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INDICATIVE TESTS ON THE EFFECT OF FLY ASH - β CYCLODEXTRIN COMPOSITE ON CONCRETE WORKABILITY AND STRENGTH

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Abstract: *Fly ash – β cyclodextrin interaction has shown to have formed a composite that might be useful in concrete technology [1]. Since the effect of this composite on concrete properties has not been documented in an open literature, there is a need to run indicative tests that will give guidance for on-going research. This paper presents the results of the indicative tests on the effect of fly ash-β cyclodextrin composite on concrete workability and strength (compression and split tensile). All the mixtures included 30 % fly ash (FA) by mass. The β cyclodextrin (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, 0.0103 M, 0.0206 M and 0.0516 M β-CD solutions were added to the concrete at the mixing stage for solution mixtures. The results obtained showed that an increased content (0.5 %) of β-CD in concrete both for dry and solution mixtures had a detrimental effect on both workability and strength. However, lower percentages (0.1 % and 0.2 %) of β-CD improved both workability and the strength of the concrete.*

Keywords: β cyclodextrin, compressive strength, fly ash, split tensile strength, workability

BIOGRAPHY

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1 INTRODUCTION

One of the main aims of concrete technology research is to identify materials especially industrial waste that can be incorporated in concrete for the purpose of cost reduction, concrete durability, concrete sustainability and environmental protection. It would also be advantageous if such material is easily available. Fly ash (FA), a by-product of the coal combustion process is vastly available in South Africa and its contribution to concrete technology is of immense value as reported by researchers [2-7]. According to ESKOM (South African power utility) report released in 2011 [8], less than 6 % of the FA produced annually is being reused for different purposes. There is a need to increase the usage of FA for environmental protection. Excellent reviews of the general FA applications were done by AHMARUZZAMAN, 2010 [9] and BLISETT and ROWSON, 2012 [10]. Attempts to improve the usage of FA in concrete technology by modifying its chemical structure would be of a great interest to concrete technologists and researchers.

Research has shown that when polysaccharide (complex carbohydrate) is used optimally (0.5 % of cement) in concrete, it retards setting time, improves fluidity and workability and gives higher final compressive strength. The higher the polysaccharide content in concrete, the higher the detrimental effect it has on concrete [11-16]. However, this research aims at investigating the effect of oligosaccharide (less complex carbohydrates) on concrete, which has not been given attention. Cyclodextrins (CDs) are cyclic oligosaccharides, which are formed by the enzymatic modification of starch.

Fly ash – β cyclodextrin (β-CD) interaction has previously been shown, by the authors, to have formed a composite that might be useful in concrete technology [1]. β-CD is a type of cyclodextrin which is composed of seven α-(1,4)-linked glycosyl units. β-CD is commonly used because of its favorable complexities and low cost [17-18]. Since the effect of this composite on concrete properties has not been documented in an open literature, there is a need to run indicative tests that will give guidance to the on-going research. This paper presents the results of the indicative tests of the effect of FA-β-CD composite on concrete workability, compressive strength and split tensile strength.

2 MATERIALS AND EXPERIMENTAL METHODS

2.1 Materials and Mixtures

FA, β-CD and Portland cement (CEM I 52.5 N) were the binding materials used in this study. 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 CEM I 52.5 N cement type was obtained from Pretoria Portland Cement Company (PPC), South Africa. Granite crusher sand and coarse aggregate with a nominal size of size 22 mm were used in concrete mixtures. These aggregates were obtained from Afrisam, South Africa. The characteristics of β cyclodextrin as supplied by the producer and the chemical analysis of the fly ash used are presented in **Tables 1** and **2**, respectively. The fly ash used is classified as "Class F" since it has a sum of SiO_2, Al_2O_3, and Fe_2O_3 contents greater than 70 % by mass as speculated in ASTM C 618 [19].

Table 1: General characterisation β cyclodextrin used

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

Table 2: Chemical composition of the fly ash

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

Two samples preparation procedures were followed for FA-β-CD composites mixtures. Firstly, physical mixtures of pre-weighed amount of β-CD and fly ash were adopted for the dry mixtures and secondly, 0.0103 M, 0.0206 M and 0.0516 M β-CD solutions were added to the concrete at the mixing stage in the case of the solution mixtures. A proportion of 30 % fly ash (FA) was maintained in the mixtures proportions, 0.1 %, 0.2 % and 0.5 % β-CD were used together with FA in both dry and solution mixtures.

