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Indicative tests on the effect of fly ash-β cyclodextrin composite on mortar and concrete permeability, sorptivity and porosity

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Abstract. The focus of this research is to modify fly ash (FA) structure using β -cyclodextrin (β-CD) to increase its pozzolanic function in concrete. A previous study by the authors has shown that a composite is formed due to structural changes of fly ash by its interaction with cyclodextrin [1]. The objective of this paper was to assess the effect of fly ash-βcyclodextrin composite (FA-β-CD) on the transport properties (durability performance) of mortar and concrete, with a view to optimise its incorporation in concrete. The durability performance of mortar and concrete were assessed from the measurement of oxygen gas permeability, sorptivity and porosity using the South Africa durability index approach. Six mixtures were tested, comprising a control mixture (100% Portland Cement, PC), and five mixes containing the FA-β CD composite. All the composite mixes included 30 % FA by mass. The β-CD was mixed with the FA, in separate mixtures, in proportions of 0.1 %, 0.2 % and 0.5 %. Two sample preparation procedures were followed for FA-β-CD composites mixtures; firstly, physical mixtures of a pre-weighed amount of β-CD and FA were adopted for the dry mixtures and secondly, 0.0103M, 0.0206M and 0.0516Mβ-CD solutions were added to the concrete at the mixing stage for solution mixtures. The results indicated that FA-β-CD composite improved the flowability and durability performances when β-cyclodextrin (β-CD) was used at a lower percentage (0.1% and 0.2%).

Keywords. βcyclodextrin, concrete, FA-β-CD composite, fly ash, mortar, permeability, porosity and sorptivity

Introduction

The global interest in optimising and improving concrete performances with reduction in environmental pollution caused by cement production has inspired many researchers into exploring the inclusion of different by-products from industrial processes into concrete technology [2-3]. The industrial by-products that have been proven to be pozzolanic and improve concrete performance include ground granulated blast furnace slag (GGBS), fly ash (FA) and condensed silica fume (CSF) [4-9].

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FA, a by-product of coal combustion, which is in abundance in South Africa, has been of great use in blended cement [8, 10-15]. Fly ash has the characteristics of improving concrete workability, reducing heat of hydration, and improving late strength and durability [8, 16-17]. Apart from the advantages of using FA in blended cement, reduction in disposal costs and minimization of its impact on the environment are part of the main reasons for further research towards increasing the use of FA [14, 18-23]. The research focus that is of interest to the authors and some researchers [20, 22, 24] is the modification of FA structure to aid its function in concrete technology. A previous study by the authors [1] has shown that a composite is formed, due to structural changes, by the interaction of FA with β-cyclodextrin (β-CD) (oligosaccharides formed by the enzymatic modification of starch). This composite provides promising application in concrete technology. The incorporation of the composite formed by fly ash- β-cyclodextrin (FA-β-CD) interaction in concrete production is a very new area in research. Hence, limited information is available and indicative tests are required to give guidance for on-going research.

The key durability assessment of concrete is based on the factors that affect concrete transport mechanisms [3, 25-27]. This paper presents the indicative tests results of the effect of FA-β-CD composite on the durability behaviour of mortar and concrete. The durability performance of mortar and concrete were assessed from the measurement of oxygen gas permeability, sorptivity and porosity using the South Africa durability index approach [28]. Two major parameters that are related to transportation mechanisms, which aid these tests, are permeation and absorption.

1. Materials

The materials used in this study were class F FA, β-CD, Portland cement (CEMI52.5N), silica sand, crusher sand and coarse aggregate. FA was obtained from one of the South African ESKOM power stations through Ash Resources, South Africa. β-CD from Wacker chemie were obtained from Industrial Urethanes (Pty) Ltd, South Africa and the CEMI52.5N cement was obtained from Pretoria Portland Cement (PPC), South Africa. Granite crusher sand and coarse aggregate with a nominal size of 22 mm were obtained from Afrisam, South Africa. Silica sand sized within the ranges of 0.8-1.8 mm (coarse), 0.4–0.85 mm (medium) and 600 μm (fine), produced by Rolfes silica, South Africa, was used in mortar mixes. Standard silica sand was prepared with the available size ranges in accordance with the SANS 50196-1 [29] and used for mortar mixes. The characteristics of β-CD, as supplied by the producer and the chemical analysis of the fly ash used, are presented in Tables 1 and 2, respectively.

