	5.01
A-15	8.66	6.47	6.26
A-20	9.44	9.18	8.97
B-0	8.24	7.93	7.73
B-5	8.8	8.48	7.9
B-10	13.49	10.63	8.46
B-15	14.66	13.07	12.11
B-20	14.66	13.32	12.36
C-0	17.35	13.27	12.21
C-5	17.32	16.66	15.59
C-10	19.77	15.88	15.03
C-15	23.58	18.4	15.91
C-20	23.54	19.06	17.97

Table 5

The effect of limestone on the electrical resistivity ($\text{k}\Omega \text{ cm}$) at various ages.

Mix	28 days	90 days	180 days
A-0	16.7	26.3	30.5
A-5	18.0	24.0	30.0
A-10	19.3	28.3	31.5
A-15	20.5	25.3	29.5
A-20	16.0	23.3	28.3
B-0	16.5	20.0	21.7
B-5	15.0	20.3	20.7
B-10	14.7	19.7	20.3
B-15	13.2	20.0	20.0
B-20	13.9	19.0	20.2
C-0	12.9	16.8	16.8
C-5	11.5	16.3	16.3
C-10	11.0	14.7	15.3
C-15	11.7	13.7	15.3
C-20	11.7	14.7	15.0

Table 6

The effect of limestone on the rapid chloride ions permeability (Coulomb) at various ages.

Mix	28 days	90 days	180 days
A-0	3329	2551	1392
A-5	3445	2528	1565
A-10	3027	2311	1525
A-15	3344	2707	1700
A-20	3707	2900	1938
B-0	4842	3905	2150
B-5	5142	3824	2277
B-10	4448	3406	2124
B-15	5154	4003	2747
B-20	5432	4101	3390
C-0	5073	4461	3310
C-5	6151	5225	3965
C-10	5754	4950	3755
C-15	4870	4376	3624
C-20	6171	5625	4125

Table 3

The effect of limestone on the water penetration depth (cm) at various ages.

Mix	28 days	90 days	180 days
A-0	1.1	0.6	0.35
A-5	0.9	0.65	0.3
A-10	0.8	0.6	0.25
A-15	1.05	0.85	0.4
A-20	1.25	0.95	0.9
B-0	1.3	0.8	0.55
B-5	1.2	0.75	0.4
B-10	1.4	0.85	0.6
B-15	1.3	1	0.8
B-20	1.7	1.3	1
C-0	1.7	1.1	1
C-5	1.6	1.1	0.9
C-10	1.8	0.9	1
C-15	1.9	1.5	1.2
C-20	2.1	1.75	1.45