2.2 Experimental Procedure

2.2.1 Concrete mixtures and curing

A quantity of 0.06 m^3 concrete was batched for each of the eight mixtures produced, according to the mixture proportion presented in **Table 3**. Each batch catered for twelve 100 mm cubes and nine cylinders with a diameter and length of 150 mm and 300 mm, respectively. The mixture proportion for the control sample was maintained for all the samples, at the same time the effect of water content on workability was monitored, since the behaviour of β-CD on concrete had not been established. This approach was used to observe the behaviour of β-CD on fresh concrete and to establish a relationship that will form a basis for proper mixture proportion for on-going research.

During mixing, the rotary mixer with a 100 litre capacity was charged with coarse aggregate, fine aggregate and binders, respectively. These materials were mixed in their dry state for one minute. Thereafter, water was introduced into the mixture 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 and the cubes and cylinders were cast and compacted (using a vibrating table).The moulds containing the samples were covered with polythene sheets and left for 24 hours at room temperature before demoulding. 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 ± 2) °C for curing until the testing ages.

Table 3: Mixture proportions per 1 m^3 of concrete samples

Sample	Cement	Crusher sand (kg)	Coarse aggregate (kg)	FA (kg)	CD (kg)	CD sl.n (litres)	Water (kg)	W/B	Slump (mm)
Control	410	788	980	0.00	0.00	0.00	205	0.5	50
FA/C	287	788	980	123.00	0.00	0.00	205	0.5	90
F/0.1CD/D	287	788	980	122.59	0.41	0.00	192.7	0.47	95
F/0.2CD/D	287	788	980	122.18	0.82	0.00	172.2	0.42	120
F/0.5CD/D	287	788	980	120.95	2.05	0.00	172.2	0.42	130
F/0.1CD/S	287	788	980	122.59	0.00	35.00	192.29	0.47	150
F/0.2CD/S	287	788	980	122.18	0.00	35.00	171.38	0.42	150
F/0.5CD/S	2870	788	980	120.95	0.00	35.00	161.95	0.42	160

Note: Control (100 % Cement), FA/C (30 % FA + Cement), F/0.1 CD/D (29.9 % FA + 0.1 % CD dry + Cement), F/0.2 CD/D (29.8 % FA + 0.2 % CD dry + Cement), F/0.5 CD/D (29.5 % FA + 0.5 % CD dry + Cement), F/0.1 CD/S, (29.9 % FA + 0.1 % CD in solution (0.0103M) + Cement), F/0.2 CD/S (29.8 % FA + 0.2 % CD in solution (0.0206M) + Cement), F/0.5 CD/S (29.5 % FA + 0.5% CD in solution (0.0516M) + Cement)

2.2.2 Workability test

Workability was measured by slump test according to SANS 5862-1:2006 [20].

2.2.3 Compressive strength test

The compressive strength test was performed according to SANS 5863: 2006 [21] on each sample after 7, 14, 28, 90 and 180 curing days. Three samples were tested for each mixture at each curing testing age. The average of triplicate results was recorded as the strength at the relevant age. An Amsler compression testing machine, with a load capacity of 2000 kN was used. A uniaxial load was applied to the centrally placed sample, perpendicular to the direction of casting. The load was applied at a rate of 150 kN/minute.

2.2.4 Split tensile strength test

The split tensile test was performed on the cylinders, according to SANS 6253: 2006 [22] on each sample at ages of 14, 28 and 90 days after curing. Three samples were tested for each mixture at each curing age. The average of triplicate results was recorded as the strength at the relevant age.