Two samples preparation procedures were followed for FA-β-CD composites mixtures. Firstly, physical mixtures of pre-weighed amount of β-CD and FA were adopted for the dry mixtures and secondly, 0.0103M, 0.0206M and 0.0516Mβ-CD solutions were added to the mortar and concrete at the mixing stage in the case of the solution mixtures. A proportion of 30% FA was maintained in the mixtures proportions. Furthermore, 0.1% and 0.2 % β-CD, in both dry and solution mixtures were used together with FA.

Table 1. General characterisation of β-CD used

Property	βCD
Empirical formula	$C_{42}H_{70}O_{35}$
Bulk density	400-700 kg/m^3
Solubility in water at 25 ºC	18.5 g/l
Content (on dry basis)	Min. 95 %

Table 2. Chemical composition of the FA

Content in oxide form.	% mass
SiO_2	50.26
Al_2O_3	31.59
Fe_2O_3	3.08
MgO	2.04
CaO	6.78
Na_2O	0.56
K_2O	0.81
TiO_2	1.64
SO_3	0.55
LOI	1.42
SiO_2/Al_2O_3	1.59

2. Experimental procedures

2.1. Mixes and curing of mortar

A total of six mortar mixtures were designed, in accordance with the mixture proportions shown in Table 3. The mortar samples were prepared and mixed in accordance with SANS 50196-1 [29] without any alteration in the case of dry mixes.

In the case of the solution mixes, the 0.0103M and 0.0206M β-CD solutions for F/0.1CD/S and F/0.2CD/S samples, respectively, were added after the standard silica sand had been introduced to the mix. 100 mm cubic moulds were prepared and demoulded in accordance with SANS 50196-1 [29]. The samples with β-CD were kept covered with polythene sheets for three days before demoulding because of the nature of the material. After demoulding, the samples were placed in a water bath maintained at 23°C ± 2°C for curing until the testing ages.

The mix proportion used for the mortar production is as shown in Table 3. To maintain relative consistency with the control sample, the samples were prepared with varied w/b ratios. This approach was adopted to assess the behaviour of β-CD on fresh mortar and to establish a relationship that will form a basis for proper mixture design for on-going research. The flow test was conducted on mortars according to ASTM C 1437 [30] immediately after mixing.

Table 3. Mortar mixture proportion per 1m^3 of mortar

Samples	Cement (kg)	Silica sand (kg)	Fly ash (kg)	CD (kg)	CD solution (litres)	Water (kg)	w/b	Flow
Control (100 % Cement)	586.00	1758.00	0.00	0.00	0.00	293.00	0.5	101
FA/C (30%FA + Cement)	410.20	1758.00	175.80	0.00	0.00	293.00	0.5	106
F/0.1 CD/D (29.9% FA + 0.1% CD dry + Cement)	410.20	1758.00	175.21	0.59	0.00	275.42	0.47	115
F/0.2 CD/D (29.8% FA + 0.2% CD dry + Cement)	410.20	1758.00	174.63	1.17	0.00	246.12	0.42	120
F/0.1 CD/S (29.9% FA + 0.1% CD in solution (0.0103M) + Cement)	410.20	1758.00	174.63	0.00	50.00	226.01	0.47	120
F/0.2 CD/S (29.8% FA + 0.2% CD in solution (0.0206M) + Cement)	410.20	1758.00	172.87	0.00	50.00	197.29	0.42	125

2.2. Mixes and curing of concrete

During mixing, the rotary mixer with a 100 litre capacity was charged with coarse aggregate, fine aggregate, cement, FA and β-CD, respectively. These materials were mixed in their dry state for one minute. Thereafter, water was introduced into the mix over the period of one minute and mixing continued for a further minute. In the case of the solution mixtures, the β-CD solution was added, over a 30 second period, after the dry material was mixed for one minute. Thereafter, the remaining water was added after a period of 30 seconds and mixing continued for a further minute.

