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Pozzolanic potential of Sugarcane Bagasse Ash as verified through TGA and XRD Techniques

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Abstract: *Sugarcane bagasse ash (SCBA), a sugar-mill waste, has the potential of a partial cement replacement material if processed and obtained under controlled conditions. This paper discusses the reactivity of SCBA obtained by control burning of sugarcane bagasse procured from Punjab province of India. X-ray diffraction (XRD) and Thermo-gravimetric Analysis (TGA) techniques were employed to ascertain the amorphousness and morphology of the minerals ash particles. Ash-blended cement paste specimens were analyzed by XRD, thermal analysis, and SEM methods to evaluate the hydration reaction of SCBA with cement. Results showed that the SCBA processed at 600°C for 5 hours was reactive as ash-blended mortar specimens with up to 15% substitution of cement gave better strength than control specimens.*
Keywords - *Bagasse ash, Blended mortar, Pozzolanic Activity, X –ray diffraction, SEM, Thermal Analyses.*

I. Introduction

Sugarcane bagasse ash (SCBA) is an abundantly available waste of sugar-mills. Sugarcane is one of the major crops grown in over 110 countries and its total production is over 1500 million tons (FAO, 2006). After the extraction of all economical sugar from sugarcane, about 40-45% fibrous residue (Deepchand, 1986) is obtained, which is reused in the same industry as fuel in boilers for heat generation that leaves behind 8 -10 % ash as waste (Payá et al., 2002), known as SCBA. It contains high amounts of un-burnt matter, silica, aluminum and calcium oxides (Deepchand, 1986). It is a very valuable pozzolanic material if carbon free and amorphous ash could be obtained by further combustion. Sugarcane production in India is over 300 million tons/year. The processing of it in sugar-mills generates about 10 million tons of SCBA as waste material. Very few studies have been reported on use of bagasse ash directly obtained from the sugar-mills A few studies (Baguant, 1995; Hernández et al., 1998; Singh et al., 2000) have reported on the suitability of sugar cane bagasse ashes as partial cement replacement binders that are obtained directly from the sugar-mills. Present study was carried out to evaluate the SCBA processed and obtained by control burning of sugarcane bagasse which was procured from the Punjab province in India. After characterizing the SCBA with regards to its chemical and physical properties, hydration reaction of SCBA with cement was analyzed by X-ray diffraction (XRD) and thermal analyses(TGA) techniques.

II. Experimental Program

2.1 Ash Production from Sugar Cane Bagasse

In order to obtain amorphous SCBA, optimum burning with respect to time and temperature was evaluated by conducting trial burnings of sugarcane bagasse at 400, 500, 600, 700 and 800°C for 3, 5, 6 and 8 hours (James and Rao, 1986; Chandrasekhar et al., 2006; Ajay et al., 2007). Biricik et al. (1999) on wheat straw and Patel (1987) on rice husk; have reported that burning time, temperature, cooling time, and grinding conditions effect the pozzolanic reactivity of the ashes. Carbon contents (%) were measured after each burning (Fig.1**)** which indicated that combustion was almost complete at 800°C for 5 hours burning. Visvesvaraya (1986) have reported that crystallization of minerals occurs at temperatures higher than 650°C. Chopra et al. (1981) have also reported that at burning temperatures up to 700°C, silica remains in amorphous form and silica crystals grow with increase in the time of incineration. Hence considering the rate of burning, residence time and the carbon contents, the suitable burning condition was identified as 600°C for 5 hours. The temperature was raised at a rate of 5°C/minute with residence time of 3 hours. At this condition brownish white color indicated complete burning. Amorphous nature of the ash was further ascertained by XRD analysis.

To obtain SCBA for further tests, burning was carried out in two stages – open burning of sugarcane bagasse to reduce the volume of dry matter, followed by controlled burning at 600°C for 5 hours in a thermostatically controlled electronic furnace (KDFP-90). To achieve fineness comparable to OPC, the SCBA obtained after burning was grinded in a ball mill (25×35g – 30mm ϕ balls) for about 4 hours and subsequently screened through 53μm (No.270) sieve (Nair et al., 2006).

