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High replacements of reactive pozzolan in blended cements: Microstructure and mechanical properties

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ABSTRACT

A study comparing silica fume (SF) and dealuminated kaolin (DK) as pozzolanic materials was carried out. 10, 20 or 30-wt.% SF or DK were substituted for Portland cement. The kinetics of hydration were studied at water/solid ratios of 0.30 and 0.50. The hydration reactions were monitored by determining the compressive strengths, the free lime contents and the amounts of combined water. The hydration products were investigated using XRD and SEM/EDX. Both pozzolans increase the total porosities, when used without water reducers or superplasticizers. Both also decreased the compressive strengths at early ages. However, the strengths recovered with time exceed that of the reference Portland cement paste. Strength gain was more rapid in DK mixes than in SF mixes. The optimum content of each pozzolan varied with the water/solid ratios of the blends. Unreacted silica fume, in the highly-substituted mix (30%), caused a reduction in the mechanical properties by providing weak points for the crack propagation.

1. Introduction

Partial replacement of OPC by a pozzolanic material generally has a desirable effect on the strength [1–5] and the durability [6–9] of mortar and concrete. The advantages of silica fume (SF) as a pozzolanic material in blended cements have resulted in its worldwide use for a variety of applications [10,11]. However, two of the difficulties in using silica fume are its tendency to agglomerate and the high water demand. Thus, mix designs involving silica fume also typically use water reducers or superplasticizers [12–16]. Because of its tendency to agglomerate, SF may persist long after the principal hydration reactions have ceased [17,18], its high agglomeration strength makes it difficult to disperse satisfactory in the mix. Diamond [19] showed that undispersed silica fume agglomerates can induce, rather than prevent, alkali silica reaction (ASR) distress in certain instances. Mitchell [17] suggested that SF agglomerates rather than those of the individual SF particles control the pozzolanic activity. Agglomeration is also responsible for the high water demand; large amounts of mixing water are retaining in the interstices between the SF spheres. Bonen and Khayat [20] found that SF was able to retain 40% free water after its immersion. These observations have caused some authors [20,21] to suggest that the primary effect of SF is generated by its physical (micro-filler) properties rather than its pozzolanic properties.

Other highly reactive silicas have been used in attempts to obtain the desirable effects of SF in enhancing the strength and the durability [22–24]. For example, Chandra and Beryauist [22] found that a small amount of colloidal silica was sufficient to produce the same pozzolanic effect at early ages as the addition of larger amount of silica fume. Surface treatments have also been used to reduce the tendency of SF to agglomerate [25,26]. Treating SF with a silane coupling agent, Xu and Chung [25] obtained mortars with high workability and high tensile and compressive strengths even without a water reducing agent.

In a previous study [27], we compared the reactivities of silica fume and dealuminated kaolin (DK). It was found that, dealuminated kaolin has a higher surface area (90.5 m^2/g) and a much higher reactivity than silica fume especially at early ages. This higher activity of DK was a result of the higher surface area, of the structure of silica and of the presence of hydrated silica (silonal groups; Si–OH). It has been shown that silica in DK is present as sheets of large dimensions [28,29]. Silica with this structure tends not to agglomerate as severely as silica fume. This leads to high reactivity of DK using isothermal conduction calorimetry investigation [30]. Based on previous studies, we expect DK to show an effect similar to that of colloidal silica. If so it may used successfully to produce blended cements with desirable properties without the need of superplasticizers.

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The current investigation compares the effects of various replacement levels of silica fume and dealuminated kaolin without using superplasticizers on the properties of the blended cements.

2. Materials and methods

Silica fume (SF), with the BET surface area of 18.8 m^2/g, was obtained from Ferrosilicon Co. Edfo, Egypt. Dealuminated kaolin (DK), with BET surface area of 90.5 m^2/g, was obtained from Egyptian Shaba Co., Egypt. It is produced as a waste by-product of aluminum extraction from calcined kaolin by sulfuric acid. The detailed characterization of DK and SF is given in another paper [27]. The chemical composition of the Portland cement (PC) used is; CaO, 63.82, SiO_2, 20.88, Al_2O_3, 6.91, Fe_2O_3, 2.12, MgO, 2.31, SO_3, 1.89, K_2O, 0.21, Na_2O, 0.12 and L.O.I. 1.81 and the Blaine surface area is about 3500 cm^2/g.

