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XRD, FTIR and SEM studies on calcined sugarcane bagasse ash blended cement

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ABSTRACT

The utilization of waste material as mineral admixture in cement provides a satisfactory solution to some of the environmental concerns and problems associated with waste management. Agro waste such as Sugarcane Bagasse Ash (SCBA) is one among such a potential material. In this study, the effect of partial replacing of cement with 10% calcined SCBA on the properties of produced cement pastes were analyzed using Compressive strength measurements, XRD, FTIR, and SEM techniques. The spectroscopic techniques were performed at 7 and 28 days to identify the hydration products such as AFt, AFm, C-S-H and $Ca(OH)_2$ and the obtained results were compared with Strength measurements.

Keywords: Cement, SCBA, Calcinations, Compressive strength, Hydration reactions.

INTRODUCTION

Bagasse is a byproduct during the manufacture of sugar and it has high calorific value. It is utilized as a fuel in boilers in the sugar mills to generate steam and electricity. The obtained Sugarcane Bagasse Ash causes a great disposal problem. Using waste SCBA as a pozzolanic material to replace cement can reduce the consumption of cement and reduce landfill area requirements. This is turn helps solve environmental issue caused by cement production, decreasing both energy based and CO_2 emissions. It is well known that CO_2 is a major contributor to the greenhouse effect and the global warming of the earth [1].

Several studies [2-6] have found that the use of SCBA as a mineral admixture in cement can improve the strength of the cementitious materials. According to Martinera Hernandaz et al [7] reported that the main products from the reaction between $Ca(OH)_2$ and SCBA are calcium-silicate-hydrate (C-S-H). Usually SCBA may contain black particles due to the presence of carbon. To eliminate the carbon content, SCBA burns under controlled conditions (600°C) for 4 hours. According to Sing et al [8], the ash produced by controlled burning of agro waste materials below 700°C transforms silica content of the ash in to amorphous silica and can be used as a pozzolanic material.

In the present work investigates the pozzolanic activity of SCBA (fired at 600°C) by the way of measuring compressive strength. In order to verify the pozzolanic reaction of SCBA admixtured paste were evaluated by FTIR, XRD and SEM techniques.

MATERIALS AND METHODS

In the present study, the OPC was a commercially available and SCBA was obtained from E.I.D.Parry Sugar Mill, Nellikuppam, Tamil Nadu, India. As received ash containing unburnt matter, silica and alumina. To reduce this content, SCBA was fired under controlled temperature at 600°C for 4 hours in muffle furnace. After cooling, the ash was ground in to fine powder and allowed to pass through a sieve of 75µm and stored in desiccators for further use. The chemical composition of the OPC and SCBA were identified by XRF (Table.1.).

Table.1.Chemical compositions of OPC and SCBA

OXIDES	*OPC*	*SCBA*
SiO_2	21.40	70.97
Al_2O_3	6.03	8.55
Fe_2O_3	4.40	3.61
CaO	61.14	6.50
MgO	1.65	2.83
K_2O	0.58	1.77
Na_2O	0.24	0.92
TiO_2	-	0.53
MnO	0.25	0.18
P_2O_5	-	0.78
SO_3	2.33	0.80
Insoluble	1.43	-
Loss On Ignition	0.55	2.56

The Chemical analysis of data indicates that SCBA has approximately four times high silica content than OPC and also contains considerable amount of Al_2O_3, Fe_2O_3 and CaO.

Compressive Strength

The compressive strength of hydrated OPC and SCBA admixtured cement mortar (5%, 10%, 15% and 20%) were determined using compression testing machine. Mortar specimen size of (7×7×7 cm) were prepared and cured with different ages (1, 7 and 28 days). These cubes were taken out from water prior to compressive testing.