Pozzolanic reactivity of SCBA with OPC was evaluated by studying the mineralogy and morphology of hydrates present in the ash blended hydrated pastes, with XRD and TGA

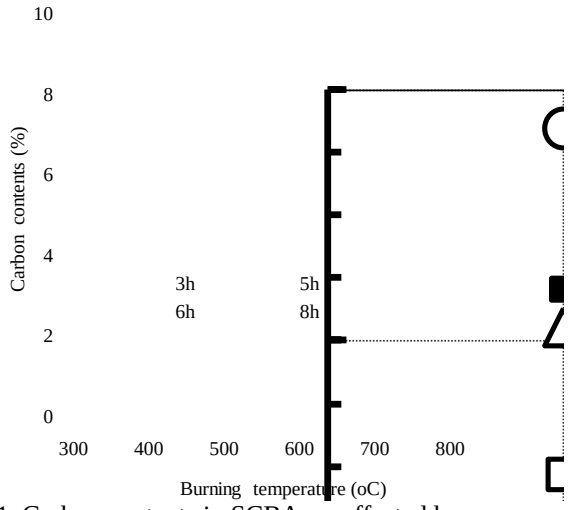

Fig.1. Carbon contents in SCBA, as affected by burning temperature and residence time

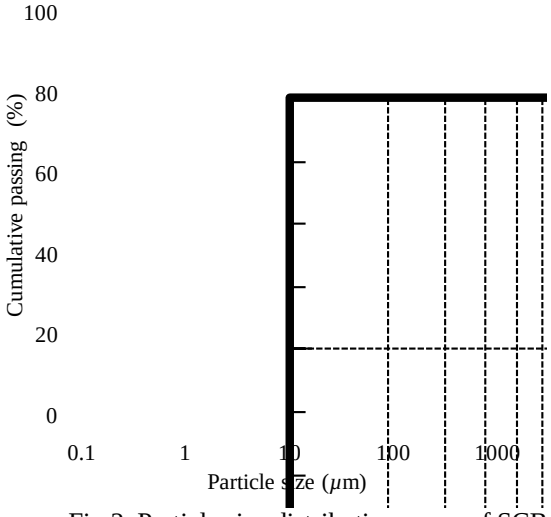

Fig.2. Particle size distribution curve of SCBA samples

2.2 Preparation of Blended Paste Specimen

To study the effect of ash substitution on hydration, mineralogical and morphological studies of hydrated pastes were conducted. For this, pastes were prepared, replacing OPC with 10, 15, 20 and 25% SCBA and water cementitious material ratio (*w/c*) as 0.35. Effect of SCBA on the water consistency was also measured as shown in Table 1. Effect of SCBA substitution on the setting time was evaluated directly by measuring the initial and final setting time. The pH values were also measured to check the effect of SCBA substitution on the alkalinity of freshly prepared blended pastes. The distribution curve has been presented in Fig 2.

Micro-structural features and mineralogical composition of the hydration products formed in CTR and ash-blended pastes were ascertained and compared after 28 days and 91 days of curing by using XRD and thermo-gravimetric tests.

Table.1. Water consistency[a], setting time[a], flow[b] and pH values[b] of CTR and ash-blended specimens

Specimen	W.C. (%)	IST (min)	FST (min)	pH value*	Mortar flow (mm)
CTR	33.0	125	230	12.5	178
SC-10	33.5	145	300	12.6	168
SC-15	33.8	155	310	12.6	165
SC-20	34.2	160	310	12.7	165
SC-25	34.5	170	315	12.7	160

W.C.: Water consistency, IST : initial setting time

FST : final setting time

a: measured for paste specimens (average of 3 readings)
b: measured for mortar specimens (average of 6 readings)

III. Result And Discussion

3.1 Physical and Chemical Properties

Physical and chemical properties of SCBA in comparison to OPC are shown in Table 2. SCBA has low density and higher surface area (Blaine surface area) as compared to OPC. The combined chemical composition; $SiO_2 + Al_2O_3 + Fe_2O_3$ (>70%) and CaO (>10%) testified the pozzolanic and cementitious nature of SCBA as per ASTM C 618-03 specifications. Particle size distribution curve of SCBA samples (Fig. 2)

indicated that average size of the ash particles was 28.9 μm. Kraiwood et al. (2001) has reported that large surface area favors the pozzolanic reactivity of amorphous silica and other minerals.

