A RAPID AND SIMPLE TEST SCHEME TO ASSESS THE EFFECT OF FLY ASH ON KEY PROPERTIES

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ABSTRACT

The use of pulverized fuel ash (pfa) as a partial cement replacement is growing as it improves many properties. However, its effect on key properties, such as compressive strength, drying shrinkage and porosity, remains to be dependant on the particular pfa utilized. It is necessary to routinely test for these key properties when pfa is to be employed. In this investigation a simple and rapid test scheme is proposed for this purpose. The scheme involves testing the flow of fresh pfa mortars. In addition, pfa mortars, with different w/c ratios and percentages of pfa, are tested for compressive strength at 7 and 28 days after curing in accordance with a number of curing regimes. Finally the drying shrinkage and porosity of the pfa mortar are tested at 28 days. An acceptance criteria for the proposed test scheme has also been set. One pfa was tested in accordance with this scheme as a demonstration. The results of the tested pfa were thoroughly analyzed in order to gain insight into the action of pfa in cement based matrices.

INTRODUCTION

The use of mineral admixtures, such as slag, silica fume and fly ash (pfa) in concrete is steadily rising for many reasons [1]. It was found that pfa reduced the water demand of mixes [2], decreased the early heat output compared to ordinary Portland cement (OPC) [3], improved the sulfate resistance, provided 20-50% of low calcium ash is used [4], controlled the expansion of heat cured mortars [5], enhanced the resistance to alkali silica reaction [6] and reduced the tendency for chloride ion ingress [7].

Conflicting reports have been cited in the literature regarding the effect of pfa on the compressive strength of cement based matrices. Many investigators like Patoary and Nimityongskul [8] found that the inclusion of pfa reduced the early strength and the reduction was more significant with the increase of percentage replacement. However, Kearsley and Wainwright [9] reported up to 67% of cement could be replaced with pfa without any significant reductions in strength. On the other hand, Ramezanianpour and Malhotra [10] reported that concretes containing pfa were more sensitive to poor curing than

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the OPC counterparts, with the sensitivity increasing with the increase in replacement ratio. But Gebler and Klieger [11] found that at the early ages the compressive strength of pfa concretes was essentially unaffected by moisture availability. More over, under the worst curing conditions pfa concretes showed a slow but steady strength gain, but their OPC counterparts did not [12]. A number of investigators found that the compressive strength of pfa mortar or concrete was significantly enhanced with the increase in curing temperature [13]. However, Brooks and Sikhararulidze [14] found that the influence of temperature on strength and does not always follow the trend of a higher initial rate and lower ultimate value as the curing temperature is raised. Different parameters have been noted as the main causes of strength gain in pfa systems. It has been reported that the final strength was roughly proportional to the content of active silica in the concrete volume [15]. Alternatively, the pozzolanic reaction was found to increase with the increase in pfa fineness [16]. Therefore, it would appear that compressive strength of a certain pfa/cement mixture is a unique function that depends on many parameters and should assessed on an individual basis.

The drying shrinkage of cement based matrices depends on the fractional volume of the paste, cement content and type and volume of aggregates. Since replacing cement by pfa on weight basis, while keeping the water content constant, increases the paste volume, the drying shrinkage of a pfa mortar should be slightly more than that of a plain OPC one [17]. It is also known that pozzolans, like pfa, tend to increase the volume of fine pores leading to the increase in drying shrinkage because shrinkage is governed by the water held in small pores (3-20 nm) [18]. However, many investigators found that the drying shrinkage was reduced when pfa is used [19-22]. Whereas, Gebler and Klieger [11] and Brooks [23] concluded that fly ash had no effect on the drying shrinkage. On the other hand, Bouzoubaa et al. [24], found that the drying shrinkage of pfa concrete at 28 days was lower than the OPC one, but this was reversed at 112 days. The authors of the above mentioned studies gave no explanation for these observations. Therefore, it is necessary to examine this property for each pfa.