Sample Preparation

The OPC and OPC + 10% SCBA admixtured cement paste were prepared and its hydration were stopped after 7 and 28 days by adding acetone. The hydrated samples were heated at 120°C for 2 hours [9]. The dryed samples were grounded using agate mortar and the powdered samples were kept in desiccators for further use. XRD spectrums were taken using a XRDML Gonio X-ray diffractometer with Cu-Kα radiation operated at 40 kV and 30 mA. The scan rates are 0.1 to 120 degrees with 2°/m in the Bragg's angle range (2θ) from 10° to 70°. FTIR measurements were carried out using a Avator–Nicolet 360 spectrophotometer. The microstructures of samples were performed with JEOL make SEM.

RESULTS AND DISCUSSION

Fig.1. shows the compressive strength of OPC compared with that of admixtured cements containing 5%, 10%, 15% and 20% SCBA.

As expected, compressive strength values increase progressively with curing time. The compressive strength of the SCBA admixtured cements are higher than those of the OPC samples at 1 day and latter.

The increase in strength between 1 and 28 days is more significant for SCBA 10%, when compared to other mixtures with SCBA. The increase in strength may be partly result from the filler effect and the pozzolanic reaction between reactive SiO_2 from the SCBA and $Ca(OH)_2$ from cement hydration. However at 15 and 20% SCBA results in a decrease in compressive strength, compared to 10% SCBA admixtured samples, due to the dilution effect [5]. The current research further focused to analyse the 10% SCBA admixtured cement paste.

XRD analysis of the OPC and SCBA paste

An XRD analysis used to study the pozzolanic behavior of the SCBA used in this research and to identify the hydration products formed during the hydration of OPC. Fig.2. shows the XRD pattern of OPC and 10% SCBA admixtured cement at 7 days and 28 days. It can be seen that diffraction peaks of ettringite, $Ca(OH)_2$ and C-S-H phases appeared in all samples obtained during hydration. The main obvious change in SCBA samples is the peaks of $Ca(OH)_2$ diminished, whereas those of C-S-H increased [10]. This indicates the pozzolanic reaction between $Ca(OH)_2$ and amorphous silica present in SCBA. This additional C-S-H improves the strength. Low intensity peaks for AFt formation are observed, this may be due to the conversion of AFt into AFm phase after 1 day of hydration [8].

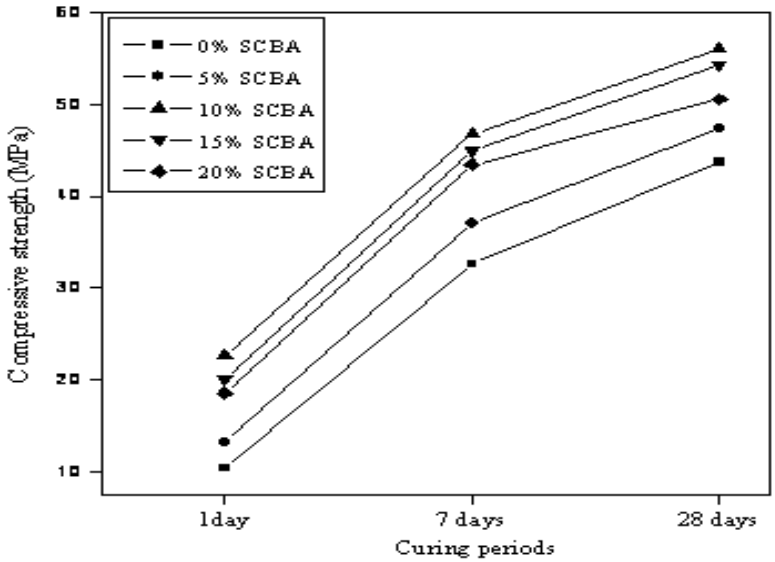

Fig.1. The compressive strength of OPC and OPC + 10% SCBA

FTIR analysis of the OPC and SCBA paste

The FTIR patterns of hydration products of OPC and 10% SCBA admixtured pastes after 7 days and 28 days curig times are shown in Fig.3. In the OPC paste, the increase in band observed at 3630 cm^{-1} which is due to the stretching vibration of OH group of $Ca(OH)_2$, whereas it decreases with the progress of hydration in the SCBA admixtured paste [11]. The peak at 3410 cm^{-1} is due

to water and the bands at 1425 cm^{-1} and 875 cm^{-1} are attributed to presence of $\gamma_3 - CO_3^{2-}$ and $\gamma_2 - CO_3^{2-}$ of calcite, respectively [12].