Table.2. Physical and chemical properties of OPC and SCBA

	Chemical properties								Physical properties			
	Chemical Composition (wt.%)								Density	Blaine surface area	Particle size	color
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	LOI	(g/cm^3)	(cm^2/g)	(μm)	
OPC	18.40	5.61	3.05	66.80	1.42	2.84	0.50	2.00	3.15	3,250	36.22	Dark grey
SCBA	62.43	4.38	6.98	11.81	2.51	1.48	3.53	4.73	2.52	5,140	28.92	Reddish grey

3.2 Analyses of Hydrates in SCBA-blended Cement Pastes

Different hydrates formed in SCBA-blended cement pastes were examined and analyzed by XRD and thermal analyses. Discussion is mainly based on the typical pozzolanic reaction of SiO_2 and alumina (Al_2O_3) present in SCBA with available calcium hydrates CH in the hydrated gel, forming additional calcium silicate hydrate (C-S-H) and calcium aluminate silicate hydrates (C-A-S-H)

3.2.1 X-ray Diffraction (XRD) Analyses

Comparison of XRD patterns (Fig.3) obtained for SCBA-blended and CTR paste specimens after 28 days and 91 days of curing showed that in 91 days cured specimens; the intensity of peaks 'C' representing CH was significantly reduced with corresponding increase in the C-S-H peaks represented by 'S'. Also the peaks 'Q' at 2θ = 26.66°, representing SiO_2 got diminished in 91 days cured specimens than as compared to 28 days specimens. It was a clear indication of the fact that the free CH available in 28 days cured specimens was gradually consumed by the excess amount of SiO_2 present in the SCBA. As shown in Fig.3, the intensity of CH peaks in the SCBA-blended specimens got diminished with the appearance of additional peaks. According to Pane and Hansen (2005), these peaks represent the formation of additional C-S-H and C-A-S-H. It confirmed the pozzolanic reactivity of SCBA beyond any doubt. Appearance of peak representing C_4AH_x was also noticed in SC-20 and SC-25 specimens. Gengyiong and Xiaohua (2003) have reported that these products act as nucleating sites that hinder the further hydration reaction. This explained the reason for low strength development in specimens with higher percentages of SCBA. Higher peaks of SiO_2 were noticed in SC-20 and SC-25 paste specimens, both in 28 and 91 days patterns, which indicated that more amount of SCBA added more silica, and was left un-reacted. This could be another reason for low strength development in specimens with higher percentages of SCBA in SC-20 and SC-25 specimens.