Manmohan and Mehta [25] reported that at 28 days, all cement pastes with up to 30% pfa had a coarser pore structure than the control OPC paste. But at 1 year, the situation was reversed indicating substantial progress in the pozzolanic reaction. More pore refinement was evident with higher pfa percentages. Cabrera et al. [26] found that at 28 days, curing at 20 °C, results in a higher porosity for pfa mortars, but curing at higher temperatures results in lowering the porosity compared to the OPC mortars. Soroushian and Alhozaimy [27] reported that partial substitution of cement with fly ash causes a reduction in the volume of large capillary pores. Poon et al. [28]

reported that pfa inclusion increased the total porosity, but the pore system was refined and the interfacial porosity was significantly reduced, for mortars with w/c =0.3 and 0.5 at ages 28 and 56 days. These observations were magnified with the increase in pfa substitution level. However, Pandey and Sharma [29] found that the mean pore diameter is increased but the total porosity remained the same with pfa inclusion for sample ages up to 90 days. Thus, the effect of pfa on total porosity is unique to the pfa used.

From the foregoing discussion it became clear that it is difficult to predict the performance of pfa by it characterization (chemical, physical and phase analysis) only. The properties of pfa mixtures should be investigated by tests prior to its utilization. This paper proposes a simple and rapid scheme for this purpose. The scheme covers key controversial properties (i.e. flow, compressive strength, drying shrinkage and porosity) and takes approximately one month to complete. To demonstrate the proposed test scheme, one pfa was tested. More importantly an attempt was made to give enlightened explanations for the effect of pfa on some of the tested properties with the aim of providing a better understanding of the factors affecting the action of pfa.

PROPOSED SCHEME PHILOSOPHY

The proposed scheme covers tests on compressive strength for mortars having w/c ratio of 0.3, 0.4 and 0.5 with and without the pfa to be assessed. The first part (Series I) covers testing the compressive strength of mortars with 0, 10, 20, 30, 40 and 50 % pfa which are either air cured or moist cured for 7 or 28 days at 20, 40, 60 °C. These curing regimes cover different climate/work conditions (i.e. poor curing for air cured samples, moderate climate for moist curing at 20 °C, temperate climate for moist curing at 40 °C and finally hot weather for moist curing at 60 °C). Curing for 7 days only would simulate the work conditions where moist curing is terminated at an early age. The second part (Series II) covers testing the effect of pfa on the flow of fresh mortar and its effect on the drying shrinkage and porosity. The w/c ratio was 0.4, 0.5 or 0.6, but the pfa replacement ratio was constant at 30% in this part. If pfa is to be used at a different replacement level, the actual percentage should be employed at this part. This part is designed to predict the effect of pfa on the key properties that would have a profound effect on the placing conditions (flow), long term volume stability (drying shrinkage) and durability (porosity). It should be stressed that the scheme does not exclude the need for trial mixes. It is merely a pfa assessment tool. Trial mixes should be carried out after the pfa passes the scheme in order to obtain a mix suitable for the project's specifications.

SCHEME TEST PROGRAM

Materials

Ordinary Portland cement conforming to ESS 373/1991 [30] was used in preparing the test samples. The pulverized fuel ash (fly ash or pfa) was obtained in a single batch. Properties of the pfa used are shown in Table 1. The fine aggregate was a single sized sand obtained by passing natural sand, conforming to ESS 1109/2002 [31], through sieve 2.36 mm and retaining it on sieve 1.18 mm. Tap water was used in mixing and curing the test specimens.

Mortar Mix Proportions

Two series of mortars were prepared. In this investigation, the term cementitious materials shall refer to the OPC only in the OPC mortars, but in the pfa mortars it will refer to the sum of weights of OPC and pfa. In Series I, the mortar mixes had water to cementitious materials (W/C) ratio of 0.4, 0.5 or 0.6 and cementitious materials to sand ratio of 1:2. When pfa was used it replaced 10, 20, 30, 40 or 50% of the cement. In Series II, the cementitious materials to sand ratio was also 1:2, the pfa replacement ratio was constant at 30%, but the W/C ratio was 0.4, 0.5 or 0.6.