Immediately after each mixture was produced, the slump test was performed (according to SANS 5862-1:2006 [31]) and 100 mm cubic moulds were cast and compacted (using a vibrating table). The curing procedures explained in Section 2.1 were followed. Table 4 presents the mixture proportions used for the concrete production. The samples were prepared with varied w/b ratio for the same reason as explained in Section 2.1.

2.3. Permeability, sorptivity and porosity tests

The oxygen permeability and sorptivity tests were performed at the AFRISAM laboratory in Roodepoort, South Africa. The South African durability index approach which was developed by Ballim and Alexander [32-34] was adopted in this study. The approach was developed to cater for the practical durability tests that could be site-applicable. Measurements of oxygen permeability and sorptivity/porosity were used in this study to assess the durability performance of the samples. The oxygen permeability index (OPI) test gives an indication of the degree of pore connectivity in a concrete matrix, while sorptivity measures the rate of movement of a water front through the concrete under capillary suction. These tests were conducted on discs samples with a diameter of 68mm and thickness of 30 ± 2 mm which were core drilled from the cover zone of the 100 mm cubes samples, after being cured for 14 and 28 days. The disc samples were preconditioned in an oven at 50^{0}C for 7 days before testing. The detailed procedures, which have been previously described and documented [33, 35-39] were followed.

Table 4. Mixture proportions for 1m^3 of concrete

Samples	**Cement**	**Crusher sand (kg)**	**Coarse aggregate (kg)**	**FA (kg)**	**CD (kg)**	**CD solution (litres)**	**Water (kg)**	**w/b**	**Slump (mm)**
Control	410.00	788	980	0.00	0.00	0.00	205	0.5	50
FA/C	287.00	788	980	123.00	0.00	0.00	205	0.5	90
F/0.1 CD/D	287.00	788	980	122.59	0.41	0.00	192.7	0.47	95
F/0.2 CD/D	287.00	788	980	122.18	0.82	0.00	172.2	0.42	120
F/0.1 CD/S	287.00	788	980	122.59	0.00	35.00	192.29	0.47	150
F/0.2 CD/S	287.00	788	980	122.18	0.00	35.00	171.38	0.42	150

3. Results and discussion

3.1. Flow of mortar and workability of concrete samples

The flow of mortar and workability of Concrete Samples as shown in Tables 3 and 4 respectively showed that β-CD increased both flow and workability with reduced water content. The characteristic of β-CD of enhancing wettability in water [40-41] contributes to this behaviour, with a higher dissolution being experienced in the solution mixed samples. The reduction of water content in the mix and increased flow/workability is an advantage to FA-β-CD composite in improving mortar and concrete quality.

3.2. Effect of βCyclodextrin on the permeability of fa/c mortar

The oxygen permeability index (OPI) test is sensitive to the amount and continuity of larger pores and voids where most of the flow will occur [28]. The coefficient of permeability (k) and oxygen permeability index (OPI) values for mortar samples are presented in Figures 1 and 2, respectively. The results for concrete samples were not reported because of some invalid values, which are anomalous. It is evident from Figure 1 that all the samples showed decrease in permeability at increased curing age. The highest permeability was exhibited by the FA/C sample at 14 days curing age. However, a decrease in permeability was observed in the case of the FA/C mortar sample relative to the control sample at 28 days curing age. It is evident in Figure 2 that, in general, the higher the OPI, the less permeable the concrete. This is in agreement with the findings of Ballim and Alexander [28], who stated that the OPI provides an indication of the degree of pore connectivity in the matrix and how permeable the samples are.

