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Mechanical properties and durability of mortar and concrete containing natural pozzolana and limestone blended cements

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Abstract

The benefits of limestone filler (LF) and natural pozzolana (NP) as partial replacement of Portland cement are well established. Economic and environmental advantages by reducing CO_2 emission are well known. However, both supplementary materials have certain shortfalls. LF addition to Portland cement causes an increase of hydration at early ages inducing a high early strength, but it can reduce the later strength due to the dilution effect. On the other hand, NP contributes to hydration after 28 days improving the strength at medium and later ages. Hence, ternary blended cement (OPC–LF–NP) with better performance could be produced. In this paper, mortar prisms in which Portland cement was replaced by up to 20%LF and 30%NP were tested in flexure and compressive strength at 2, 7, 28 and 90 days. Some samples were tested under sulfate and acid solutions and for chloride ions permeability. Results show that the use of ternary blended cement improves the early age and the long-term compressive and flexural strengths. Durability was also enhanced as better sulfate, acid and chloride ions penetration resistances were proved.

Keywords: Limestone filler; Natural pozzolana; Ternary blended cement; Mortar; Concrete; Compressive strength; Durability

1. Introduction

Most cement plants consume much energy and produce a large amount of undesirable products, which affect the environment. In order to reduce energy consumption and CO_2 emission and increase production, cement manufacturers are blending or intergrinding mineral additions such as slag, natural pozzolana, sand and limestone [1].

Large number of studies [2–7] have shown that natural pozzolana have been widely used as a substitute for Portland cement in many applications because of their advantageous properties which include cost reduction, reduction in heat evolution, decreased permeability and increased chemical resistance. However, they are often associated with shortcomings such as the need to moist-curing for longer time and a reduction of strength at early ages and up to 28 days.

The consumption of calcite, the formation of carbo-aluminates, the acceleration of the hydration of C_3A and C_3S, the change in the C–S–H and the formation of transition zone between the filler and cement paste demonstrate the reactivity of limestone fillers [8–10]. Consequently, this reactivity improves the early strength, but an associated effect of limestone addition is the reduction of later strength by the dilution effect [11]. The European Standard EN 197-1 [12] identifies two types of Portland-limestone cement containing 6–20% limestone (type II/A-L) and 21–35% limestone (type II/B-L), respectively. But, the addition of limestone in concrete increases the chloride ion diffusion depending on the level of addition [13,14]. Also, the serious problem associated with the use of limestone in cement paste mortar or concrete is the formation

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of thaumasite as a result of sulfate attack at low temperature (below 15 °C) [15]. In hot arid countries, as typified by North Africa and the Middle East, the mean temperature is greater-than 20 °C, and the production of Portland Limestone Cement containing up to 20% of limestone filler has grown during the last years.

Different additions, when used as partial cement replacement materials in mortar and concrete, have, as outlined above, various advantages and different disadvantages. There is growing belief that ternary and quaternary-blended composite cements can be optimized in which a synergistic effect allows component ingredients to compensate for their mutual shortcomings [16]. In recent years, several researchers [17–26] have reported that the use of the ternary cements, present more advantages compared to both binary cements and OPC.

It should be possible, by the systematic adjustment of the proportions, to produce ternary blended cement (OPC–LF–NP) witch utilizes the desirable characteristics of one addition while compensate for the undesirable characteristics of the other. For example, the limestone filler contributes to the early strength and the natural pozzolana increases the long-term strength. On the other hand, the best combination of these mineral additions, can lead to an enhanced durability performance.

In Algeria, most of the cement is being blended with additions such as limestone and natural pozzolana. Natural pozzolana is being used for cement manufacture by at least six of the twelve Algerian cement plants whereas two cement plants uses limestone. These cement plants add usually about 15% of natural pozzolana and 10% of limestone filler as cement replacement by weight. Apart from the internal quality control testing for conformity to standards requirements, no detailed investigation has been done to evaluate the effect of the interaction between limestone and natural pozzolana additions on the properties of cement mortar and concrete. Also, the effect of utilizing both LF and NP on concrete properties is not well documented in the literature.

The objective of this paper is to present the optimization of the compressive strength in ternary blended cement containing limestone filler and natural pozzolana. The durability performance of mortar and concrete containing ternary blended cement (OPC–LF–NP) is also presented.

