

Utilization of Bagasse Ash as Supplementary Cementitious Material

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Abstract— The physico-chemical characterization of raw bagasse ash have been studied by XRF, XRD, TGA, FTIR and SEM. Raw bagasse ash composed of unburned carbon, amorphous silica, crystalline silica in form of quartz, tridymite and cristobalite in addition to residual amounts of calcium carbonate, silicate and aluminosilicate minerals. Raw bagasse ash have been calcined at 700-1000°C for 1-5 hrs. Calcination of bagasse ash transforms amorphous silica into quartz at 700°C which in turn transform into tridymite after 800°C. The pozzolanic activity of calcined bagasse ash was evaluated by estimating the fixed lime content and XRD, TGA, FTIR and SEM analyses. The pozzolanic activity decreases with calcination temperature of bagasse ash. Because crystalline silica (quartz, tridymite and cristobalite) have low pozzolanic activity. It was recommended that calcination of bagasse ash at 700°C up to 3 hrs is the optimum conditions required to produce calcined bagasse ash with high pozzolanic activity. The hydration characterization of 5-20 wt % calcined bagasse ash blended cement pastes have been evaluated by compressive strength, combined water content, bulk density and total porosity as well as XRD, TGA, FTIR and SEM analyses. At the early ages of hydration, calcined bagasse ash acts as a filler whereas at later ages it acts as pozzolana. Increasing calcined bagasse ash content make a dilution effect, require higher water demands and forms a layer around anhydrous cement grains delaying the hydration of cement.

Keywords— Bagasse ash; Calcination; Pozzolanic activity; Hydration; Blended cement paste.

I. INTRODUCTION

Bagasse ash is the waste generated by the combustion of bagasse in boilers of sugar factories for cogeneration of heat and electricity. Around 0.005-0.066 tons ash is generated per ton of sugarcane crushed [1]. Apart from silica that is the major component, it contains other metal oxides as well as unburned carbon because of incomplete calcination condition inside boilers [2]. Many researchers studied the pozzolanic activity of bagasse ash and concluded that it does not act like a reactive pozzolana [3-5]. This is mainly due to the presence of unburned carbon, crystalline silica as well as the variable and coarse particle size distribution which arises from the high burning temperatures and incomplete combustion conditions of bagasse ash [5-8]. The pozzolanic activity of bagasse ash also depends significantly on its fineness, rather than the chemical composition [9 and 10]. Calcination of bagasse ash under controlled conditions transforms the silica content of the ash into amorphous phase, which has high pozzolanic properties [11]. The pozzolanic reactivity of bagasse ash was found to vary substantially with the calcination temperature [12]. Hence, there are contradictory reports in literature about

the pozzolanic effectiveness of calcined bagasse ash, possibly due to the use of different calcination temperatures [4, 5, 11 and 13]. Bagasse ash is considered as an important by-product for application in construction materials, due to its high amorphous silica content [3 and 6]. Bagasse ash used as an additive for production of building bricks [14] and as a cement replacement up to 20 wt% [3 and 15]. Bagasse ash improves the mechanical properties of cement mortar [16], normal concrete [17], self compacting concrete [18] as well as conventional hollow concrete [19]. Bagasse ash improves the durability of concrete under aggressive environment [11]. The aim of this paper is to investigate the effect of calcination process on the mineralogical composition, microstructure pozzolanic activity of bagasse ash. As well as, to evaluate the hydration characterization of 5-20 wt % calcined bagasse ash blended cement pastes.