Mortar Preparation

The mortars were mixed in accordance with EC 1-14 [32]. All samples in Series I were 50 mm cubes, as this series was devoted to studying the compressive strength of the mortars under different curing regimes. The samples in Series II were 25 x 25 x 285 mm prisms with or without gauge studs fixed at the center of the end faces. The gauge length was 250 mm $\pm$ 2.5 mm. The prisms with gauge studs were used for length change determination during the drying shrinkage test, whereas those without the studs were used for total porosity determination. The samples were placed in their moulds under wet burlap covered with plastic sheets for 24 hours. They were then demolded and cured in accordance with the chosen curing regime until testing.

Curing and Testing of Mortar Samples

After the samples of Series I were demolded, they were subjected to one of the four curing regimes to applied in test scheme. These were air curing at room temperature, standard water curing at 20 ^{o}C, water curing at temperature of 40 ^{o}C and water curing at temperature of 60 ^{o}C. The Series I samples were cured in accordance with these regimes until the age of 7 or 28 days when they were tested for compressive strength. Each reported compressive strength result was the mean value obtained from testing three cubes.

During the preparation of the samples for Series II the flow of the fresh mortar was measured using the mortar flow table in accordance with the method described in EC 1-9 [33]. The drying shrinkage of the prisms with gauge studs was measured after storing the samples till the age of 28 days in a dry cabinet at room temperature. Silica gel was used to desiccate the humidity from the cabinet. The drying shrinkage was measured using an apparatus similar to that described in EC 1-20 [34]. Each reported drying shrinkage result was the mean value obtained from testing three identical mortar prisms.

The prisms without gauge studs were water cured at room temperature until the age of 28 days when they were tested for porosity. At that age the samples were dried in an oven at 105 ^{o}C till constant weight to determine the amount of evaporable water. The total porosity can be calculated upon dividing the evaporable water by the apparent volume of each prism. Each reported porosity result was the mean value obtained from testing three identical mortar prisms.

RESULTS AND DISCUSSION FOR THE SAMPLE PFA TESTED IN ACCORDANCE WITH THE PROPOSED SCHEME

Results For Series I Samples

Effect of w/c ratio and age of testing on compressive strength

The compressive strength of mortars having different w/c ratios and 0 or 30% pfa are shown in Figures 1-4. Each figure contains the results for samples cured in accordance with one of the curing regimes adopted in the current study (i.e. air curing, water curing at 20, 40 and 60 °C). The trend of the traditional w/c-compressive strength relationship is clear in Figures 1 to 4, however, the age effect needs further analysis.

It can be seen from Figures 1 to 4 that, at a number data points for OPC mortar, the curves are close to one another, indicating that the strength increase between 7 and 28 days was small. This may be attributed to the changes in the cement industry, which lead to the increased proportion of C_3S at the expense of C_2S in modern cements. As a result modern cements develop strength more quickly and gain little strength with time [35]. Some pfa mortars exhibited a more significant increase in strength between 7 and 28 days, especially when water cured at 20 and 60 °C.

Effect of pfa replacement level on compressive strength

The effect of pfa content on the compressive strength is shown in Figure 5 for the samples cured for 7 days and in Figure 6 for samples cured for 28 days. The results shown in Figures 5 and 6 are for mortar samples having w/c = 0.5. It can be seen that, the results for air and water cured samples at 20 °C and 40 °C for 7 days exhibited good correlation with the % pfa, and therefore these results were shown as fitted straight lines. Other results did not exhibit significant correlation.

It can be seen from Figure 5 that there was a gradual decrease in the compressive strength with the increase in pfa replacement ratio. This is expected because of the reduction of the cement content, and hence hydration products, which is mainly responsible for strength development at this early age in the mortar. The strength values in Figure 6 are naturally higher than those in Figure 5, however, it is clear that there was no definite effect of the pfa replacement ratio on the compressive strength. This is in line with the findings of Kearsley and Wainwright et al. [9], who reported that the compressive strength of foamed concrete was not significantly affected, with pfa replacement ratios up to 67%. With certain types of pfa, the strength can even increase [19]. The comments of Maltais and Marchand [36] may shed light on this behavior. They stated that pfa tends to increase the rate of cement hydration at the early ages. Similar observations were noted by Lam et al. [37] who found that the degree of hydration of cement is increased in pastes with high pfa percentages due to the higher effective water to cement ratio. In addition, Tanpagsit et al. [38] reported that the contribution of pfa to compressive strength is derived from the pozzolanic reaction and the pfa packing effect. The former is usually small in the early ages, but the latter is immediately apparent and was found to be 22% of the OPC mortar strength, when pfa replaced 20% of the cement in the mortar. It is expected that this packing effect would be higher for higher pfa replacement ratios. Seemingly, in the mortars prepared using the sample pfa under assessment, this packing effect coupled with the increased rate of OPC hydration, had counteracted the expected reduction in compressive strength due to pfa inclusion at 28 days.