2. Experimentation

2.1. Materials used

A local ordinary Portland cement with a fineness of 350 m^2/kg was used for all the mixes. The natural pozzolana used in this work was from Beni–Saf quarry in the west of Algeria. The limestone and pozzolana were ground in a laboratory mill to a specific surface of 370 m^2/kg and 420 m^2/kg^{-1} respectively. The chemical composition of the cement and natural pozzolana and limestone are given in Table 1. The mineralogical composition of the limestone and natural pozzolana was determined by X-ray diffraction and is presented in Figs. 1 and 2. The sand used in mortar mixes was a standard sand of 2 mm maximum aggregate size. A concrete mix was used only for chloride penetration and sorptivity tests. Crushed limestone coarse aggregates with a nominal size of 12 mm, and a specific gravity of 2.65, and natural sand with a specific gravity of 2.59 were used for the concrete samples. The superplastcizer used in the concrete mix is based on naphthalene sulphonates and modified lignosulphonates with specific gravity of 1.20.

Table 1
Chemical composition (%) of the cement and natural pozolana used

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	CaO (free)	SO_3	IR[a]	LOI[b]
Cement	23.7	6.58	4.0	64.95	0.32	0.33	–	–	–
Pozzolana	46.86	16.62	9.37	9.38	2.84	–	0.36	–	5.79
Limestone	0.55	0.40	0.17	53.47	1.02	–	1.50	–	43.13

[a] Insoluble residue.
[b] Loss on ignition.

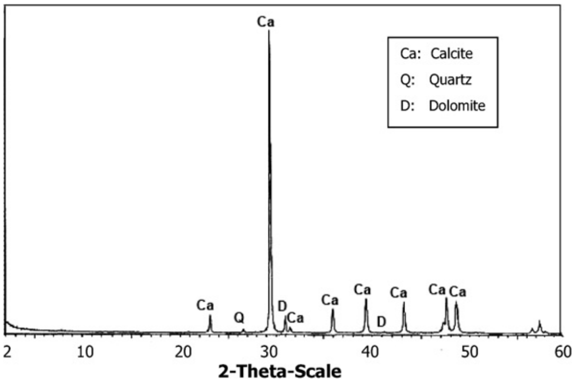

Fig. 1. X-ray diffraction of limestone.

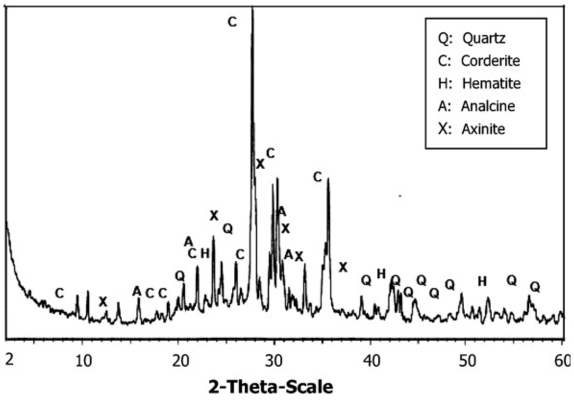

Fig. 2. X-ray diffraction of natural pozzolana.

2.2. Mixes used

The mortar mixes had proportions of 1binder: 3 Sand. The binder consisted of cement and natural pozzolana

Table 2
Compressive and flexural strengths of tested composite cement mortars

OPC	NP	LF	Flexural strength (MPa)				Compressive strength (MPa)			
			2 days	7 days	28 days	90 days	2 days	7 days	28 days	90 days
100	00	00	4.1	6.3	8.9	8.9	16.6	38.1	45.3	47.8
90	00	10	4.3	5.5	7.8	8.5	17.4	35.4	43.3	46.6
80	00	20	3.6	5.2	7.6	8.3	13.7	31.7	39.4	44.7
90	10	00	3.6	5.7	8.1	8.7	15.6	33.8	43.3	51.4
80	20	00	3.9	5.1	8.3	8.3	15.9	32.1	41.3	51.3
70	30	00	2.9	4.7	6.6	8.1	11.2	26.6	37.9	49.8
80	10	10	3.0	5.8	8.5	8.6	12.9	32.7	41.4	49.7
70	20	10	2.7	4.9	7.6	8.8	11.3	27.8	39.2	50.1
60	30	10	2.6	4.6	7.4	7.8	09.8	23.9	34.8	44.8
70	10	20	3.2	5.2	7.0	7.7	11.8	25.4	35.1	44.7
60	20	20	2.1	3.9	6.5	6.8	08.4	20.2	31.8	42.3
50	30	20	1.8	3.7	6.0	6.4	07.3	16.6	28.2	37.1

and/or limestone. The cement was formulated varying the replacement of LF from 0% to 20% and the replacement of NP from 0% to 30%. All replacements were made by mass. The water:binder (*w*/*b*) ratio was kept constant at 0.5. The concrete mix had a total binder content of 400 kg/m^3, fine aggregate content of 720 kg/m^3, coarse aggregate content of 1130 kg/m^3, and (*w*/*b*) ratios of 0.4 and 0.6 were used. For the (*w*/*b*) of 0.4, the superplastcizer was added with a dosage of 1 l per 100 kg of binder to improve workability. Table 2 shows the combination of blended cements studied in the determination of strength of mortars. The durability was conducted only on control cement and the cement containing 30% of NP, 15% of LF, and 20% of NP with 10% of LF.