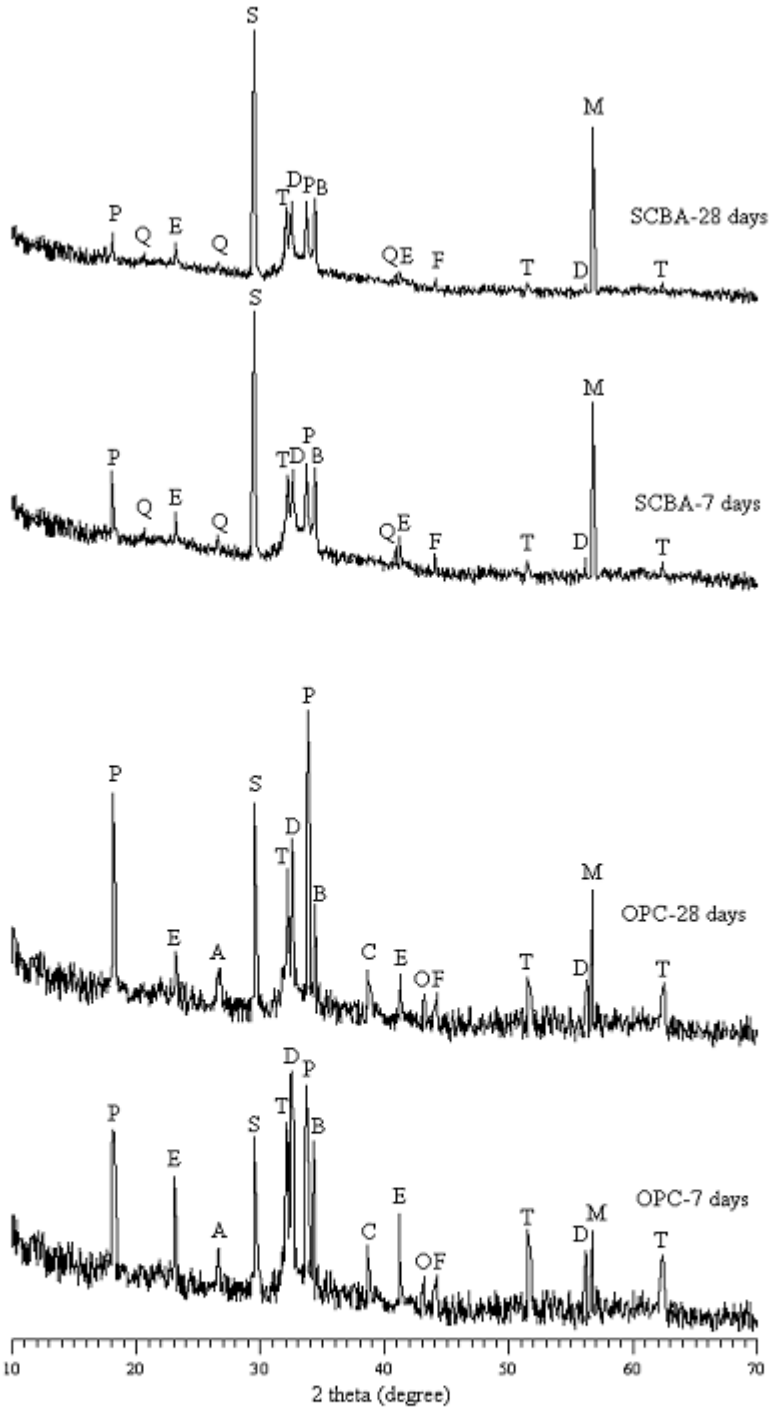

Fig.2. The XRD spectra of hydrated OPC and OPC + 10% SCBA paste at 7 days and 28 days