The two pozzolans were used to replace 10%, 20% and 30% of Portland cement to produce blended cements. These mixes are designated as shown in Table 1. The pastes were prepared at water/solid ratio (W/S) of 0.30 and 0.50. The pastes were molded into 1 × 1 × 1-in specimens using stainless steel moulds. They were cured at 100% relative humidity for 12 h to attain initial setting, then cured at room temperature in water for 1, 3, 7, 28 and 90 days. Five samples from each mix were tested for compressive strengths at each hydration age. The phases present were determined by XRD using an automated diffractometer (Scintag, Inc., Sunnyvale, CA), at a step size of 0.02°, scan rate of 2 deg/min, and a scan range from 4° to 60° 2θ. The fracture surfaces were investigated by using scanning electron microscopy (SEM) model HITACHI S-3500N, equipped with secondary electron detector and EDX. All samples were coated with gold.

3. Physicomechanical properties

The compressive strengths of the various SF and DK blended cements are shown Figs. 1 and 2. The compressive strengths of all the blended cements are lower than that of the plain cement paste, during the early hydration (1–3 days). In addition, the strength decreased with increasing pozzolan content. The reductions in strength as a result of pozzolan substitution are more significant at the low W/S ratio (0.30).

At longer hydration times, the differences between the strengths of blended and plain cements are reduced for W/S = 0.30 SF-blends. The compressive strengths of the SF-blends, W/S = 0.50 and both DK-blends, increase beyond that of the control paste at 7 days. Mix SF20 (20% SF) produces the highest strength of silica fume blends and mix DK30 (30% DK) give the highest strength for dealuminated kaolin mixes.

The long term strengths of DK-blends exceed that of the control mix at both C/S ratios used. In general, DK improves the compressive strength more than SF especially at the low W/S ratio (0.30). Although there are no great differences in the strengths of DK-blends at each W/S ratio, careful comparison of Fig. 2a and b, shows the optimum strength increases with increasing the W/S ratio. The optimum mix at W/SW/S ratio 0.30 is DK20 (20% DK) and it shifts to mix DK30 (30% DK) at W/S ratio 0.50. These results indicate that the contribution of highly reactive pozzolans to the strength is governed by the rate of the clinker hydration, i.e. the rate of release of CH. Because these mixes were made without superplasticizers, the micro-filler effects [20,24,31] of these pozzolans were eliminated. Thus, the pozzolanic materials contribute to the mechanical properties only through the pozzolanic reaction.

Table 1
Pozzolanic cement mixes.

Mix.	PC	Dealuminated kaolin (DK)	Silica fume (SF)
Ref.	100	–	–
SF10	90	–	10
SF20	80	–	20
SF30	70	–	30
DK10	90	10	–
DK20	80	20	–
DK30	70	30	–