2.3. Specimen preparation, curing and testing

The strength of mortars was determined in accordance with the European Standard EN 196-1. The mortar was placed in 40 × 40 × 160 mm-prismatic steel moulds. After casting, specimens were left covered with a plastic sheet. After removal from the moulds, at 24 h of age, mortar specimens were immersed in water saturated with lime at 20 °C until the age of testing. Compression and three point bending tests were conducted at 2, 7, 28 and 90 days of age. The results reported are the average of three flexural specimens and six compression tests.

The sorptivity was measured on 70 mm concrete cubic specimens, which were oven dried at 105 °C for 24 h. After mass stabilization, the specimens were coated with the epoxy resin on their lateral surfaces only, in order to ensure uniaxial water absorption. A schematic diagram of the water absorption test is given in Fig. 3. The masses of the specimens were measured after 1, 4, 9, 16, 36, 49 and 64 min of absorption. The sorptivity coefficient (*S*), was obtained by using the following expression:

$$\frac{Q}{A} = S \cdot \sqrt{t} \tag{1}$$

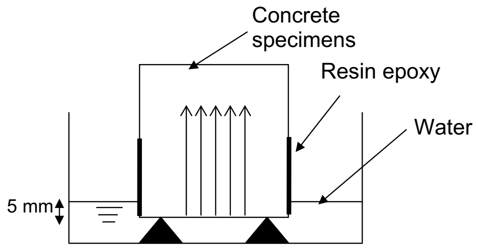

Fig. 3. Schematic diagram of water absorption test.

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; S is the sorptivity coefficient of the specimen (cm/s$^{1/2}$).

The rapid chloride permeability test was conducted in accordance with ASTM C-1202. Two specimens of 100 mm in diameter and 50 mm in thickness conditioned according to the standards were subjected to 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 the sulfate attack tests, the mortars specimens were immersed in 5% sodium sulfate (Na_2SO_4) or 5% magnesium sulfate ($MgSO_4$) at laboratory temperature (23 ± 2 °C). The sulfate solution was renewed every 30 days. The sulfate attack was evaluated through the measurement of the expansion in accordance with ASTM C-1012 on prismatic specimens measuring 25 × 25 × 285 mm.

The relative acid attack was determined in accordance with ASTM C-267. The mortars specimens were cured in water at 23 ± 2 °C for 30 days before being subjected to acid attack. Three specimen of each mortar mix were immersed in 3% sulfuric acid (H_2SO_4) or 1% hydrochloric acid (HCl). The attacked portions of the mortar specimens were cleaned with deionised water and then the acid attack was evaluated through measurement of the weight loss of the specimens determined as follows:

$$\text{Weight loss (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \tag{2}$$

where W_1 is the weight of the specimen before immersion and W_2 is the weight of the cleaned specimen after immersion. The solution was renewed every 15 days and the weight loss of the specimens measured.

3. Results and discussion

3.1. Development of the compressive strength

Table 2 reports the mean value of flexural and compressive strength for cements used at all ages. Based on the experimentally obtained results, models were developed at each age. These models are based on the quadratic response surface where the two experimental variables (the proportion of NP and LF used as partial replacement of cement) have been used. The model is as follows:

Table 3
Coefficients model for the compressive strength

Ages (days)	Coefficients						R^2
	a_0	a_1	a_2	a_{11}	a_{22}	a_{12}	
2	17.3	−12.28	−4.4	−38.3	−40	−40.3	0.984
7	37.9	−35.6	2.6	2.5	−189	−63.6	0.987
28	45.2	−15.3	5.7	−26.7	−189	−63.9	0.993
90	47.4	54.2	17.2	−154.8	−166.23	157.13	0.984

$$f_c(t) = a_0 + a_1x_1 + a_2x_2 + a_{11}x_1^2 + a_{22}x_2^2 + a_{12}x_1x_2 \tag{3}$$

where $f_c(t)$ is the compressive strength at t days, x_1 is the percentage of NP, x_2 is the percentage of LF and a_0; a_1; a_2; a_{11}; a_{22} and a_{12} are the coefficients of the model. The coefficients for the equation at 2, 7, 28 and 90 days are shown in Table 3, where R^2 is the coefficient of determination.