Effect of curing regime on compressive strength

The compressive strength results for the different curing regimes are shown in Figure 7. It can be seen that there is little difference between all the water curing regimes, but there is some increase in the observed strength with the application of water curing compared to that observed for air cured samples. However, the pfa mortars did not suffer more severely than the OPC ones when deprived from water curing. This is contrary to the findings of many investigators (e.g. Ramezanianpour and Malhotra, [10]), who reported that concretes containing pfa were more sensitive to poor curing than the OPC counterparts. But the current results are in line with the findings of Gebler and Klieger [11], who reported that at the early ages the compressive strength of pfa concretes was essentially unaffected by moisture availability. Moreover, curing at 40 and 60 °C improved the compressive strength of both types of mortars in a similar fashion. It has been reported that the hydration and pozzolanic reactions are accelerated with the increase in temperature. For every 10 °C increase in temperature, these reactions are doubled [26]. However, the magnitude of the increase in compressive strength, for mortars in the current study subject to elevated temperature water curing, was not very high. In any case it can be seen from Figure 7 that the difference between pairs of OPC and pfa mortars was not significant in many situations.

Results For Series II Samples

The test results for this series are tabulated in Table 2. The last column of the Table 2 shows the percentage change in the studied property due to replacing 30% of the cement by pfa in the test mortars. The results shall be discussed in detail in the next sections.

Effect of pfa on mortar flow

It can be seen from Table 2 that the use of pfa increased the percentage flow in the w/c = 0.4 and 0.5 mortars by more than 27%. Popovics [39] attributed this to the fact that the OPC/pfa system would have an optimum particle size distribution that would improve the flow of mortars. Paris-Mora et al. [40] found that the flow increases with the increase in the specific surface of the ash because of the lubricating effect of the particle spherical shape. In other words, pfa behaves as ball bearings and reduces the internal friction in the mortar. Helmuth [41], however, proposed that the fine pfa particles are adsorbed on portions of the cement particle surfaces in an action similar to that of the water reducing admixtures. Because of this phenomenon, many researchers considered that pfa can be used as a plasticiser [42].

It is interesting to note that for the w/c = 0.6 mortar, the use of pfa increased the flow by 2% only compared to the OPC mortar. It can be argued that at this w/c ratio the particles in the mix would be already far apart, therefore the inclusion of pfa would not have such a dramatic effect as discussed above.

An important observation can be deduced from calculating the increase in flow as the w/c ratio was increased from 0.4 to 0.6. For the OPC mortar the increase in flow was 591%, but for the pfa mortar the increase was only 453%. Neville and Brooks [43] argued that the specific gravity of pfa is typically 1.9 to 2.4, which makes it much lower than that of cement (3.15). As a result, replacing cement by pfa on weight basis results in a considerably greater volume of cementitious materials in the mix. Therefore, the increase in W/C ratio would have a greater impact on flow in the mortar without pfa.

Effect of pfa on drying shrinkage

It can be seen from Table 2 that the inclusion of pfa in the mortars of this study having w/c = 0.4 and 0.5 reduced the drying shrinkage strain at 28 days. The reverse was true for the w/c = 0.6 mortar. The authors of the current investigation will attempt to propose an explanation to the results of the current investigation, which perhaps can also help in understanding the discrepancies in previous investigations on the subject.