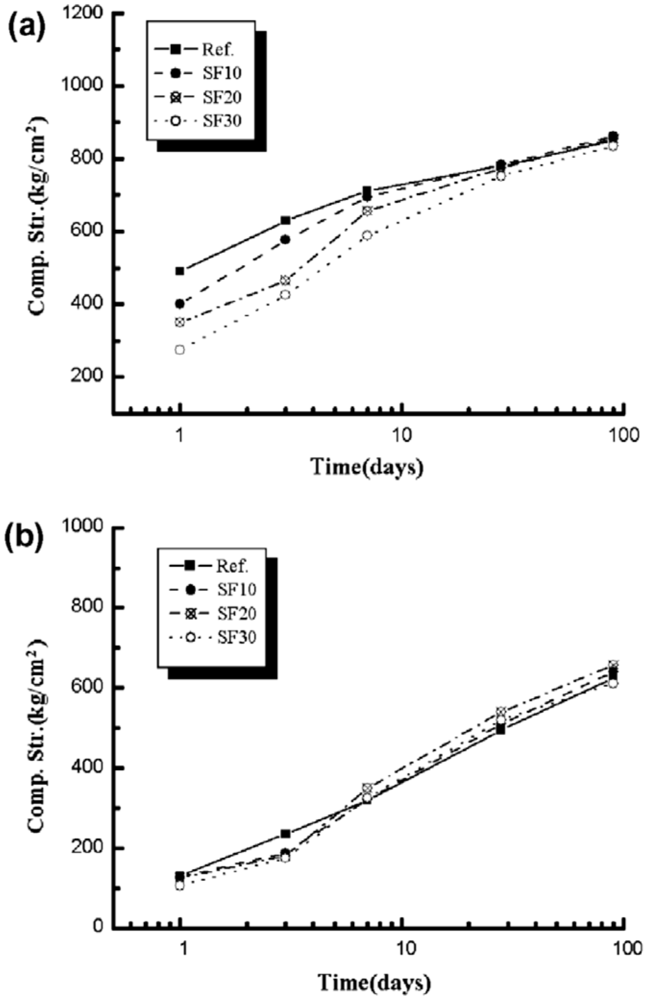

Fig. 1. Compressive strengths of silica fume blended cements: (a) W/S = 0.30 and (b) W/S = 0.50.

The apparent porosities of SF blends and DK blends hydrated for 90 days are shown in Table 2. The blends show a higher porosities than Portland cements, and the porosities increase with increasing pozzolan content. This indicates that these pozzolans do not decrease the porosities except when used with superplasticizers. Comparing porosities of SF blends and DK blends, shows that DK increases the total porosity more than SF in pastes prepared at the same W/S ratio. This due to the higher the surface area of DK (90.5 m^2/g).

4. Kinetics of hydration

The variations in the free lime contents for the mixes with time are shown in Figs. 3 and 4. The control samples show the highest free lime contents at all ages and the free lime contents increase continually with increasing hydration time. The free lime contents of blended mixes are lower than those of the control sample. This is

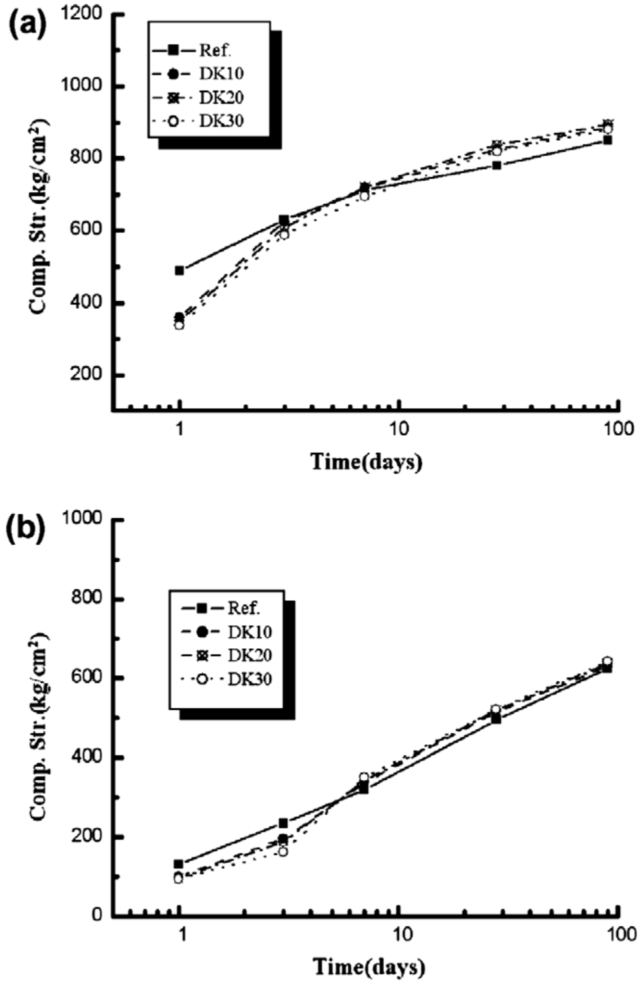

Fig. 2. Compressive strengths of dealuminated kaolin blended cements: (a) W/S = 0.30 and (b) W/S = 0.50.

Table 2
Apparent porosities of SF and DK blended cement mixes at 90 days.

Mix	Porosity (%V)	
	W/S = 0.3	W/S = 0.5
Ref.	32.0	45.2
SF10	33.3	47.9
SF20	33.9	51.1
SF30	35.6	51.0
DK10	34.8	45.2
DK20	38.4	50.1
DK30	39.9	51.1

due to the dilution effect as will as the reaction of free lime with pozzolan. The blends prepared at a W/S ratio of 0.50 show a higher free lime contents than those prepared at a W/S ratio of 0.30. This due to the more rapid rates of hydration at the higher W/S ratio [32].