Fig. 4 illustrates the isoresponse curves of the compressive strength showing the interaction effect of NP and LF for the domain studied in the ternary system. At two days (Fig. 4a), the stationary point corresponding to the maximum compressive strength is obtained by the replacement of 10% of LF and a very little content of NP. The maximum is 16% higher than the strength of PC mortar. The isoresponse curves have a similar contour for 7 and 28 days (Fig. 4b and c), while the zone of maximum strength is located around 10% of LF replacement and low level of NP replacement (0–10%). However, the contour of isoresponse curves changes significantly at 90 days, where, the maximum compressive strength is obtained with the larger proportion of NP (20%) and around 10% of LF. At this point, the strength was 7% higher than the corresponding OPC mortar.

In summary, the point of maximum strengths is around 10% of LF and low NP replacement level at the early ages. After 28 days, this point moves toward the high level of NP replacement and low LF content.

3.2. Correlation between flexural and compressive strength

The relationship between the compressive strength and the flexural strength is given in Fig. 5 and seems to fit well with the relation proposed by some other researchers [27]:

$$f_t = k(f_c)^{\alpha} \tag{4}$$

The correlation between the flexural strength and the compressive strength results were calculated for the entire population of the test results and hence the relation obtained is:

$$f_t = 0.45(f_c)^{0.76} \tag{5}$$

with a coefficient of determination of $R^2 = 0.96$. It can be observed (Fig. 5) that the mortar mixes containing limestone and/or natural pozzolana behave in a similar manner to that of OPC plain mortar.