To start with, the authors of the current study believe that little or no pozzolanic reaction took place in the drying shrinkage prisms up till the age of 28 days when they were tested for drying shrinkage. It should be remembered that these prisms were kept in room temperature in a dry chamber. This belief is based on the findings of Hanehara et al. [44], who measured the pozzolanic reaction ratio in mixes kept under similar conditions at 28 days and found it to be 0 %. Therefore, the observed reduction in shrinkage must have resulted from other causes. It has been suggested that the drying shrinkage of cement is increased with the increase in C_3A and alkali contents in the cement [45]. Indeed He and Li [46] found that the restrained cracking sensitivity of a cement based material is increased with the increase in alkali content. Pfa, in some cases, has a lower alkali content than OPC, and its inclusion would certainly dilute the C_3A phase in the mortar system. The pfa used in the current investigation, had a very low alkali content (see Table 1). This may explain the reduced shrinkage when the pfa under assessment was used in the mortars. The fact that pfa seems three times more efficient, in controlling the drying shrinkage of the w/c=0.5 mortar compared to the w/c= 0.4 one, merely reflects the increased water content in the w/c=0.5 mix, hence the action of pfa was more apparent. On the other hand, with the excessive water content in the w/c = 0.6 mix, pfa increased the shrinkage as moisture was lost more readily from the pfa mortar because of its lower content of hydration products, at the time of testing. It can be argued that the results of previous studies regarding the effect of pfa inclusion on drying shrinkage, may be explained, by the same analogy, in light of the alkali content of the pfa used and its replacement ratio. Moreover, the results of the study by Bouzoubaa et al. [24] can be explained by the fact that the pozzolanic reaction products formed by the late age of 112 days caused a refinement of the pore system and at that age the pfa mixes exhibited the expected increase in drying shrinkage.

Effect of pfa on porosity

Table 2 shows the porosity results for mortars with or without pfa cured in water for 28 days. It is clear that the increase in w/c ratio from 0.4 to 0.6 resulted in an increase in total porosity by 76.9 and 62.4% for OPC and Pfa mortars, respectively. Therefore, pfa inclusion controlled the increase in porosity due to the increase in w/c ratio. It has been established that the amount of $Ca(OH)_2$ increases with the increase in w/c ratio of the paste [47]. As a result, the reaction of pfa was found to increase with the increase in w/c ratio [44]. Therefore, it can be postulated that some pozzolanic reaction took place in the w/c = 0.6 mortar of this study and this has been reflected in the lower increase in porosity compared to the OPC counterpart. This may be considered valid even though the pozzolanic reaction would still be at an early stage at this age.

It can be seen from Table 2 that the pfa inclusion resulted in an increase in the total porosity for all w/c ratios in this study. It should be noted that in this study, the total porosity was obtained from measuring the evaporable water content in the mortars. Hussain and Rasheeduzzafar [48] found that under the same curing and w/c ratio the pfa concrete had higher evaporable water content. In addition, the reaction of pfa would be limited at 28 days, when the mortars of the current study were tested for porosity, hence increased total porosity of pfa mortars was observed.

It can be seen that increasing the w/c ratio from 0.4 to 0.5, resulted in a higher increase in porosity with pfa inclusion. This is in line with results of Poon et al.[28] for 25% pfa substitution level at 28 days. They found that mortars with w/c ratio of 0.3 exhibited less than half the increase in porosity for mortars with w/c = 0.5. However, with w/c = 0.6 mortar pfa inclusion resulted in a lower increase in porosity. Again this may be attributed to the occurrence of some pozzolanic reaction at this w/c ratio due to the availability of a higher amount of $Ca(OH)_2$ in the system.

PROPOSED SCHEME ACCEPTANCE CRITERIA

The authors propose that, in order to pass the test scheme, the tested pfa should not reduce the compressive strength of the test mortar by more than 40% and 30% of the strength of control OPC counterpart at 7 and 28 days, respectively. These values are for the 30% pfa mortar having w/c ratio of 0.5. The values should be adjusted accordingly with different replacement levels and w/c ratios, for example up to 60 and 50% reductions at 7 and 28 days may be allowed for replacement levels of 50% and w/c = 0.4, respectively. The flow should be increased with all pfa samples tested. The drying shrinkage should not be increased by more than 20% of that for the control OPC mortar, whereas the porosity should not be increased by more than 30, 40 and 50% of that for the OPC control mortar for mortars with w/c ratio of 0.4, 0.5 and 0.6, respectively.