There is a marked increase in the consumption of free lime with increasing pozzolan content. Regardless of the extent of replacement and W/S ratio, the free lime contents passed through maxima before decreasing. Mix DK30 with W/S = 0.30 showed a maximum during the first day. A comparison of the change in the free lime contents for the DK mixes and SF mixes shows DK reacts faster with lime than SF at early ages. This explains the more rapid contribution of DK to the strength gain in the low W/S ratio pastes. However at the later ages SF mixes fix higher amounts of lime than DK mixes. This is due to the higher the reactive silica content of the SF.

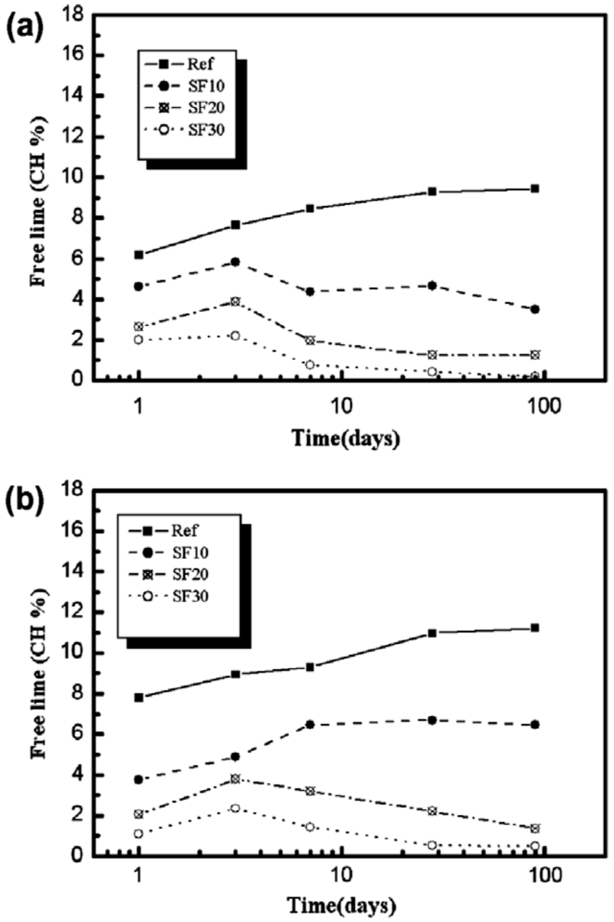

Fig. 3. Free lime contents of silica fume blended cements: (a) W/S = 0.30 and (b) W/S = 0.50.

The results of combined water determinations for each mix at the different curing times are given in Tables 3 and 4. As expected the combined water increases with increasing hydration time for the Portland cement. At early ages of hydration (1 day), the combined water contents of the low W/S ratio blended cements are lower than the Portland cement paste. This is due to the reduction in the cement content and the pozzolan–lime reaction has not proceeded to the degree required to balance this effect. The reverse occurred at 1 day in the high W/S ratio samples; more rapid cement hydration produced more calcium hydroxide (CH), the pozzolanic reaction was more extensive and produced more calcium silicate hydrates (C-S-H), thus combining more water.

All W/S = 0.50 pozzolan mixes show a decline in combined water at 7 days of hydration. However, only mixes with high pozzolan contents (20% and 30%) show such decline in samples made with W/S = 0.30. Generally, at 90 days of hydration, combined water contents decrease as the pozzolan contents increase and only mixes with 10% pozzolan (SF10 and DK10) show higher combined water contents than those of the reference mixes.

5. X-ray diffraction analysis (XRD)

Figs. 5 and 6 show the XRD patterns of W/S = 0.30 cement pastes containing 10% and 30% of SF and DK, respectively, and hydrated up to 90 days. The XRD pattern of the cement paste is given for comparison. The results are in a complete agreement with the