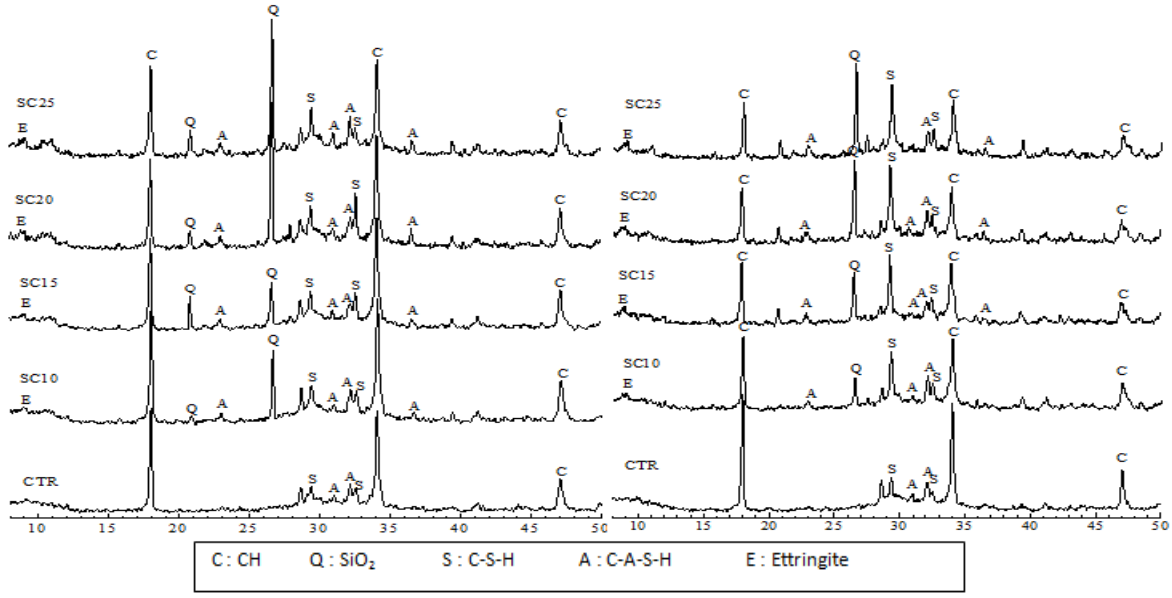

Fig.3. Comparison of XRD patterns obtained for SCBA-blended and CTR paste specimens after 28 days and 91 days of curing

3.2.1 Thermal Analyses

According to Langan et al. (2002); Pane and Hansen (2005), differential thermal analysis (DTA) combined with thermo-gravimetric analysis (TGA) is more suitable for studying the hydration or pozzolanic reaction that takes place at later stages of hydration. DTA locates the ranges corresponding to thermal decompositions of different phases in the hydrated paste, while TGA measures the simultaneous weight loss due to the thermal decomposition. In this study thermo-gravimetric tests were performed on Rigaku-TG810 ID Thermoflex TAS200 by gradually raising the temperature from 20°C to 1,000°C at a rate of 6°C/min. TGA and DTA diagrams for 91 days cured CTR and ash-blended specimens are shown in **Fig.4**and **Fig.5**, respectively. DTA diagrams show two significant peaks for CTR and all ash-blended specimens hydrated at 91 days; (a) endothermic peak at 130 – 150°C indicate the hydration of C-S-H (Mackenzie, 1972; El-Didamony et al., 1996) and (b) the peaks at temperatures between 420 - 480°C correspond to CH decomposition (Oriol and Pera, 1995). As seen from the DTA curves, the endo-thermal effects in the temperature range of 100 – 150°C was attributed to the loss of free water and dehydration of inter-layer water from the C-S-H phase (Mackenzie, 1972; El-Didamony et al., 1996). A small step obtained at temperatures close to 700°C was probably due to the release of CO_2. It was through the decomposition of $CaCO_3$ formed by carbonation (Taylor, 1993).

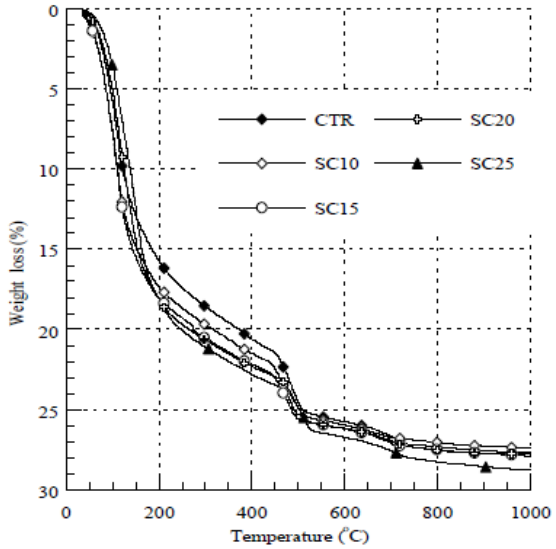

Fig.4 TGA diagram of CTR and SCBA-blended specimens tested at 91 days of hydration hydration