The samples containing the FA-β-CD composite exhibited lower permeability than the control sample and FA/C sample, for both curing ages. The β-CD solution was more effective in decreasing permeability, compared to the dry β-CD. When considering both dry and solution β-CD mixes, no general correlation was established between the quantity of β-CD and its effectiveness in reducing permeability. The mixtures containing the β-CD solutions resulted in the greatest relative reductions in permeability, being 64.8 % and 62.8 % in the case of the F/0.2 CD/S and F/0.1 CD/S, respectively.

This showed that despite the dissolution observed with the solution mixed samples, a positive effect on sample permeability was observed. The results confirmed the previous observation by the authors [1] that cylcodextrins in solution will try to occupy the pores. This will thereby limit continuous pores and in turn reduce permeability. Generally, the results indicated that FA-β-CD composite boosted the pozzolanic reaction with evidence of permeability reduction at both 14 and 28 days after curing period, compared to control sample.

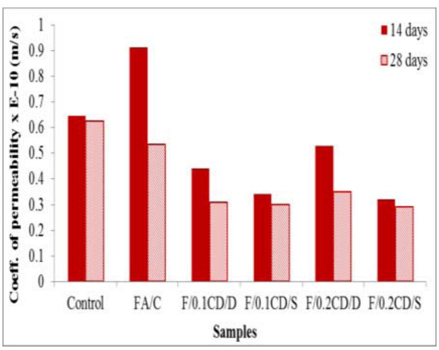

Figure 1. Permeability of mortar samples

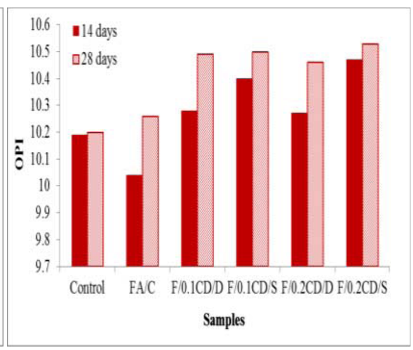

Figure 2. Oxygen permeability index (OPI) values of mortar samples

3.3. Effect of βCyclodextrin on the sorptivity of FA/C mortar and concrete

The water sorptivity test measures the rate of movement of a water front through the exposed face of the mortar or concrete samples, under capillary suction. The lower the water sorptivity index, the better the potential durability of the concrete [28]. Figures 3 and 4 present the sorptivity results of mortar and concrete samples, respectively. A decrease in sorptivity was observed with an increase in curing age for all samples for both mortar and concrete. The reason for this is that as the curing age increased; the mortar or concrete became denser, starting from the exposed face, which resulted in decrease in sorptivity. A higher sorptivity was observed for FA/C mortar compared to the control samples at both curing ages due to the slow pozzolanic reaction, which might have not been completed to result in a denser mortar at these ages. However, a lower sorptivity was observed for FA/C concrete relative to the control sample at a curing age of 14 days. This is attributed to the presence of coarse aggregate, which resist to a smaller extend the movement of water under capillary suction. However, the slower pozzolanic reaction resulted in higher soptivity of FA/C concrete relative to the control sample at a curing age of 28 days.

The mortar and concrete samples with FA-β-CD composite exhibited a lower sorptivity than the control sample and FA/C sample at both curing ages. The mortar samples showed lower sorptivity compared to concrete samples. This is based on the understanding that cyclodextrin will have more space of reactivity in mortar than concrete, since there is no coarse aggregate in mortar that can stand as a barrier to the reaction.

The results showed that the F/0.1CD/D sample exhibited highest reduction in sorptivity compared with FA/C, with approximately 29 % reduction at a 28 day curing period. Furthermore, the FA-β-CD composite dry mixes proved to be more effective in reducing the sorptivity than their corresponding solution mixes, in the case of both curing ages. This is attributed to the dissolution ability of the solution mix samples, which resulted in less dense mortar/concrete at the water front compared to the dried mix samples. Cylcodextrins in solution will try to occupy the pores as explained earlier, resulting in lower permeability, but its presence will cause the material at the space it occupies to be less dense and consequently increases sorptivity. In general, the sorptivity results agree with the permeability results in that FA-β-CD composite boosted the pozzolanic reaction.