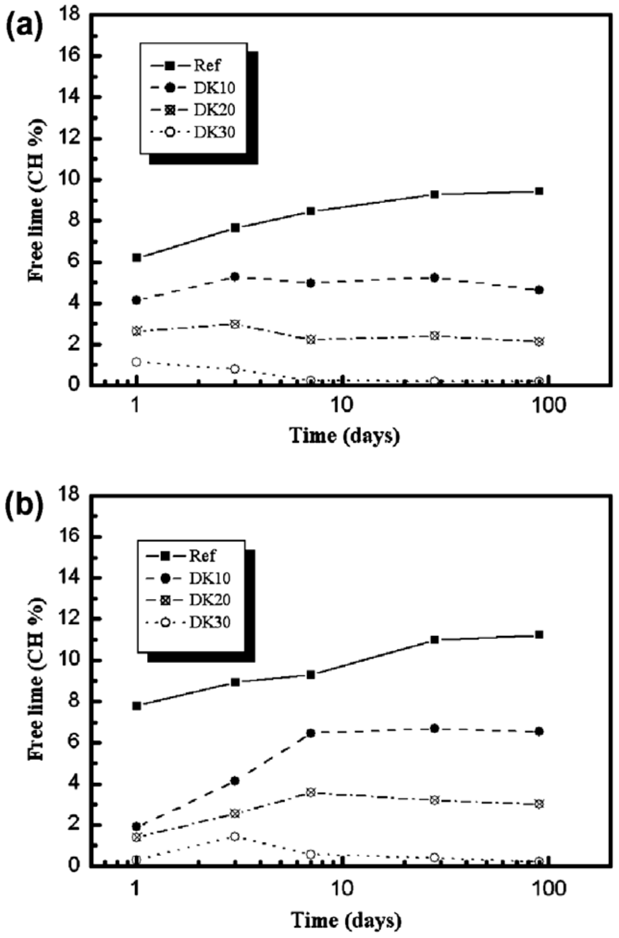

Fig. 4. Free lime contents of dealuminated kaolin blended cements: (a) W/S = 0.30 and (b) W/S = 0.50.

Table 3
Combined water contents of SF blended cement mixes.

Mix	W/S ratio	Combined water (%)				
		1 day	3 days	7 days	28 days	90 days
Ref.	0.3	9.2	9.8	11.5	11.6	11.8
SF10	0.3	8.7	9.9	11.3	13.0	13.9
SF20	0.3	8.6	9.3	11.9	10.8	10.1
SF30	0.3	8.7	8.5	10.6	10.6	9.9
Ref.	0.5	10.0	10.5	12.0	12.4	14.2
SF10	0.5	10.2	11.7	13.8	12.6	14.4
SF20	0.5	11.3	11.7	13.6	12.4	13.6
SF30	0.5	12.3	12.5	14.3	11.7	13.2

Table 4
Combined water contents of DK blended cement mixes.

Mix	W/S ratio	Combined water (%)				
		1 day	3 days	7 days	28 days	90 days
Ref.	0.3	9.2	9.8	11.5	11.6	11.8
DK10	0.3	9.0	11.5	12.0	12.2	11.9
DK20	0.3	8. 5	9.7	11.9	11.6	10.8
DK30	0.3	8.4	10.5	11.4	10.7	10.4
Ref.	0.5	10.0	10.5	12.0	12.4	14.2
DK10	0.5	12.5	12.5	13.6	13.2	14.7
DK20	0.5	11.0	11.5	13.0	11.8	14.0
DK30	0.5	10.5	11.3	13.2	10.4	13.1

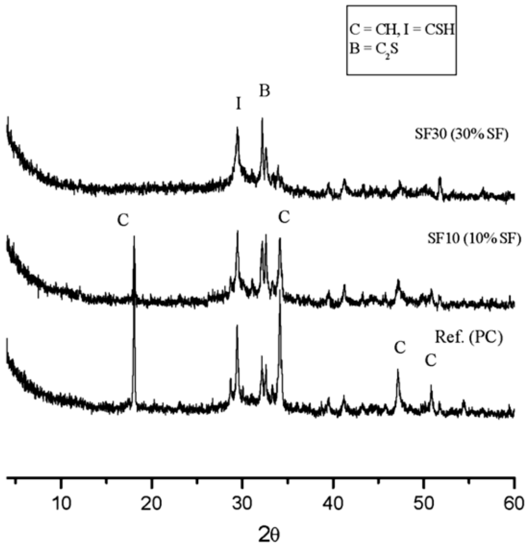

Fig. 5. XRD patterns of silica fume blended cements hydrated for 90 days (W/S = 0.30).