The percentage decrease or increase in compressive strength or split tensile strength was calculated using **Equation (1)**, to determine the effect of β-CD on the strength of Fly ash Pozzolanic concrete (FA/C). Positive values indicate increase in the compressive or split tensile strength while negative value indicates a decrease in the compressive or split tensile strength.

$$\%increase\,/\,decrease = \frac{FA - \beta\ CD\ composite\ Comp.orSplit - FA\ pozzolanic\ Comp.orSplit}{FA\ pozzolanic\ Comp.orSplit} x100 \quad (1)$$

3 RESULTS AND DISCUSSION

3.1 Slump Test Results

Slump tests were carried out on the mixtures shown in **Table 3** as well on additional w/b ratios. **Table 4** shows the variation in slump of the samples at different water-binder ratio as observed in this study. At a W/B of 0.5, a collapse slump was observed in the case of samples with a β-CD content of 0.2 % or greater. In the case of W/B ratios lower than 0.5, the workability of the concrete increased with an increase in β-CD content. Furthermore, Mixtures s containing higher contents (0.5 %) of β-CD exhibited higher increases in slump with small increases in water. mixtures comprising β-CD solutions yielded higher increases in slump corresponding to small increase in water compared with samples made with powdered β-CD. This shows more dissolution with β-CD solutions in the samples. The slump was maintained between 50 and 150 mm for all samples.

It was observed that for samples containing β-CD (both powdered and solution), curing in water could not take place after 24 hours of casting. A few samples from these set that were cured in water after 24 hours of casting dissolved in the curing tank. Samples β-CD contents of 0.2 % and higher were left to cure in the mould and covered with a polythene plastic for three days (after casting) before being cured in water. The observation was in agreement with the fact that organic admixture (e.g. polysaccharides) act as retarders in concrete, which is attributed to the adsorption of the admixture to the surfaces of the hydrated and/or anhydrous phases of concrete [23-25]. This slows down the growth of calcium hydroxide and consequently retards setting [26]. The retarding effect observed will aid delay in the initial setting of concrete when special placement is required such as placing concrete in large piers, foundation or over considerable distances [27] and consequently might reduce early strength. However, longer concrete retardation than necessary might virtually prevent setting of concrete [25, 27]. The degree of retardation depends on the dosage of the admixture, method of application and curing conditions [27].

Lower contents of β-CD (0.025 % and 0.05 %) will be investigated as part of the on-going research so as to limit the effect of longer retardation of concrete and direct effect of β-CD on concrete without fly ash will be studied.

Table 4: Concrete slump test details (slump values given in [mm])

Sample	W/B 0.50	0.47	0.46	0.42	0.40	0.35
Control	50	-	No slump	No slump	-	-
FA/C	90	-	-	-	-	-
F/0.1CD/D	-	95	80	No slump	-	-
F/0.2CD/D	Collapsed	-	-	120	30	-
F/0.5CD/D		-	-	130	40	-
F/0.1CD/S	-	150	-	-	-	-
F/0.2CD/S	-	-	-	150	40	-
F/0.5CD/S	-	-	-	160	60	25

3.2 Compressive Strength Results

Figure 1 shows the relationship between the sample compressive strengths and curing ages. With the exception of the control samples, compressive strength increased with curing age up to 180 days. The control sample with no yielded a higher compressive strength at earlier curing ages than the remaining samples. From a curing age of approximately 125 days, the FA- β-CD composite samples exhibited a higher compressive strength than the control and FA/C samples, except for composite samples with 0.5% β-CD. The mixtures containing the highest content of β-CD (0.5 %) yielded the lowest compressive strength values at all the curing ages. This might be as a result of the reduction in concrete cohesiveness at higher content of β-CD, which resulted in longer setting time and loss of strength. The highest compressive strength value was observed with sample containing 0.1% β-CD. No trend was identified regarding the strength of the concrete mixtures containing the dried β-CD versus the solution β-CD composite samples. The variations in the mixture W/B ratio and workability might also have affected the overall strength results. Lower W/B ratio and higher workability generally aid compressive strength. Further research on this investigation will adopt lower contents of β-CD so as to maintain the W/B ratio of the mixture.