SUMMARY AND CONCLUSIONS

It has been established that the use of pulverized fuel ash as a partial cement replacement improves the flow or workability of fresh mixes, reduces the early heat evolution, enhances the resistance to alkali aggregate reaction, sulfate and chloride attack and controls the expansion of heat cured samples. However, contradicting reports were found in the literature regarding the effect of pfa on compressive strength, especially with regards to development rate and the effect of curing at higher temperatures, drying shrinkage and porosity. It became clear that the effect of pfa on these key properties is unique to the pfa used. A simple and rapid test scheme is proposed to routinely test the pfa before it is employed. The scheme takes approximately one month to complete. Mortar samples are prepared having different water to cementitious materials ratio and various pfa replacement levels. Different curing regimes are applied. The flow of the fresh mortar is measured. The compressive strength is tested at 7 and 28 days, whereas the drying shrinkage and total porosity are determined at 28 days. An acceptance criteria for the tested pfa samples has been proposed.

One pfa was assessed using the proposed scheme. Although some of the results of the tested pfa do not agree with the reported results in many literature reports, there is always a logical explanation for these results. The fact that any pfa exhibits unexpected results only proves the importance of running each pfa through the proposed test scheme. Where applicable enlightened explanation were provided for the reported results (e.g. the drying shrinkage may be mainly dependant on the alkali content of the pfa and age at testing). It is important that the test results are always thoroughly analyzed with care and understanding.

The pfa tested in accordance with the proposed scheme increased the flow of the fresh mortar to varying degrees depending of the w/c ratio. The compressive strength of the pfa mortar exhibited some reduction with the increase in pfa replacement ratio at 7 days, but was comparable to that of the OPC mortar at 28 days. The curing regimes employed had a similar influence on the mortar with and without pfa. The drying shrinkage was reduced with pfa inclusion for the w/c=0.4 and 0.5, but an opposite result was recorded for the w/c =0.6 mix. The total porosity was increased by different magnitudes depending on the w/c ratio. Where applicable these observations were explained. After examining the results of the tested pfa it is concluded that the tested pfa may be used as a partial cement replacement. It is recommended that the proposed test scheme is applied for the assessment of pfa.

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Table 1 Properties of pulverized fuel ash.

Oxide composition (%)	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	TiO_2	MnO	P_2O_5
	2.9	51.4	24.5	13.9	0.8	0.9	1.7	0.5	1.1	0.1	0.4
Loss on Ignition	1.8										
Physical Properties	Fineness, % retained on 45 μm sieve	5.7	Specific gravity	2.44							

Table 2 Test results for the Series II samples

Studied Property	**W/C ratio**	**Type of Mix**		**(2) – (1) / (1) (%)**
		OPC (1)	**PFA (2)**	
Flow (%)	0.4	20.0	25.5	27.5
	0.5	58.9	75.0	27.3
	0.6	138.2	141.0	2.03
Drying Shrinkage (microstrain)	0.4	293	273	- 6.8
	0.5	430	351	- 18.4
	0.6	898	1016	13.1
Porosity (%)	0.4	8.00	9.85	23.1
	0.5	9.85	12.92	31.2
	0.6	14.15	16.00	13.1