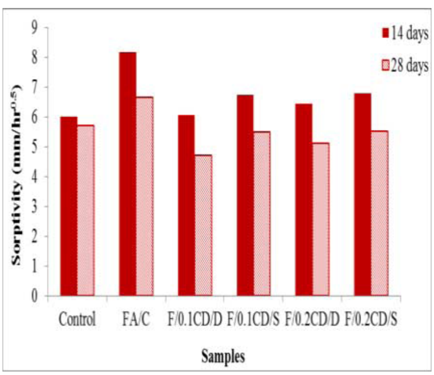

Figure 3. Sorptivity of mortar samples

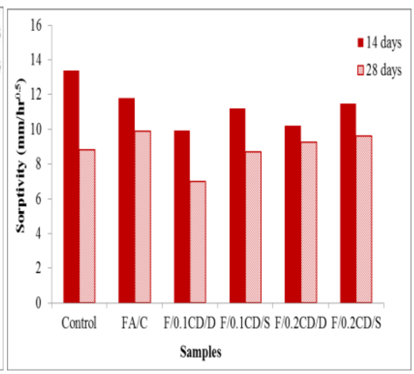

Figure 4. Sorptivity of concrete samples

3.4. Effect of βCyclodextrin on the porosity of FA/C mortar and concrete

The porosity results are reflection of what was observed in both the permeability and sorptivity tests. The trend of results observed in sorptivity was also observed in porosity results as shown in Figures 5 and 6. As curing age increased, porosity decreased. The samples with FA-β-CD composite exhibited lower porosity than the control sample and FA/C sample for both curing ages. A higher porosity was observed for FA/C mortar relative to the control samples at both curing ages, due to the slow pozzolanic reaction. In the case of the concrete samples, a lower porosity was observed for FA/C mix compared to the control sample at a curing age of 14 days, while higher porosity was observed for FA/C concrete compared to the control sample at a curing age of 28 days. As reflected in sorptivity results, the FA-β-CD composite dried mix samples showed lower porosity compared to FA/C sample than their corresponding solution mix samples for both curing ages. The above observations might be as a result of the explanation given for sorptivity results. The porosity results further buttressed the fact that FA-β-CD composite boosted the pozzolanic reaction.

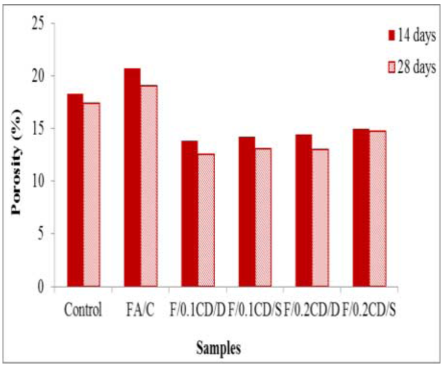

Figure 5. Porosity of mortar samples

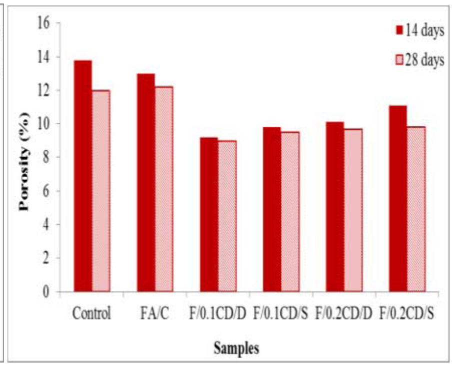

Figure 6. Porosity of concrete samples

4. Conclusions

The durability performance of FA-β-CD composite on mortar and concrete was investigated using oxygen permeability, sorptivity and porosity tests, based on the South African durability index tests. The FA-β-CD composite improved the flow of mortars and workability of concrete. The results obtained for the three tests indicated that FA-β-CD composite boosted the pozzolanic reaction, with the dried mix samples showing better sorptivity and porosity than solution mixed samples. Improved durability performance of pozzolanic concrete can be achieved when used as a composite with β-CD.

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