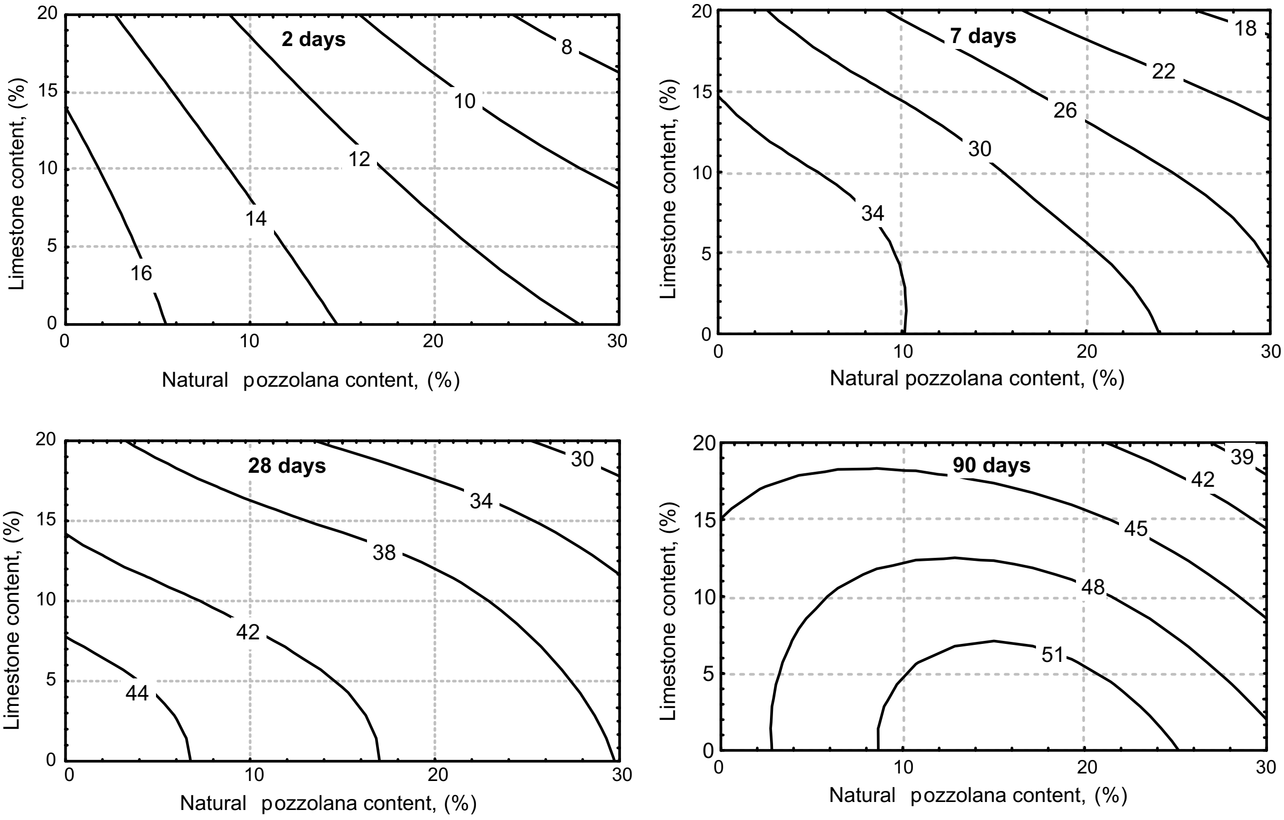

Fig. 4. Compressive strength (in MPa) isoresponse curves for the ternary system at different ages.

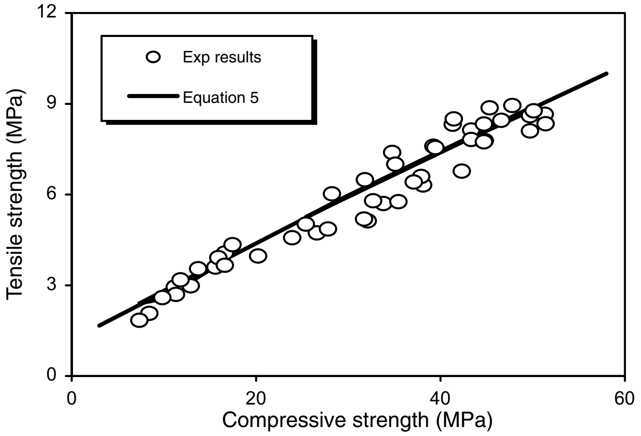

Fig. 5. Correlation between flexural strength and compressive strength.

3.3. Durability tests

3.3.1. Sorptivity

Fig. 6 shows the influence of the waters/binder (*w*/*b*) ratio on the sorptivity of concretes containing different amounts of addition at the age of 28 and 90 days. As is evident, the diminution of the (*w*/*b*) ratio reduces the sorptivity. At 28 days of age, with regard to the plain concrete, the sorptivity coefficient of concrete containing 30% of natural pozzolana diminished from 56% to 29% when the (*w*/*b*) ratio was reduced from 0.6 to 0.4. In the same way, at 90 days of age, the sorptivity coefficient was 34% lower than the corresponding OPC mortar for both (*w*/*b*) ratio of 0.4 and 0.6. In this case, pores in the bulk paste or in the interfaces between aggregate and cement paste are filled by these mineral admixtures. The capillary pores are reduced by the formation of secondary C–S–H gel due to the pozzolanic reaction, and hence the reduction in the capillary sorption of concrete. However, the incorporation of 15% of limestone diminished the sorptivity coefficient of concrete by 2% and 9% at 28 and 90 days of age, respectively, for (*w*/*b*) ratio of 0.6. Tsivilis et al. [28] reported that replacing 15% of Portland cement with limestone at a constant (*w*/*b*) ratio of 0.7 had little effect on the sorptivity of concrete.

The 20%NP + 10%LF mixture diminished the sorptivity coefficient by 25% and 31% for a (*w*/*b*) ratio of 0.4 and 0.6, respectively.

3.3.2. Rapid chloride ion permeability

The results for chloride penetration, measured in terms of the electric charge passed through the specimens in coulombs, obtained at the age of 28 and 90 days are presented in Fig. 7. The diminution of the (*w*/*b*) ratio from 0.6 to 0.4 reduces significantly the rapid chloride ion permeability.

