Strength and durability effect of modified zeolite additive on concrete properties

B.D Ikotun^{*}, S. Ekolu

School of Civil and Environmental Engineering, University of the Witwatersrand,

Private Bag X3, WITS 2050, Johannesburg, South Africa

Abstract

This paper presents an investigation into the effect of a modified zeolite additive (PWC) on strength and durability behaviour of concrete. The additive, a blend of selected alkaloids and zeolite, is commercially available and effectively used in soil stabilization for road construction. However, its influence on behaviour and performance for use in concrete has not been explored. In this research, concrete samples were prepared by incorporating PWC and/or 30% fly ash in the mixes. PWC was used in proportions of 0.6 %, 0.8%, and 1.0 % of the weight of cement. Concretes or mortars of 0.5 water/cementitious ratio were subjected to split tensile strength, compressive strength, oxygen permeability, sorptivity, and porosity tests. Hydration behaviour of the cementitious systems and the pozzolanic effect of PWC additive were studied using differential thermal analysis and thermogravimetric analysis. Results show that PWC is effective in improving concrete strength when used at optimum proportions found to be between 0.6 % and 1.0 %. Sorptivity property of concrete was improved with use of PWC regardless of its proportion in the mix, while permeability of concrete improved only in the presence of 30 % fly ash. PWC was found to be most effective when used in the presence of fly ash.

*Corresponding author, email address: ayekot200@yahoo.com

Keywords: Modified zeolite additive; Fly ash; Compressive strength; Split tensile strength; Workability; Permeability; Sorptivity

1.0 Introduction

There is ongoing interest and effort by many researchers towards the quality and performance of concrete by improving its durability and strength without increasing the cost of construction. This interest has led to a series of studies investigating the use of extenders or mineral admixtures also known as pozzolans, to improve concrete properties.

The PWC additive used in this study to investigate the strength and durability improvement of concrete is manufactured and processed in the Netherlands according to ISO 9001 and 14002. The product is a blend of selected alkaloids and zeolite. It is claimed that the product modifies the chemistry of cement hydration process enhancing the crystallization process by forming long needle-like crystalline structures and calcium hydroxide besides dicalcium and tricalcium silicate [1]. Improvement of concrete strength and durability by incorporation of pozzolans in mixes has been of interest to researchers in the field of construction materials for ages. In most cases, use of pozzolans in concrete results in reduction of early strength and increment in late strength when used within optimal proportions. The optimal proportions always require high replacement contents of pozzolans [2-4]. Zeolite, a type of pozzolan also exhibits this behaviour [5-7].

Natural zeolite contains large quantities of reactive SiO_2 and Al_2O_3 [5]. These elements combine with $Ca(OH)_2$, a by-product of cement hydration to form further calcium silicate. This reaction called pozzolanic reaction is responsible for increasing

the long term strength and refining the pore structure of blended concrete. Typical oxide compositions of zeolites are shown in Table 1 [8].

Use of zeolite in improving concrete properties can be enhanced if modified, such that the cation exchange of the modified product is greatly increased [7]. The modified zeolite can have the advantage of enhancing concrete strength better and also reduce the dosage of zeolite additive that will be needed. PWC is a modified zeolite, which is commercially available and has been effectively used in soil stabilization for road construction. Its influence on mortar properties has been reported in another paper by the authors [9]. The objective of this study was to determine the optimum proportion of the product needed to improve the strength and durability properties of concrete. Also, to study its effect on concrete properties in the presence of fly ash (FA).

2.0 Experimental

2.1 Materials and mixes

The binding materials used for this study consisted of commercially available modified zeolite, ordinary Portland cement CEM I 42.5N (equivalent of ASTM type 1), and fly ash. The different aggregates used consisted of 19 mm coarse aggregate (stone), locally produced silica sand, and granite crusher sand. Coarse aggregate of size 19 mm were used in concrete mixes. Reagents used throughout the experimental work were ordinary portable water and water reducing admixture.

PWC was used as an additive to cement in proportions of 0.6%, 0.8%, 1.0%, and 2.5 % by weight of cement with or without 30 % FA. Constant water: binder ratio (w/b) of 0.5 was used throughout the study. Details of the mixes are shown in Table 2 and Table 3 for mortars and concretes respectively. As shown in Table 4, Fly ash

additive was observed to be finer than CEM 1 42.5N. The loose and relative densities of CEM 1 42.5N were higher than FA and PowerCem additives. This observed trend could indicate PWC additive is lighter but more bulky than CEM I 42.5N and FA.