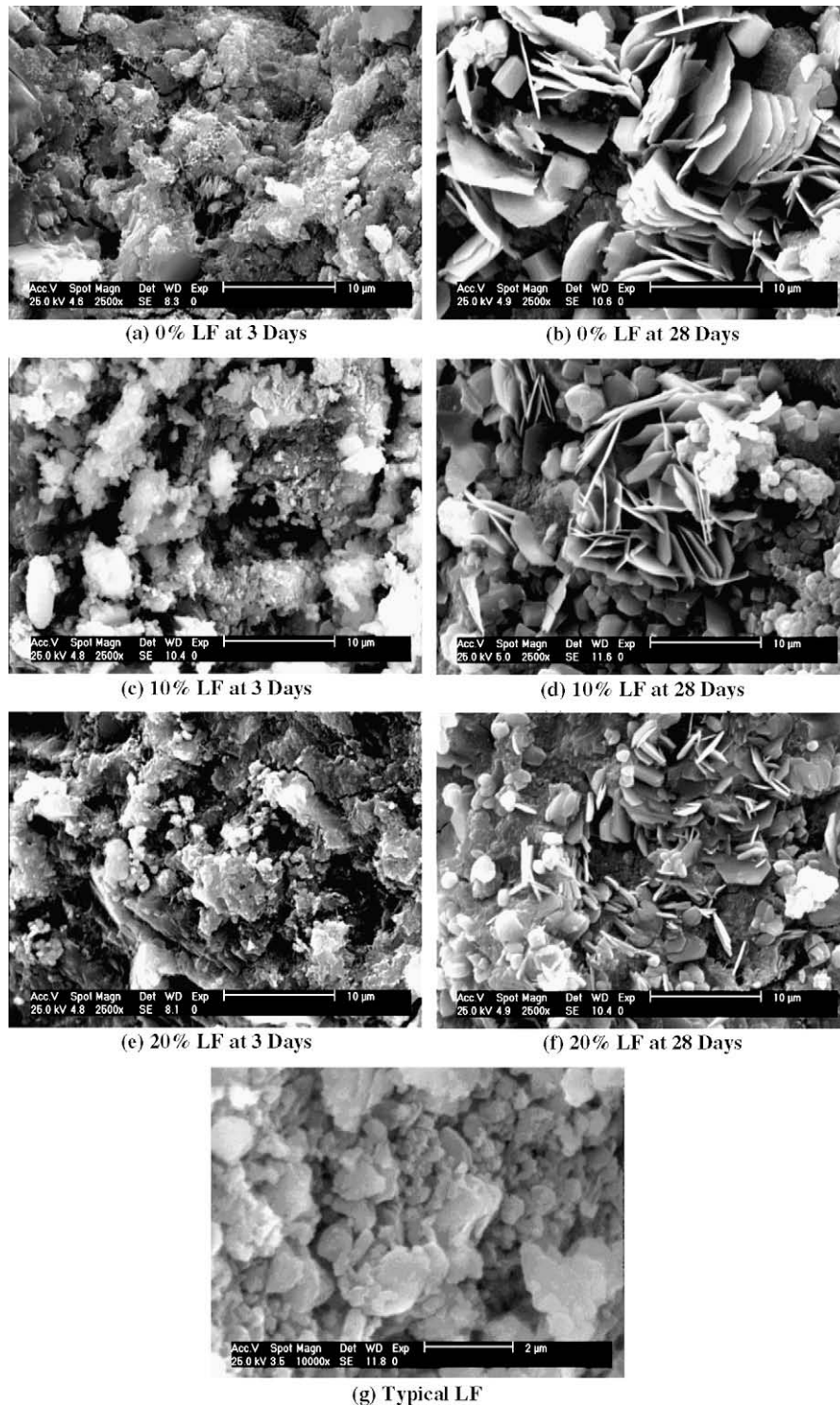


Fig. 3. SEM photograph of cement pastes and typical limestone.

Tsivilis et al. reported that replacing 15% of PC with limestone at $w/b = 0.7$ had an insignificant effect on the sorptivity of concrete [21].

The corrosion-resisting performance of concrete is influenced by its electrical resistivity, which refers to the resistance that an electrical charge experiences while passing through the concrete. The increased electrical resistivity of concrete hinders the movement of electrons from the anodic to the cathode regions, and thereby retards the propagation of the corrosion process.

Results obtained from electrical resistivity tests (Table 5) indicate that the electrical resistivity decreases with increasing the w/b ratio. The electrical resistivity decreased in the B and C series with increasing limestone replacement. However in the A series, PLC concretes containing 10% and 20% limestone replacement show maximum and minimum resistivity values, respectively.

The highest value of electrical resistivity is $31.5 \text{ k}\Omega \text{ cm}$ for the A-10 mixture after 180 days and the minimum is $15.0 \text{ k}\Omega \text{ cm}$ for the C-20 mixture. The results for chloride penetration, measured

in terms of the electric charge passed through the specimens in coulombs, obtained at the age of 28, 90 and 180 days are presented in Table 6. The decrease of the w/b ratio from 0.55 to 0.45 and also from 0.55 to 0.37 significantly reduces the rapid chloride ion permeability.

In the A and B series, the PLC concrete containing up to 10% limestone and in C series PLC concrete containing up to 15% limestone indicates suitable performance. Also, in the A series all of the 180 day concrete specimens are categorized as low chloride permeability, according to the ASTM C1202. Hornain et al. show that the addition of limestone to neat cement pastes and mortars reduces the diffusion coefficient of chloride ions. The reduction is attributed to the filler effect on the tortuosity of the concretes [22].

Ghrici et al. [20] showed that the penetration of chloride ion increases in concretes containing 15% limestone. Also, Bonavetti et al. reported that the penetration of chloride ion increased from 43% to 114% for concretes containing 10% and 20% of limestone, respectively [23].

In Fig. 3a–f the microstructures of the cementitious paste with normal consistency of hydraulic cement (ASTM C187), containing 0%, 10% and 20% limestone replacement are shown at 3 and 28 days after hydration. In addition, a typical SEM photograph of the limestone is presented in Fig. 3g. The morphology in cement pastes with and without limestone is different. The pore structures (tortuosity and constriction or disconnection) are improved with increasing the curing time. This trend deals with the hydration progress. As expected, the Ca/Al ratio changes while the limestone replacement increases. This ratio for cement pastes containing 0%, 10% and 20% limestone is 5.51, 7.1 and 13.62 respectively. Ye et al. showed that small limestone particles still exist in the pastes even after 28 days of hydration [24].

4. Conclusions

The following conclusions can be drawn from the obtained experimental data:

- Generally, the PLC concretes having up to 10% limestone, provide competitive properties with PC concretes. Adding higher percentages of limestone to cement produces less competitive concrete properties.
- The PLC concretes indicate satisfactory compressive strength and generally demand less water than PC concretes.
- In all three series (A–C), sorptivity decreases with the period of curing and increases with the limestone replacement percentage. Also, there is no significant difference between the sorptivity of specimens containing 0% and 5% limestone.
- PLC concretes decrease water penetration depth with limestone contents up to 10%.
- The partial replacement of cement with limestone reduces the electrical resistivity of concrete. However in Series A, PLC concrete containing 10% and 20% limestone replacement have maximum and minimum values respectively.
- Chloride ion permeability is increased considerably when the (w/b) ratio is increased from 0.37 to 0.55. For the materials in this study, the optimum replacement of limestone is between 10% and 15%.

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