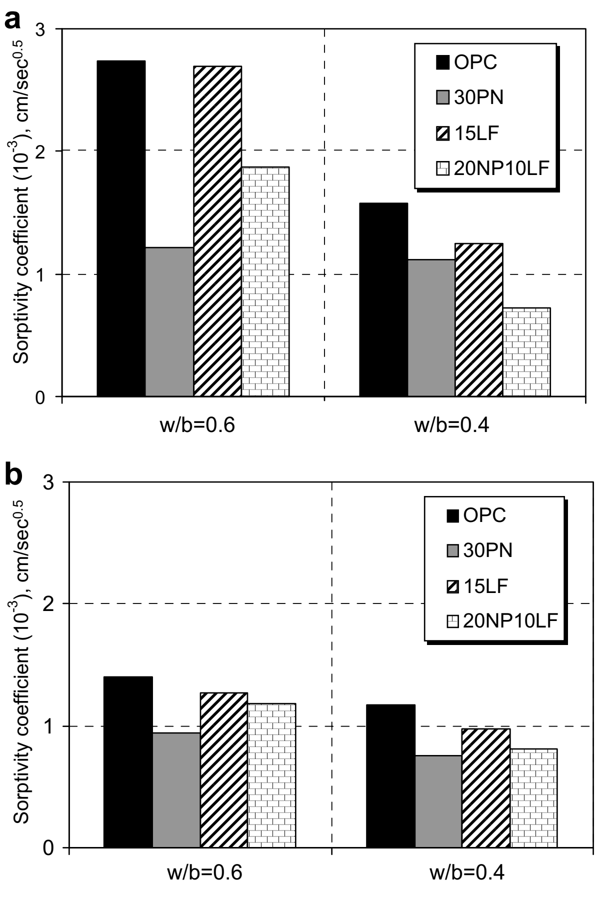

Fig. 6. Sorptivity coefficients of concrete at (a) 28 days and (b) 90 days.

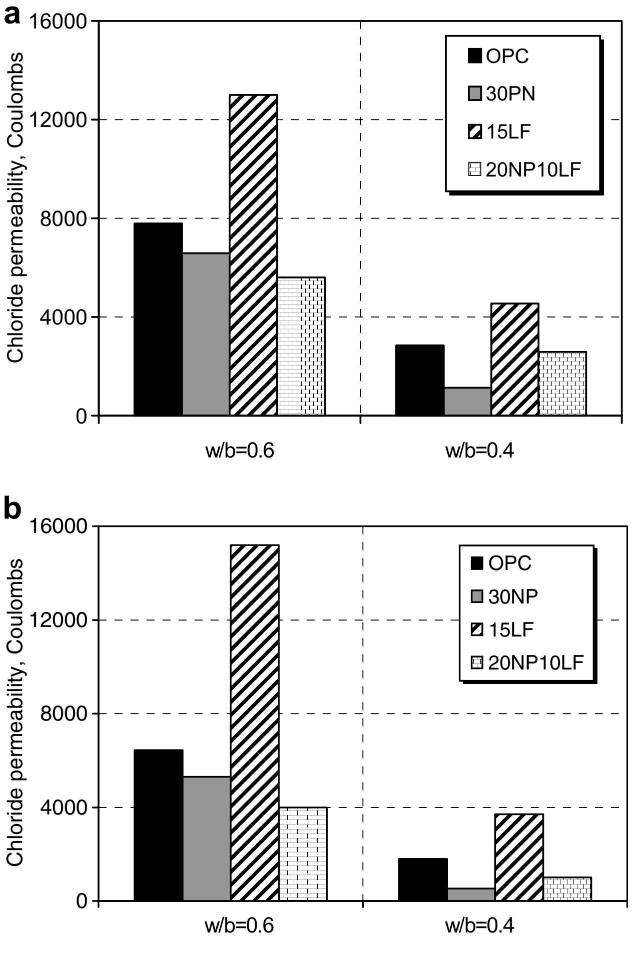

Fig. 7. Chloride permeability of concrete at (a) 28 days and (b) 90 days.

At 28 days of age, with regard to the plain concrete, chloride ion permeability factor for concrete containing 30% of pozzolana diminished from 7800 to 6540 C for a (*w*/*b*) ratio of 0.6. It can be seen that the charge passed is more than 4000 C, which characterized high chloride permeability according to the ASTM C1202. However, for a (*w*/*b*) ratio of 0.4 chloride ion permeability diminished from moderate permeability (2852 C) to low permeability (1140 C). At 90 days of age, a similar trend for chloride ion permeability was observed. But, for a (*w*/*b*) ratio of 0.4 chloride ion permeability diminished from low permeability (1800 C) to very low permeability (535 C). This may be related to the refined pore structure of these concretes and their reduced electrical conductivity [29]. Results reported elsewhere [7,30] have shown a reduction in the chloride ion permeability with the addition of pozzolanic materials, with further reductions obtained at higher replacement levels and lower (*w*/*b*) ratios.

For a (*w*/*b*) ratio of 0.6, with regard to the plain concrete, chloride ion permeability factor for concrete containing 15% of LF increased by 67% and 135% at 28 days and 90 days, respectively. These results were similar to the results of Bonavetti et al. [14] who reported that the penetration of chloride ion increased from 43% to 114% for concretes containing 10% and 20% of limestone, respectively. The higher chloride ion permeability of the concrete containing LF, as compared to that of plain concrete, may be related to the higher level of OH^- ions present in the pore fluid of the concrete made with LF. Andrade [31] proposed that the OH^- ions present in the pore fluid act as a supporting electrolyte and are responsible for the transportation of a significant amount of charge during chloride ion permeability because of its higher ionic conductivity than the other ions present in pore fluid (Na^+, K^+ and Ca^{2+}). On the other hand, the higher chloride ion permeability could be attributed to the porous and connected paste–aggregate interfacial transition zone (ITZ) associated with limestone filler particles addition. The water permeability of concrete containing limestone was also found to be greater-than that of plain concrete [13].