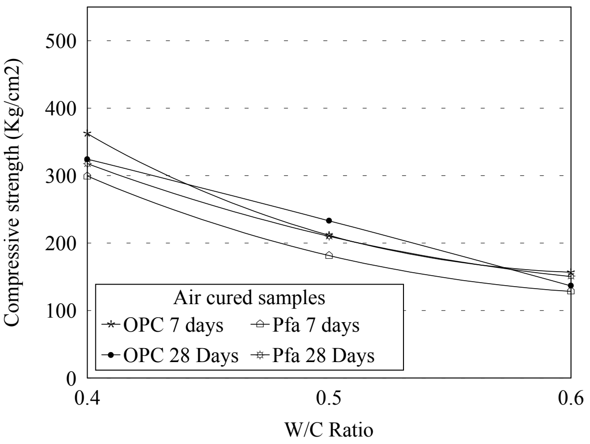

Figure 1 Compressive strength for air cured mortars

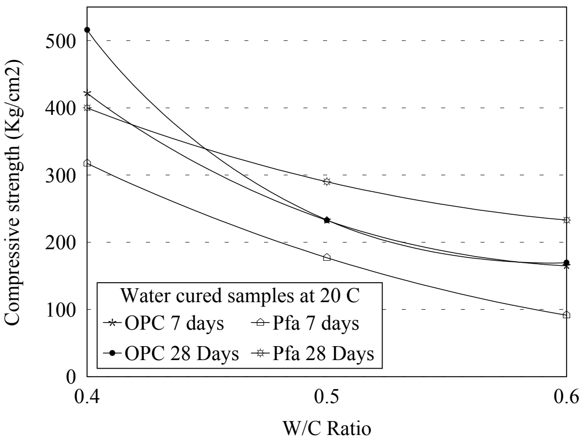

Figure 2 Compressive strength for water cured mortars at 20 °C

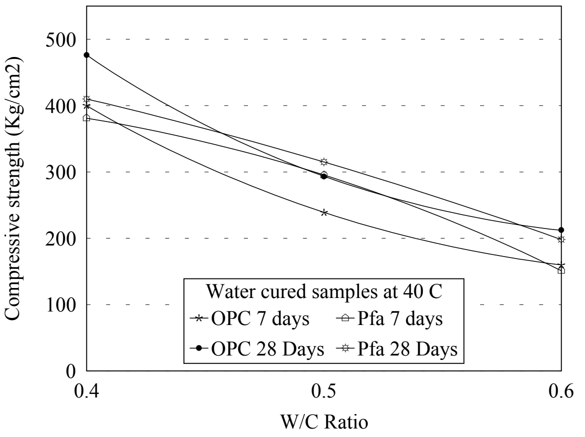

Figure 3 Compressive strength for water cured mortars at 40 °C

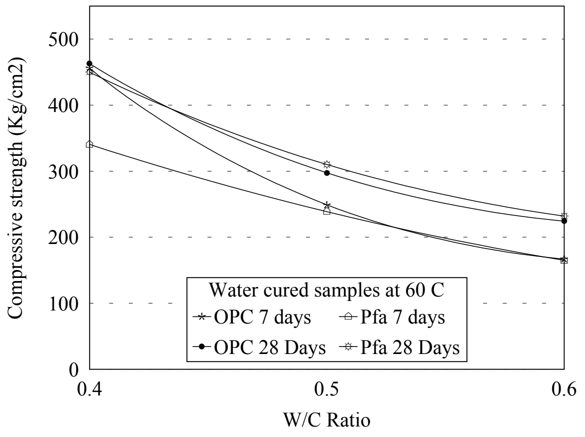