Local South African (SA) silica sand was prepared for compressive strength testing of mortars. The grading analysis of SA silica sand (SASS), prepared for compressive strength test and commercially available European silica standard sand (ESSS) are shown in Figure 1. 50 mm cubes mortar were prepared using both types of standard sands and tested to compare their results.

The compressive strength results for the two types of standard sand showed relatively the same trend (Figure 2) when cured for 3, 7, 28, and 90 days. It was observed that the SA silica sand grading analysis fits in the range specified in SABS EN 196-1 [10]. Comparison of the grading analysis and compressive strengths of SASS and ESSS gave similar results. It was inferred from the results, based on the trends observed that SASS can be locally used in laboratory testing as standard sand for cement testing in lieu of commercially imported and expensive ESSS. The use of SASS is an advantage over ESSS as the former is prepared locally and is cost effective.

2.2 Sample preparation

Hydration study was done on pulverized hydrated mortar samples. These samples were cured for 115 days and then kept in the oven maintained at 50 $^{\circ}$ C for 24 hours. The samples were pulverized after being removed from oven and sieved through 90 μ m sieve to obtain only pastes powder for use in thermal studies. The thermal decomposition behaviour of powder samples was studied by tracing mass and

heat changes using a Setaram TG92 thermogravimetric analyzer coupled to a Setaram differential analyzer.

Accelerated compressive strength test was carried out on mortar samples as a confirmatory test, using 50 mm cube moulds. Samples were mixed in a HOBART mortar mixer for 3 minutes and cast in 50 mm cube moulds. The moulds were placed in the oven at 50 °C immediately after casting for 8 hours, after which the hardened cubes were removed from their moulds and placed in an accelerated water curing tank. The temperature of curing water was maintained at 50 \pm 2 °C with a built in thermostat and a small circulation pump. Samples were stored in the curing tank until testing period.

Concrete samples were prepared such that the materials (crusher sand, CEM I with or without fly ash, PWC, and coarse aggregate) were batched on a laboratory balance to an accuracy of 100 g. These materials were mixed firstly in their dry state in a 50 L pan mixer in the order of crusher sand, CEM I with or without fly ash, and coarse aggregate. Water with added PWC and water reducing agent, were then introduced into the mix over a period of 1 minute. Mixing was then continued for another 1 minute. Casting of concrete samples was done by using 100 mm cube moulds. The samples were water-cured for the required testing ages. The temperature of curing water was maintained at $23 \pm 2^{\circ}$ C with a built in thermostat and a small circulation pump.

2.3 Test procedures

2.3.1 Concrete spilt tensile strength test

Split tensile test was carried out on concretes based on ASTM C 496 [11] using 100 mm cubes. A centre line was drawn on the opposite sides of the cube samples. During testing, the cube sample was placed in position in the Tinus Olsen testing machine. Then steel bars of 16 mm diameter, sealed on to steel plates were placed on the centre line drawn on the opposite sides of the cube samples. Load was applied on the two opposite centre line, such that the splitting along these lines was caused by the principal tensile stress in the plane joining the loaded lines. The split tensile strengths were determined at curing ages of 28 and 180 days.

2.3.2 Concrete compressive strength test

The compressive strength test was carried out on concrete samples based on SABS Method 863 [12]. At testing age, samples were removed from the curing tank and weighed. The samples were centrally placed in a compression testing machine and load was applied at a rate of 150 KN/minute. Cubes were tested using Amsler type 103 compression testing machine, which has a load capacity of 2000 KN. Compressive strengths were determined at curing ages of 3, 28, 90 and 180 days.

2.3.3 Oxygen permeability

Permeability of mortars was measured using a falling head gas permeameter [13]. The method is based on Darcy coefficient of permeability determined by monitoring a falling pressure head. Test samples consisted of discs of diameter 68 ± 2 mm and thickness 25 ± 2 mm. At testing age, these were core drilled from 100 mm cubes (parallel to the casting direction) then oven dried at 50 °C for 7 days prior to testing.

The disc samples were then removed from the oven and allowed to cool for 2 hours in a room maintained at 23 °C. After recording of dimensions, disc samples were inserted in a compressible collar with PVC sheath and the assembly fitted in the permeameter chamber, then tightened.

The oxygen pressure in the permeameter chamber was increased to 100 KPa and then the inlet valve was closed. Time and pressure were then recorded. Pressure decay was recorded at intervals of approximately 5 KPa and test stopped when pressure reaches approximately 60 KPa or 6 hours from start of test. After the permeability test, the disc samples were removed from the collar and later used for sorptivity test.