The percentage increase in FA-β-CD composite samples compressive strength as compared to FA/C sample compressive strength is presented in **Table 5**. From this Table it is obvious that the strengths of F/0.5CD/D and F/0.5CD/S samples are far lower than the strength of FA/C, therefore these samples were excluded from further discussion. One of the characteristics of pozzolans is the reduced compressive strength at early curing age [28-29]. The incorporation of the FA- β-CD composite in the mixture improved early compressive strength, with highest increase of 45.05% observed for the F/0.1CD/D sample at 7 days curing age as compared to the FA/C samples. The results indicated that FA-β-CD composite boosted the pozzolanic reaction with evidence of increased compressive strength at all ages. This might be as a result of reduced pore sizes of FA-β-CD composite compared to FA, as reported previously by the authors [1], lower water content of FA-β-CD composite samples and higher workability. Also, there is a structural interaction between fly ash and β-CD according to the previous study by the authors [1]. The FT-IR results from the previous study [1] showed stronger intensities of Si-O-Si asymmetric stretching in the composite formed between fly ash and β-CD, which was claimed to be as a result of the C-O deformation vibration in β-CD. The presence of Al-O-Si bonding was also observed in the composite. The positive effect of β-CD on final strength can be attributed to improved pozzolanic reaction due to stronger Si-O-Si and Al-O-Si bonding present in the composite, which allows more active SiO_2 to react with $Ca(OH)_2$ to form more CSH (calcium silicate hydrate) that is responsible for strength formation. Finally, in general, the higher the age of curing, the less the effect of the β-CD on the compressive strength increases relative to the FA/C mixtures.

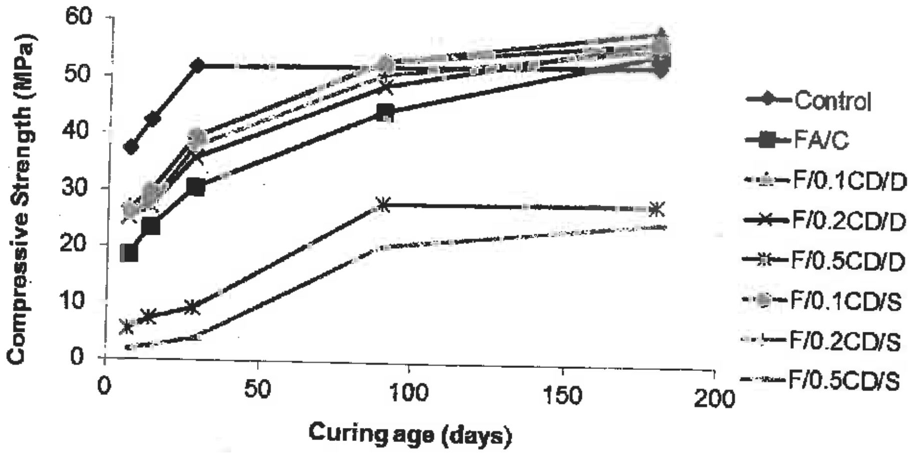

Figure 1: Compressive strength of samples at different curing ages

Table 5: Percentage increase in FA-β-CD composite samples compressive strength compared to pozzolanic sample compressive strength (mean ± SD, n = 3)

	7 days [%]	14 days [%]	28 days [%]	90 days [%]	180 days [%]
F/0.1CD/D	45.02 ± 2.65	26.38 ± 3.84	29.38 ± 1.08	19.84 ± 1.04	7.27 ± 0.65
F/0.1CD/S	42.70 ± 4.63	26.81 ± 4.76	29.71 ± 1.37	19.62 ± 0.28	4.05 ± 0.46
F/0.2CD/D	35.54 ± 0.91	15.87 ± 0.88	17.26 ± 0.71	9.89 ± 0.91	4.18 ± 0.30
F/0.2CD/S	43.94 ± 2.99	17.45 ± 1.84	23.81 ± 3.77	14.61 ± 1.83	1.81 ± 0.54

3.3 Split Tensile Strength

Concrete is generally weak in tension and strong in compression, therefore it is expected to have a lower split tensile strength than compressive strength, as it is revealed in **Figure 2** for all the samples. The FA-β-CD composite samples with a relatively low content of β-CD (0.1 % and 0.2 %) exhibited a higher split tensile strength than the pozzolanic samples (FA/C) for all curing ages (up to 90 days). Furthermore, these samples yielded higher split sample strength than the control sample at a curing age of 90 days. As also observed for compressive strength, higher content of β-CD (0.5 %) in the mixture resulted in unsatisfactory lower split tensile strength. The split tensile strength of dried mixed composites samples and solution mixed composite samples are closely related.