Figure 4 Compressive strength for water cured mortars at 60 °C

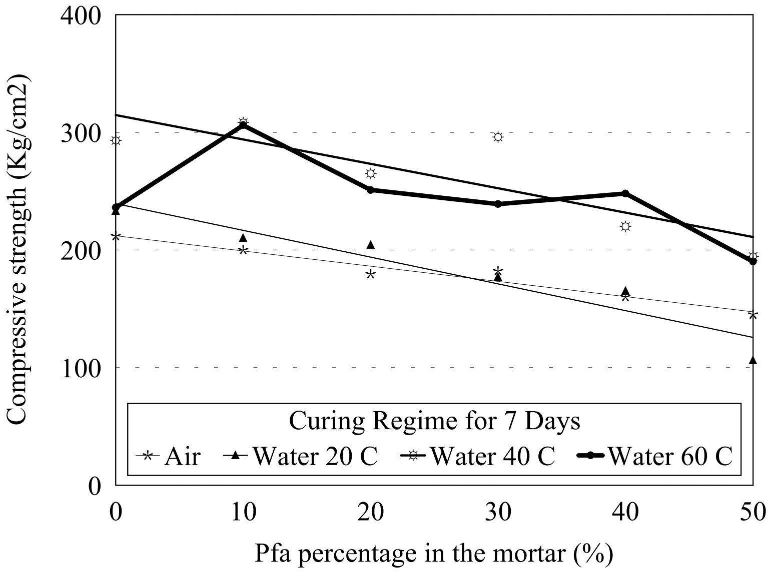

Figure 5 Compressive strength for samples cured for 7 days

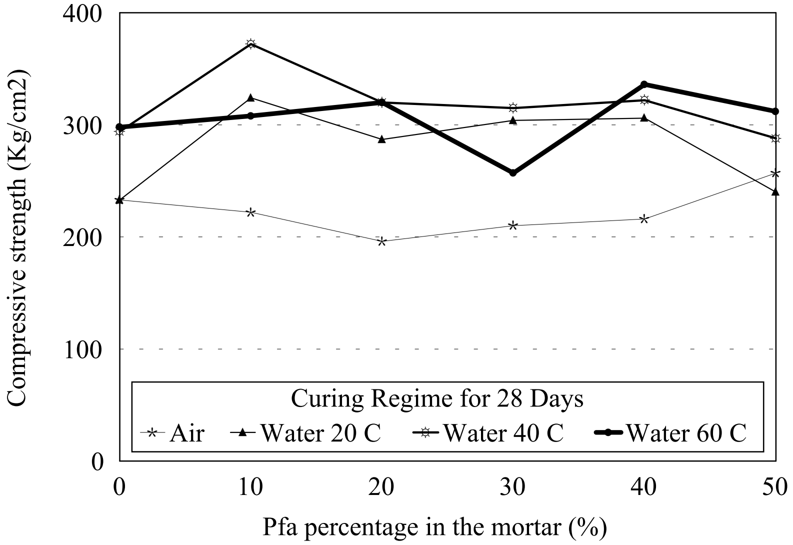

Figure 6 Compressive strength for samples cured for 28 days

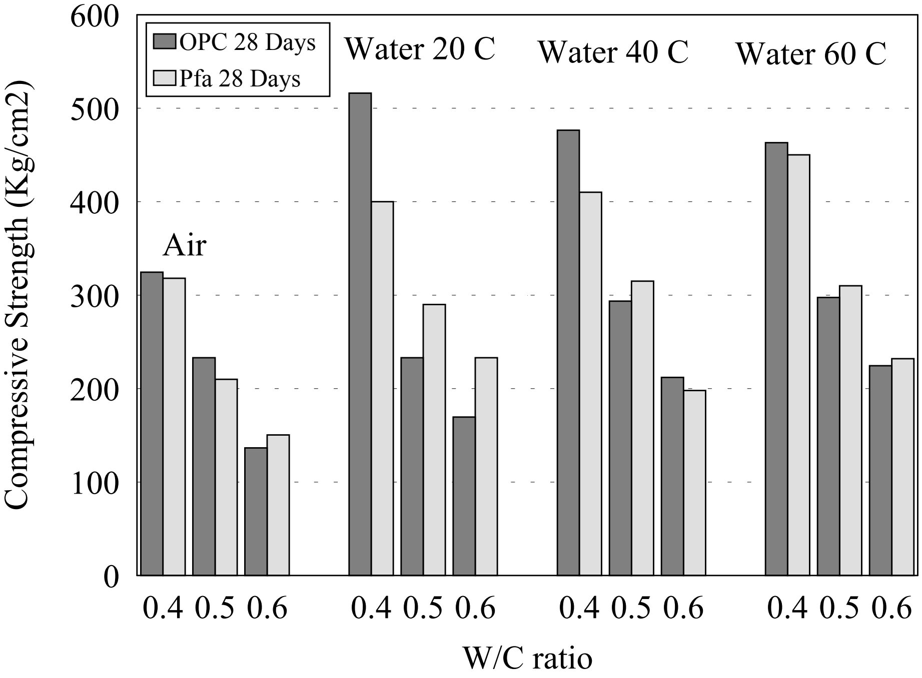

Figure 7 Effect of curing regime on compressive strength