The coefficient of permeability (m/s) is calculated from:

$$\mathbf{K} = \frac{WVg}{RA} \left(\frac{d}{\theta t}\right) \ln\left(\frac{P_o}{P}\right)$$

where: K = coefficient of permeability in m/s

W = molecular mass of oxygen, 32 g/mol

V = volume of oxygen under pressure in permeameter (m^3)

g = acceleration due to gravity, 9.81 m/s²

R = universal gas constant, 8.313 Nm/Kmol

A = superficial cross- sectional area of sample (m^3)

d = average sample thickness (m)

 θ = absolute temperature (K)

t = time (s) for pressure to decrease from P_o to P

 P_o = pressure at the beginning of test (KPa)

P = pressure at the end of test

2.3.4 Sorptivity and Porosity

In this test, the curved surface of disc samples was sealed with tape up to 5 mm above test face to allow one directional capillary flow of water. The test face of disc samples was placed in lime-saturated solution contained in a tray such that the final level of solution was slightly above the edge of sample. These samples were weighed at regular intervals of 3, 5, 7, 9, 12, 16, 20, and 25, followed by vacuum saturation of samples at -75 KPa suction. Vacuum was applied for 3 hours on discs placed in a dessicator followed by five hours of vacuum suction with the disc samples submerged in saturated lime-water. After a further 18 hours of soaking, the samples were weighed. Porosity was calculated using from:

$$n = \frac{M_{sv} - M_{so}}{A.d.\rho_w}$$

where: M_{sv} = vacuum saturated mass of the samples to the nearest 0.01 g M_{so} = initial mass of the specimen to the nearest 0.01 g A = cross- sectional area of the samples to the nearest 0.02 m² d = average samples thickness to the nearest 0.02 mm P_w = density of water

The mass of water absorbed at each weighing period was calculated using:

$$M_{wt} = M_{st} - M_{so}$$

where: M_{st} = mass to the nearest 0.01 g of the sample at time t

Sorptivity was calculated from the slope of graph of water absorbed (M_{wt}) versus the square root of time (in hr).

$$S = \frac{F.d}{M_{sv} - M_{so}}$$

where: F = the slope of the best fit line obtained by plotting M_{wt} against $t^{1/2}$

S = sorptivity

3.0 Results and discussion

3.1 Effect of test age and PWC additive on concrete split tensile strength

Increase in split tensile strength between 28 and 180 days was observed for all samples except control and 1.0 % PWC samples as shown in Figure 3. Control samples have higher strength at 28 days than other samples with PWC and FA additive. However, strength values of samples containing PWC and FA were higher at 180 days than the control samples, with exception of premix samples containing 1 % PWC + 59 % FA + 40 % CEM 1 42.5N which gave fairly low strengths at both 28 and 180 days. Samples with PWC as the only additive, showed higher tensile strength at 28 days than samples containing FA additive. At 180 days, samples containing FA additive showed higher tensile strength than samples containing only PWC additive. The highest tensile strength results were observed with 1.0 % PWC +30 % FA samples at 180 days.

The tensile strength gains at 180 days were 54.2 %, 80.8 % and 95.2 % for 30 % FA, 0.6 % PWC + 30 % FA, and 1.0 % PWC + 30 % FA samples respectively, as represented in Figure 4. It can be inferred from the results that PWC additive enhanced the strength behaviour of FA additive. It is also shown in Figure 4 that PWC

additive reduced the split tensile strength of concrete by a maximum of 18.5 % at 28 days with 0.6 % PWC proportion, and increased it to maximum percentage of 18.3 % at 180 days with 0.6 % PWC.

In the case of 30%FA incorporation, the split tensile strength of concrete was reduced by maximum of 23 % at 28 days, and increased by up to 54.2 % at 180 days. When PWC and FA additives were both used in a mix, the combination reduced the split tensile strength of concrete by a maximum of 19.1 % at 28 days for 0.6 % PWC + 30 % FA, and increased it by a maximum percentage of 95.2 % at 180 days for 1.0 % PWC + 30 % FA.

The relative reduction in early strength followed by increase in tensile strength due to use of PWC and FA, when compared to control samples is characteristic of most pozzolanic material [14]. PWC additive exhibited excellent tensile strength results when used with FA additive.

3.2 Effect of test age and PWC additive on concrete compressive strength

Increase in compressive strength was observed in all samples as curing period increases as seen in Figure 5. Premix samples, made of 1 % PWC + 59 % FA + 40 % OPC gave compressive strength results that were unsatisfactory throughout the testing period, as was the case for split tensile strength of the samples. Reduction in early strength was observed for samples containing FA additive while early strengths higher than those of control were observed with samples having PWC as the only additive. Higher strengths than for control samples were observed with samples containing FA additive when cured for at least 90 days.