P-$Ca(OH)_2$; E-Ettringite ; A-C_3A ; T-C_3S ; D-C_2S ; B-C_3S/C_2S
O-MgO; F-C_4AF; S-CSH; M-Monosulphate ; Q-Quartz ; C-$CaCO_3$

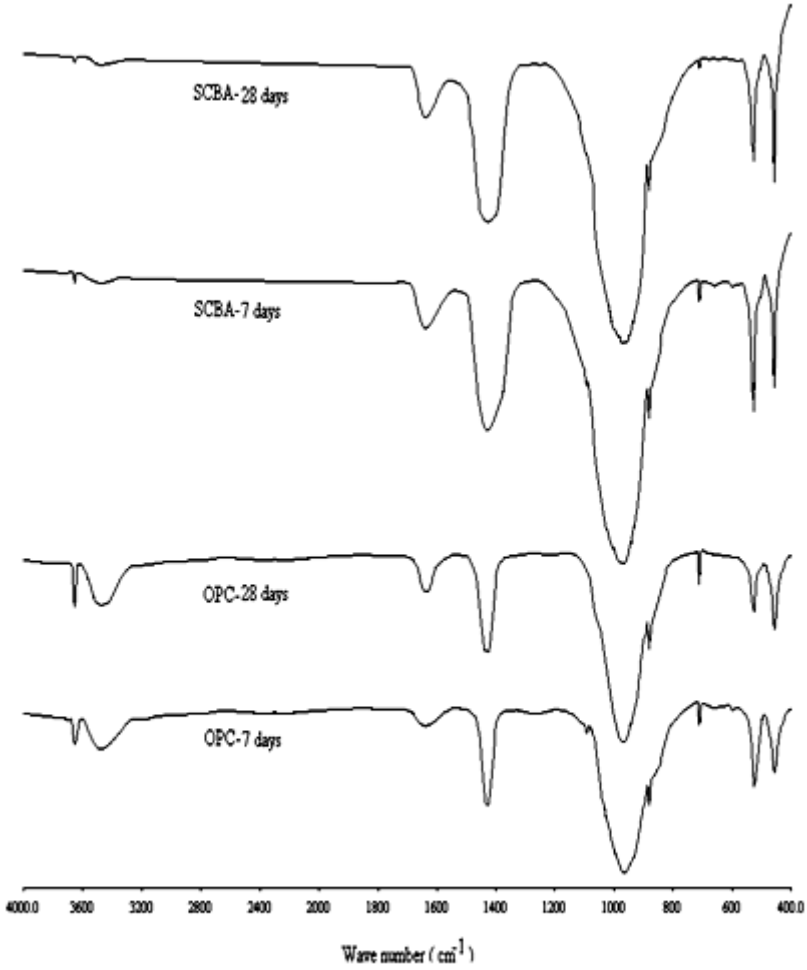

Fig.3. The FTIR spectra of hydrated OPC and OPC + 10% SCBA paste at 7 days and 28 days

The results are in harmony with XRD analysis. It should be observed that the band at 950 cm^{-1} region is due to Si-O stretching vibration of C-S-H and increases with the stage of curing indicating increasing the formation of C-S-H gel [13]. The higher intensities of the bands (970 cm^{-1}) at 7 and 28 days shows a greater extent of reaction product formation due to SCBA, which contributes finely the development of compressive strength (Fig.1.).

SEM analysis of the OPC and SCBA paste

The microstructure of cement paste without and with the replacement of 10% SCBA at 7 days and 28 days are shown in [Fig.4,5]. Needle-like formation of ettringite, C-S-H and crystals of $Ca(OH)_2$ were observed in the cement paste.