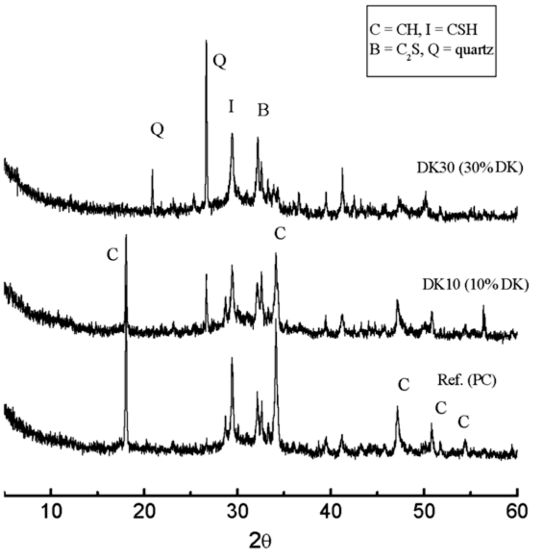

Fig. 6. XRD patterns of dealuminated kaolin blended cements hydrated for 90 days (W/S = 0.30).

free lime determination, the CH peaks decrease with increasing pozzolan content and completely disappear in mixes with 30% replacement (SF30 and DK30). The peaks associated with C-S-H intensify with increasing pozzolan content. C_2S is the only unhydrated cement phase still detected at 90 days of hydration.

6. Scanning electron microscopy (SEM)

The fracture surfaces of cement pastes hydrated for 90 days were studied using SEM/EDX. A previous study [31] of the fracture characteristics of plain cement pastes made at low W/S ratio revealed that crack propagation takes a path through the CH-rich regions facilitated by the characteristics parallel cleavages planes of CH. CH crystallites also act as point defects that help in the crack initiation and crack propagation in the cement matrix [32].

Fig. 7 shows sample made with 30% SF at W/S = 0.30 and hydrated for 90 days. The fracture surface shows regions

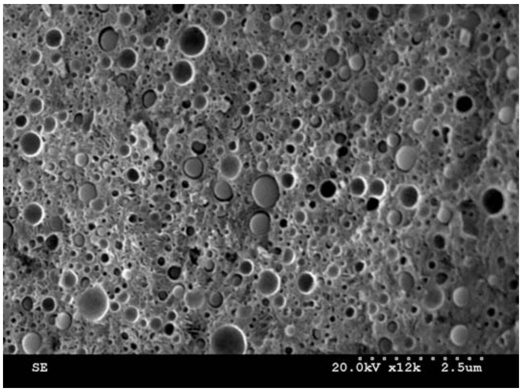

Fig. 7. Microstructure of mix SF30 with W/S = 0.30, hydrated for 90 days.

containing considerable amounts of unreacted silica fume particles as a result of its poor dispersion. This silica fume remains unreacted after the consumption of most of the lime produced from the cement hydration and these regions act as point defects in the cement matrix. The presence of unreacted silica appears to be greater in the low W/S samples.

Fig. 8 shows the SEM micrograph of DK30 mix made with W/S ratio of 0.30 and hydrated for 90 days. The consolidated structure of C-S-H contains tubular pores with diameters less than 0.25 μm.

Fig. 9 shows the SEM micrograph of DK30 mix made with W/S ratio of 0.50 and hydrated for 90 days. Like all the high W/S ratio samples, a network C-S-H and micropores are distributed over the fracture surface. In these samples, crack propagation takes a path through the capillary pores and the points of contact of the hydration products, which represent the weakest points in the cement matrix. This type of fracture has been named interparticle fracture by Berger et al. [33]. Dalgleish et al. [34] found the same type of fracture in young cement pastes due to the weak bonding between hydrated particles.

7. Discussion

The reductions in early compressive strength and increases in total porosity of the pozzolan blends, indicate that the later improvements in compressive strength are due to the pozzolanic effect. The pozzolanic effect in this regard can be concluded as the consumption of free lime, the formation of additional C-S-H, the change in its microstructure and chemical composition (reduction in calcium/silica ratio). The lowering of the C/S ratio of C-S-H may result in an increase in the mechanical properties [35]. The reduction in the early strength can be accounted for in terms of reduction in cement contents and increased porosity with increasing the pozzolan content at the same W/S ratio.