It is clear in Figure 5 that the PWC additive improved both early and late compressive strength of concrete. FA additive has the effect of reducing the early

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compressive strength and increasing the late strength compared to control. Strength improvement was highly enhanced when both PWC and FA additives were used. This confirms the fact that PWC additive enhanced the strength behaviour of FA additive, as observed for split tensile strength. Highest strength was observed with 1.0 % PWC +30 % FA samples, followed by 0.6 % PWC +30 % FA samples from 90 days.

In Figure 6, compressive strength of concrete increased by up to 15.8 % when only PWC additive was used at 0.6 % PWC proportion. Relative to control, 30 % FA additive reduced the compressive strength of concrete by maximum percentage of 39.7 % at 3 days and increased it by maximum percentage of 17.9 % at 180 days.

It can also be observed from Figure 6 that when the PWC and FA additives were used together, the combination of 0.6 % PWC + 30 % FA proportion reduced the compressive strength of concrete by maximum percentage of 38.4 % at 3 days, but increased it to maximum percentage of 22.7 % at 180 days for 1.0 % PWC + 30 % FA. Similar trends of changes in strength due to effects of PWC and FA (compared to control samples) were observed for both split tensile and compressive strength. Samples of 1.0 % PWC + 30 % FA had the highest strength value for both split tensile and compressive strength at 180 days.

The observed results in this study agree with observations made by Poon *et al.* [5] where the pozzolanic activity of natural zeolite was reported to be higher than that of fly ash. It was further explained that mortars and concretes blended with natural zeolite tended to give higher early strength compared to fly ash blended systems as a result of the higher pozzolanic activity of zeolites. The PWC additive is a modified zeolite and an improved pozzolanic activity may be presumed in the presence of PWC additive. This may explain the increased early strength observed in PWC concrete samples compared to the lower early strengths for FA additive samples. With

reference to split tensile and compressive strength results of concrete, 0.6 % PWC and 1.0 % PWC samples showed satisfactory results. The performance of PWC additive can be highly enhanced when used in conjunction with FA additive. Accelerated compressive strength test was performed on mortar samples to confirm this observation.

3.3 Effect of test age and PWC additive on accelerated compressive strength of mortars

In Figure 7, the compressive strength of samples containing PWC and FA was low at 3 days curing period when compared to control samples. Improvement in strength was observed for samples containing both PWC additive and FA from 14 days onwards.

The early strength improvement of PWC and FA additives combined samples may be as a result of higher curing temperature of 50 $^{\circ}$ C used. With only PWC additive, compressive strength was increased by a maximum percentage of 12.6 % at 28 days when used at 0.6 % PWC proportion. For PWC and FA additives combined samples, there was maximum of 25.6 % increase in compressive strength at 28 days with 1.0 % PWC + 30 % FA sample as shown in Figure 8. This observation confirms that FA additive enhanced the strength behaviour of PWC additive or vice versa.

The strength results obtained for mortars, reported earlier by the authors [9] as well as results for concretes given in this study, indicate that PWC additive has the ability to improve mortar and concrete strength when used at proportion as low as 0.6 %. These results were compared with the findings of Naiqian *et al.* [15], who also investigated a modified zeolite. Their study reported that the modified zeolite investigated improved concrete strength when used to replace 5 to 10 % of the

ordinary Portland cement at water-cement ratio of about 0.35. The low dosages of PWC additive needed to improve mortar and concrete strengths can be a significant advantage over other types of modified zeolites and pozzolans generally that have to be used at higher dosages.

3.4 Effect of PWC additive on concrete oxygen permeability, sorptivity, and porosity

As shown in Figure 9, reduction in permeability was observed for all samples due to age of curing from 28 to 180 days. Premix samples showed a highly remarkable decrease in permeability over this period. The PWC additive, when used as the only additive, did not seem to reduce permeability of concrete to an extent lower than the permeability of control samples.

When used in conjunction with FA additive, PWC additive performs excellently by reducing the permeability of concrete samples by up to 37.0 % at 180 days for 0.6 % PWC + 30 % FA proportion. FA additive reduced permeability of concrete when used as the only additive by up to 39.2 % at 28 days and up to 51.9 % at 180 days for the 30 % FA proportion as seen in Figure 10.

The sorptivity of control concrete was higher than that of all containing PWC and FA samples at both 28 and 180 days. This observation was the same as results for mortar samples reported earlier by the authors [9]. Figure 11 shows that FA improved sorptivity property of concrete more than PWC additive.