When comparing the pozzolanic sample (FA/C) split tensile strength and the FA-β-CD composite samples with lower content of β-CD (0.1 % and 0.2 %) split tensile strengths, it was observed that at 14 days curing period, the FA/C samples generally exhibited a higher split tensile strength than FA-β-CD composite samples as presented in **Table 6**. However, the percentage increase in the split tensile strength for FA-β-CD composite samples was higher than that of FA/C samples at curing ages of 28 and 90 days, resulting in an increase in split tensile strength of up to 28.74 % as observed for F/0.1CD/D sample at 90 days curing period. Hence, in general, the longer the curing period, the greater the effect of the β-CD on the split tensile strength of the concrete. In addition, the inclusion of 0.1 % β-CD in the mixtures resulted in an improved tensile strength compared to the strength achieved when including 0.2 % of β-CD.

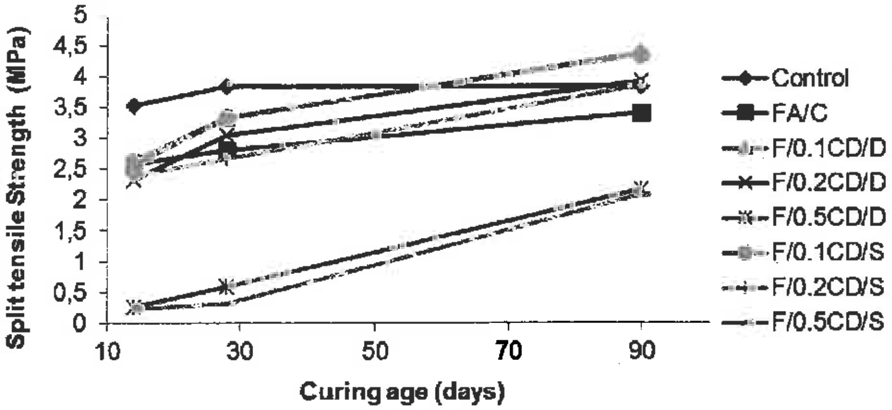

Figure 2: Split tensile strength of samples at different curing ages.

Table 6: Percentage increase/decrease in FA-β-CD composite samples split tensile strength compared to pozzolanic sample split tensile strength (mean ± SD, n = 3)

	14 days [%]	28 days [%]	90 days [%]
F/0.1CD/D	-0.78 ± 0.95	18.15 ± 1.7	28.74 ± 0.28
F/0.1CD/S	1.94 ± 0.66	18.86 ± 2.79	27.86 ± 0.72
F/0.2CD/D	-9.69 ± 2.21	8.54 ± 1.62	15.25 ± 0.84
F/0.2CD/S	-7.36 ± 1.28	-4.98 ± 2.79	12.90 ± 0.72

4 CONCLUSION

The indicative tests results on the effect of fly ash-β cyclodextrin composite on concrete workability and strength presented in this article give guidance to the on-going research. The results showed that higher content of β-CD (0.5 %) in concrete has detrimental effect on workability and strength for both dry and solution mixtures. However, lower percentages of β-CD (0.1 % and 0.2 %) improved workability without reducing the cohesiveness of concrete, increased compressive strength at all ages compared to pozzolanic sample and increased split tensile strength at curing ages of 28 and 90 days when compared to pozzolanic sample. The positive compatibility of fly ash and β-CD at lower percentages of β-CD (0.1 % and 0.2 %) to produce better pozzolanic concrete is an advantage towards utilizing fly ash more in concrete, which in turn will reduce construction cost and environmental pollution arises from dumping fly ash on the dump hill. The effect of β-CD on the pozzolanic sample is attributed to the stronger Si-O-Si and Al-O-Si bonding present in the FA-β-CD composite [1], which allows more active SiO_2 to react with $Ca(OH)_2$ to form more CSH (calcium silicate hydrate) that is responsible for strength formation.

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