The structure becomes more denser at 28 days [14, 7]. Fig.5 shows the micrographs of samples containing 10% SCBA at 7 days and 28 days of curing. In the micrographs only C-S-H is appeared and that hexagonal $Ca(OH)_2$ crystals are not appeared. This shows SCBA being an efficient pozzolanic material as a consequence of its high silica content.

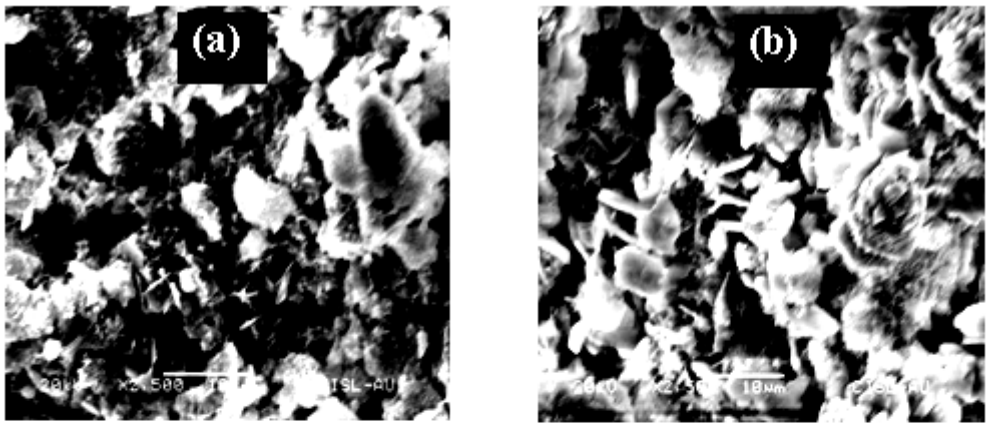

Fig.4. The SEM micrographs of OPC (×2500)
(a) 7 days and (b) 28 days

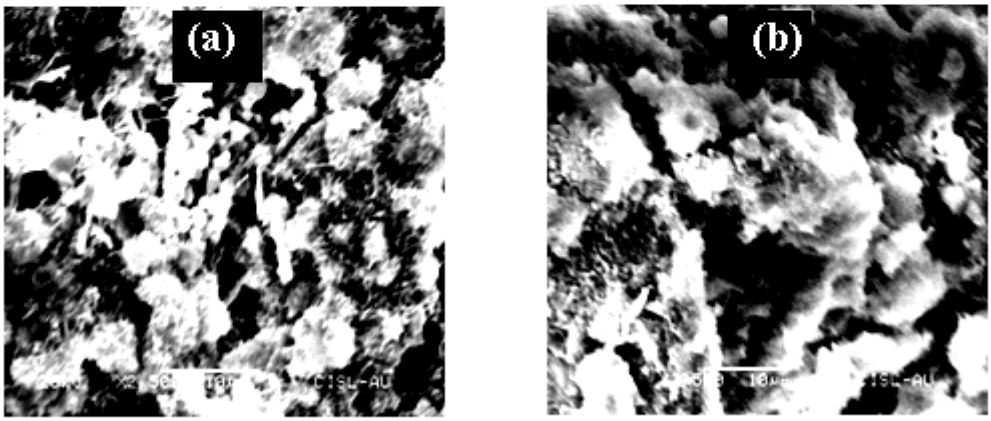

Fig.5. The SEM micrographs of OPC + 10% SCBA (×2500)
(a) 7 days and (b) 28 days

CONCLUSION

From this study, SCBA calcined at 600°C has good pozzolanic properties, suggesting suitability for use in cement. The replacement of 10% cement with SCBA effectively increases the compressive strength. The increase in the use of SCBA could reduce environmental problems and minimize the requirement of landfill area to dispose of the SCBA.

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