Use of 30 % FA reduced sorptivity of concrete by up to 40.8 % at 180 days, while 0.6 % PWC additive reduced sorptivity of concrete by a maximum percentage of 21.1 % at 180 days. When both PWC and FA additives were used, sorptivity of

concrete was further reduced, attaining up to 47.4 % reduction at 180 days for 1.0 % PWC + 30 % FA proportion as represented in Figure 12.

As seen in Figure 13, porosity of control samples was highest against all other samples, at both 28 and 180 day ages. Samples prepared with both PWC and FA additives in their mixes exhibited lower porosity than samples with only PWC additive. FA was seen to help in reducing the porosities of PWC samples.

Figure 14 shows that, when only PWC additive was used, porosity of control samples was reduced by a maximum percentage of 10.2 % at 28 days for 1.0 % PWC proportion. In the case of combined use of PWC and FA additives, porosity was reduced by a maximum percentage of 19.7 % at 180 days for 0.6 % + 30 % FA samples.

The observed reduction in permeability with age for all samples is consistent with the understanding that this property decreases with an increase in the degree of hydration or curing time [16, 17]. In this study, use of the pozzolans PWC and FA must have reduced continuity in pore distribution of the samples making the concretes less permeable.

3.5 Hydration study using thermal analysis

Different temperature peaks were observed in the differential thermal analysis curves of all samples as seen in Figure 15. These different temperature peaks represent the dehydration temperature of CSH gel, de-hydroxylation temperature of $Ca(OH)_2$ and the decomposition temperature of $CaCO_3$ (de-carbonation). DTA curves for all the samples showed a similar trend (Figure 3) indicating that the hydration characteristics and products are almost identical. Small differences were in the peak areas at different temperatures.

The summary of the dehydration temperature peak of CSH, the dehydroxylation temperature peak of CH, the de-carbonation temperature peak of CaCO₃, the mass loss between 200 °C and 1000 °C, and the glass transition zone values of all samples are shown in Table 5 The three major temperature peaks occurred at approximately the same temperature for all samples. The observed peak areas given in Table 6 for dehydration of CSH at approximately 200 °C indicate that more CSH was generally formed with increase in PWC additive compared to control.

The decrease in peak areas (Table 6) due to decomposition of $Ca(OH)_2$ at approximately 500 °C in samples with PWC additive compared to that of control sample may indicate the occurrence of a pozzolanic reaction. Consumption of CH in pozzolanic reaction gives secondary CSH, hence, corresponding increase of CSH observed at 200 °C for PWC additive samples. Caputo *et al.* [18], explains that zeolite reactivity is related to its large external specific surface, which favors its dissolution into the saturated lime solution and the subsequent precipitation of CSH (hydrated calcium silicate) and CAH (hydrated calcium aluminate) phases.

The reduced intensity of CaCO₃ peak in all samples, especially samples with PWC additive indicated the dissolution of CaCO₃ during the formation of hydration products. There was reduction in the peak areas due to de-carbonation of CaCO₃ at approximately 800 $^{\circ}$ C in samples with PWC additive compared to control sample. This is consistent with relatively less amount of CH present in the system containing PWC additive. A notable decrease in the amount of Ca(OH)₂ along with corresponding increase in CSH was observed with samples incorporating PWC additive when compared to control sample. These observations are more pronounced for 0.6 % and 1.0 % PWC samples.

Conclusions

Based on observations and trends determined from this investigation, the following conclusions were made:

- 1. The use of PWC in concrete generally increases both early and late compressive strengths when compared to strength results of the control.
- 2. At 28 days curing period, PWC concrete samples exhibited a decrease in split tensile strength compared to control, but at 180 days their split tensile strength increased to values higher than that of control samples.
- 3. It was found that the effect of PWC additive on concrete strength improves significantly when it is used in conjunction with FA. When 30 % FA was used with 0.6 % PWC additive in concrete, there was 21.3 % increase in compressive strength at 180 days when compared to the control. With 0.6 % PWC additive alone, a small increase of 7.5 % in compressive strength was observed at 180 days. Accelerated compressive strength test done at 28 days on mortar samples also showed 22.7 % increase in strength for 0.6 % PWC additive.
- 4. Oxygen permeability of concrete improved only when PWC additive was used in conjunction with FA. PWC additive improves concrete porosity and sorptivity but further effective reductions in the properties are observed when PWC is used together with FA. Results show that PWC additive is most effective when used in the presence of FA.
- 5. Thermal analysis results indicate that PWC additive is a pozzolanic material and aids the consumption of $Ca(OH)_2$. Its usage in proportions beyond 0.6 % of cement did not seem to enhance the pozzolanic effect.

Acknowledgement

The authors are grateful to the PowerCem (Africa) Technologies, for providing a research grant to carry out this work.

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