The (20%NP + 10%L) mixture yielded the lowest chloride permeability for the high (*w*/*b*) ratio of 0.6. However, it seems that the ternary blend concrete exhibits better chloride ion permeability performance than plain concrete and concrete with single addition replacements. This trend of results has also been reported by other researchers [19,24].

3.3.3. Sulfate attack

Fig. 8 shows the expansion of mortars exposed to 5% Na_2SO_4 and 5% $MgSO_4$. Higher expansion in specimens placed in ($N\overline{S}$) environment compared to those placed in ($M\overline{S}$) environment are observed. Expansion observed in 15%L and 20%NP + 10%LF mortar prisms are similar and are marginally higher than the expansion observed in 30%NP mortar prisms. However, higher expansion was observed for OPC mortar prisms.

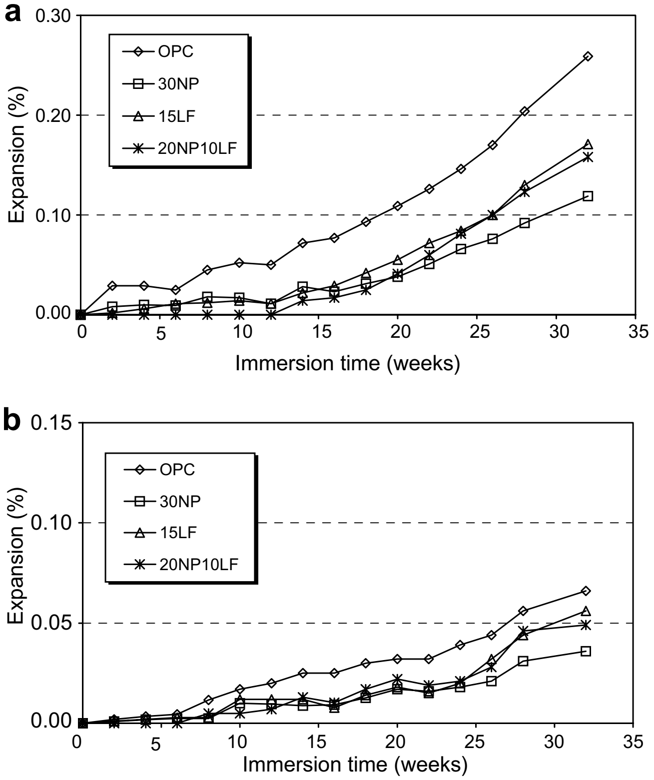

Fig. 8. Expansion of mortar exposed to (a) 5% Na2SO4 and (b) 5% MgSO4.

Expansion values at 32 weeks of exposure to ($N\overline{S}$) solution are 0.26%, 0.12%, 0.17% and 0.16% for mixes with 0%, 30%NP, 15%LF and 20%NP + 10%LF, respectively.

The addition of 30% of natural pozzolana produces an important reduction of expansion of 54% than the corresponding expansion of the OPC mortar. The higher expansion observed in plain mortar may be attributed to the formation of secondary ettringite, which is characterized by expansion and cracking. In mortar containing natural pozzolana, pozzolanic reaction consumes part of the $Ca(OH)_2$ produced by the hydration of cement. Hence, the quantity of gypsum formed in the reaction between sulphates and $Ca(OH)_2$, which is responsible for the formation of secondary ettringite, will be smaller in pozzolanic cement than in plain cements. Further, the pozzolanic reaction produces a secondary CSH gel that also results in the densification of the hardened cement paste since it is deposited in the pores and enhances the paste–aggregate interface. These effects reduce significantly the diffusion of SO_4^- ions and explain the lower expansion observed in pozzolanic cement as against plain cement.

The addition of 15% of limestone caused a reduction of expansion of 36% than the corresponding expansion of the OPC mortar. Gonzalez and Irassar [32] reported that the addition of 10% of limestone reduced the expansion by 30% after 6 months exposure to 5% Na_2SO_4. The reduction in expansion for limestone specimens is probably because of the formation of monocarboaluminate in the presence of limestone and hence the suppression or delay of the conversion of ettringite to monosulfoaluminate.