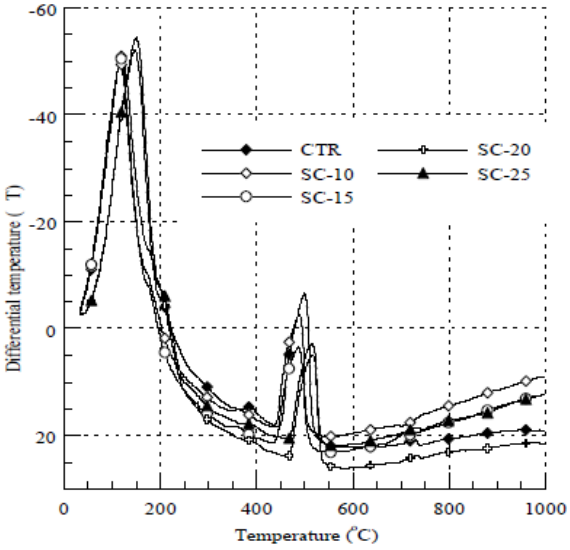

Fig.5 DTA diagram of CTR and SCBA blended specimens tested at 91 days of

As reported by Midgley (1979), the CH can be measured by the amount of water loss, which is very close to the water present in CH and therefore, is proportional to the amount of CH. Loss of water (%) measured for 91 days hydrated pastes, representing the amounts of CH at temperatures between 420 – 480°C and corresponding amounts of C-S-H at temperatures between 70 – 400°C is shown in Table 3.

In CTR, the amount of CH was higher (2.19) than that present in the SC-10 (2.00) and SC-15 (2.16). It indicated the presence of excess amount of CH available in the CTR. Corresponding amounts of C-S-H, in SC-10 and SC-15 were higher than that present in the CTR. It clearly indicated that excess amount of CH formed in the SCBA-blended specimens was consumed by the SiO_2 present in the SCBA with the formation of additional C-S-H. It was a typical pozzolanic reaction as explained by Ramachandran et al. (2003). Abrupt loss of weight between temperatures 420 – 480°C was also associated with the de-hydroxylization of CH (Orial and Pera 1995), and it was less in SC-10 and SC-15 (Table 3). It further indicated that the amount of CH present in these samples at 91 days hydration was less as compared to the CTR and other ash-blended specimens. Formation of these hydrates indicated that pozzolanic reaction took place between the SiO_2 present in the SCBA and the free CH present in the cement. Up to 15% substitution of OPC with SCBA was enough to consume the excess CH present in the hydrates. With more than 15% substitution of OPC with SCBA, the amount of CH increased with corresponding decrease in the amount of C-S-H hydrates.

Table.3. Thermal analysis of 91 days hydrated CTR and SCBA-blended paste specimens

Specimen	Weight loss (%)		
	C-S-H*	CH**	Total
	70 – 400^{o}C	420 – 480^{o}C	70 – 700^{o}C
CTR	17.02	2.19	24.30
SC-10	17.92	2.00	23.83
SC-15	17.64	2.16	24.16
SC-20	16.95	2.27	27.17
SC-25	16.87	2.27	25.59

*C-S-H : Calcium silicate hydrate **CH : Calcium hydroxide

Overall analyses of TGA and DTA data proved that SC-10 and SC-15 reacted more than the other blends thus signifying 15% SCBA as optimum percentage of OPC substitution. This trend was also vindicated by the XRD analyses and mechanical strength test results as explained in earlier sections.

IV. Conclusion

Following conclusion can be drawn from the present study:

- Controlled burning of bagasse at 600^{o}C for 5 hours produced amorphous bagasse ash with very low carbon contents in it. Processed SCBA possessed high specific surface area, high percentage of amorphous silica and calcium oxide which fulfilled the principal requirements of a pozzolanic material.
- Thermal analyses (DTA/TGA) of hydrates, and interpretations of XRD diagrams and observations of the SCBA-blended pastes confirmed the pozzolanic reactivity of the SCBA.