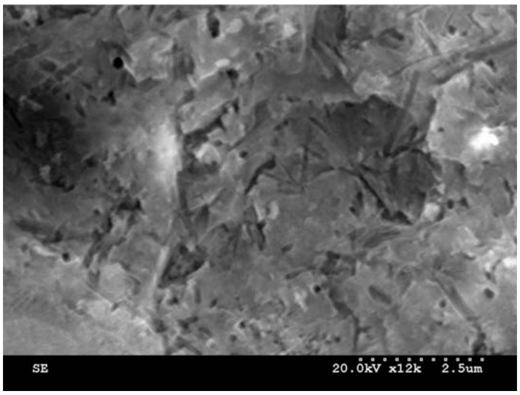

Fig. 8. Microstructure of mix DK30 with W/S = 0.30, hydrated for 90 days.

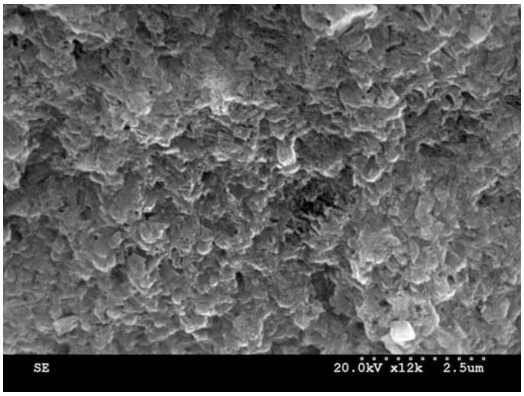

Fig. 9. Microstructure of mix SF30 with W/S = 0.50, hydrated for 90 days.

The pozzolanic contribution to the compressive strength is governed by the rate of CH release during hydration, thus it is hydration that controls the extent to which the pozzolans gain strength beyond that of the control in the blends studied. At W/S = 0.50, Portland–silica fume pastes showed this effect at 7 days. However those made at W/S ratio of 0.30 do not show this effect until 28 days. DK-blends show the effect at earlier ages than SF-blends, especially at low W/S ratio (0.30). This appears to be due to its more uniform dispersion.

At 90 days, the optimum DK and SF contents that give the highest strengths for both blends were determined by the W/S ratio, the optimum SF contents are 20% and 30% for W/S ratio of 0.30 and 0.50, respectively. Where the optimum DK contents are 10% and 20% for W/S ratio of 0.30 and 0.50 respectively. This shift in DK optimum content to higher values is due to the presence of less reactive silica in the DK.

The variations in combined water of pozzolan-containing pastes are complicated, because they are the result of three different processes, excluding the hydration reactions of cement phases:

(1) Reaction of the released free lime with pozzolan to produce additional C-S-H with variable water contents according to each condition [36].
(2) In high pozzolan mixes, at later ages of hydration, the remaining reactive silica reacts with C-S-H previously produced from clinker hydration to produce a C-S-H with a lower C/S ratio and a lower water content [32,37]. Decreasing the C/S ratio, cause polymerization in the hydrated layer silicates which accompanied by water loss [38].
(3) In low pozzolan mixes, at later ages of hydration, the CH produced after the pozzolan is consumed may react directly with weakly acidic hydroxylated C-S-H through a condensation mechanism. This causes an increase in combined water content as the C/S ratio increases [32,37].

These three processes act at different stages of hydration depending on the pozzolan content and the water/solid ratio used. Thus, the change of the maxima in free lime contents with the pozzolan content and the W/S ratio depends on the relative rates of hydration (release of CH) and pozzolanic reaction (consumption of CH).

8. Conclusion

Both SF and DK replacements decreased the compressive strengths at early ages. However, the strengths recovered with time exceeding that of the reference Portland cement paste. Strength gain was more rapid in DK mixes than in SF mixes. The optimum content of each pozzolan varied with the water/solid ratios of the blends. Unreacted silica fume, in the highly-substituted mix (30%), caused a reduction in the mechanical properties by providing weak points for the crack propagation.

The main conclusion of this investigation is that the rate of clinker hydration governs the rate of pozzolanic reaction contribution to the strength in the case of high reactivity pozzolanic materials, like silica fume and dealuminated kaolin. In the absence of a superplasticizer the improvement in the strengths of SF-cement blends is due to the pozzolanic reaction and the improvement of the structure and morphology of the hydration products in spite of the persistence of agglomerates.

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