However, natural pozzolana did not change the sulfate resistance of the limestone cement investigated in this study. This trend of result was similar to the results published by Tsivilis et al. [33]. Vuk et al. [34] studied the influence of silica fume, fly ash and blast furnace slag on the sulfate resistance of limestone cements and found that silica fume improved the resistance to sulfate attack but the addition of fly ash and blast furnace slag did not give any improvement.

3.3.4. Acid attack

Fig. 9 shows the test results of weight change versus time for mortar specimens exposed to 1% HCl and 3% H_2SO_4 solutions for 180 days. The percentage loss in weight with time for mortars immersed in HCl was much lower than that for specimens immersed in H_2SO_4. After 180 days exposure to 1% HCl solution, the total loss in weight of OPC mortar and mortar containing 15% of limestone are about 35%. For mortar containing 30% of NP and mortar containing 20%NP + 10%LF mixture, the loss in weight are lower than the corresponding OPC mortar by 16% and 6% respectively. Zivika and Bajz [35] reported that some authors have claimed that the use of pozzolans had only a weak positive effect on the increase of the acidic resistance. On the other hand, after 180 days exposure to 3% H_2SO_4 solution, the total loss in weight of OPC mortar and mortar containing 30% of NP are about 75%. For mortar containing 15% of LF and mortar containing 20%NP + 10%LF mixture, the loss in weight are lower than the corresponding OPC mortar by 24% and 10% respectively. Therefore, it can be concluded from these results that NP can be used to improve hydrochloric acid (HCl) resistance and LF can be used to improve sulfuric acid (H_2SO_4) resistance.

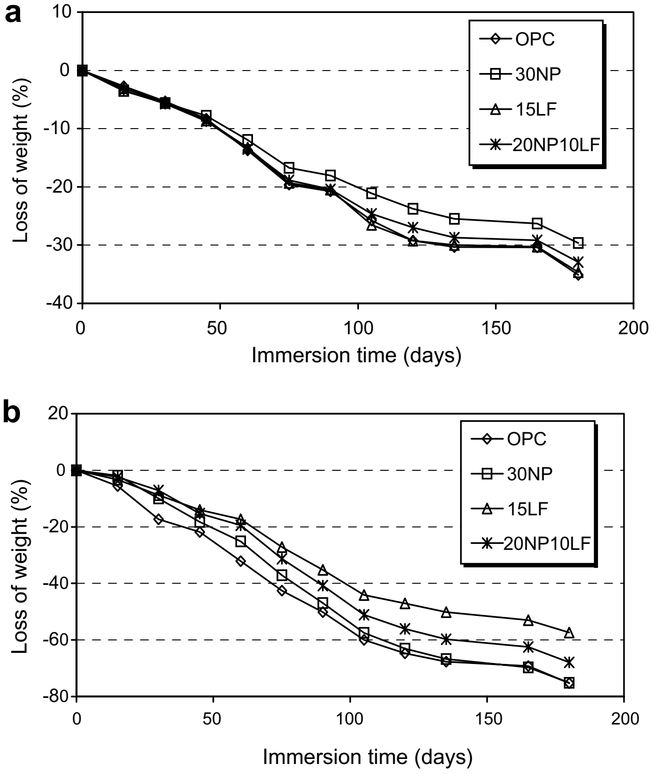

Fig. 9. Weight loss of mortar due to the acidic attack (a) 1% HCl and (b) 3% H_2SO_4.

4. Conclusion

This study was conducted to assess the effect of the interaction between limestone filler and natural pozzolana additions on the properties of mortar and concrete. The following conclusions can be drawn from the present study:

- The point of maximum strengths is around 10% of LF and low NP replacement level at the early ages. After 28 days, this point moves toward the high level of NP replacement and low LF content.
- The partial replacement of cement with addition reduces the sorption of concrete. Also, the sorption decreases with the increase in the duration of curing.
- Chloride ion permeability decreases considerably when the (*w*/*b*) ratio was reduced from 0.6 to 0.4. The ternary blend concrete exhibits better chloride ion permeability performance than plain concrete and concrete with single addition replacements for a (*w*/*b*) ratio of 0.6. But, for a (*w*/*b*) ratio of 0.4, concrete containing 30% NP exhibits better chloride ion permeability than both ternary and plain concrete mixes.
- Lower expansion as observed in pozzolanic cement and natural pozzolana did not change the sulfate resistance of the limestone cement investigated in this study.
- It can be concluded from the results of this study that NP can be used to improve hydrochloric acid (HCl) resistance and LF can be used to improve sulfuric acid (H_2SO_4) resistance.

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