II. MATERIALS AND EXPERIMENTAL TECHNIQUES

The raw materials used in this work are OPC [CEM I (No.42.5)] and sugar cane bagasse ash. OPC purchased from Beni-Suif Cement Co., Egypt. The sugar cane bagasse ash sample used in this study was collected from the boiler of nearby sugar factory in the Minia governorate, Egypt. Calcination of bagasse ash carried out in an electrical muffle furnace with a heating rate 10 °C/min at 700-1000°C for 3 hours then recharged from the muffle furnace and cooled to room temperature in desiccator. Calcined bagasse ash was ground to pass 90 µm sieve. The pozzolanic activity of calcined bagasse ash was evaluated by using the method which followed the material-saturated lime solution reaction over time. The test is carried out by putting 1 g of calcined bagasse ash in contact with 75 ml of saturated lime solution in an individual double-cap polyethylene bottles of 100 ml capacity. Samples then cured in an electric oven at 40°C up to 28 days (two bottles per period). At the end of each period, the solution was filtered and the determination of fixed lime content (mmole/l) was quantified according the following procedure. 10 ml of filtered solution added to 20 ml of EDTA (0.0178 mol/l) and calcein indicator. The fixed lime content (mmole/l) was obtained as the difference between the concentration of lime in the saturated lime solution and the lime found in the solution in contact with the sample, at the end of the given period [20].

The pozzolanic activity test residue was dried in microwave oven for 5 minutes to stop the hydration. The samples were finely ground to pass a 90 µm sieve and kept

quickly in sealed plastic bags (to avoid sample carbonation during drying) and stored for XRD, TGA, FTIR and SEM analyses. According to the following results, bagasse ash was calcined at 700°C for 3 hours and used for preparation of blended cement. Six mixes prepared by replacement of OPC with 10-25 % calcined bagasse ash. The mix composition of blended cements is shown in Table 1. The water of normal consistency and the initial as well as final setting times were determined using Vicat apparatus according to ASTM Designations C 187-98 and C 191-01a respectively [21 and 22]. Mixing of dry cement mixes was carried out using the required water of standard consistency. Moulding of the cement pastes was carried out in stainless steel (2×2×2 cm) mould. Immediately after moulding, blended cement pastes cured at about 100 % relative humidity, demolded after 24h and cured up to 90 days in water. Bulk density was determined using Archimedes principle [23]. The compressive strength measurement was carried out using a manual compressive strength machine using set of three cubes according to ASTM Designation C 109-80 [24]. Determination of free water content is carried out by microwave technique using domestic microwave oven (Olympic electric model KOR-131G, 2450 MHz, 1000 W) [25]. The determination of combined water content (W_n) was carried out using samples of hydration stopped specimen after ignition in porcelain crucibles at 1000°C for 1 hour in a muffle furnace and cooling in a desiccator. The total porosity of the hardened cement paste is calculated according to [26].

The X-ray diffraction (XRD) analysis carried out with the aid of Philips x-ray diffractometer PW 1370, Co. using Ni filtered CuK_α radiation (1.5406 Å) under 30 kV operating voltage and 24 mA emission current. The step-scan covered the angular range 15-60° (2θ). The Mineralogical composition of the investigated samples was identified by the aid of the PDF standards. The X-ray fluorescence (XRF) analysis carried out by the same apparatus. The thermogravimetric analysis (TGA) carried out with the aid of Shimadzu Corporation thermo analyzer with DTG-60H detector. About 5 mg of finely powdered sample put in an aluminium cell and heated with 10 °C/min heating rate, under nitrogen atmosphere at 40 ml/min flow rate, the hold time at the appropriate temperature is zero.

TABLE 1. MIX COMPOSITION IN WT % OF BAGASSE ASH BLENDED

Symbol	OPC	Bagasse ash
C	100	0
B1	90	10
B2	85	15
B3	80	20
B4	70	25

The Fourier transform infrared (FTIR) spectra measured by the spectrometer Perkin Elmer FTIR system Spectrum X. Samples prepared by mixing 1 mg of the sample in 200 mg of KBr. The spectral analysis performed in the range 400-4000 cm⁻¹ with spectral resolution of 1 cm⁻¹. Scanning electron microscopy (SEM) investigated by the aid of Jeol-Dsm 5400 LG apparatus to examine the morphology and microstructure of selected samples by mounting on stubs and coating with gold prior to analysis to make them electrically conductive.

III. RESULTS AND DISCUSSION

A. Characterization of raw and calcined bagasse ash

Table 2 shows the chemical composition of the OPC and bagasse ash calcined at 900°C for 3 hrs determined by XRF analysis. The chemical composition of bagasse ash illustrates that bagasse ash contains high amount of SiO₂ (75.23 wt %) in addition to small amounts of CaO, Fe₂O₃, Al₂O₃, MgO, Na₂O and K₂O. The sum of SiO₂, Al₂O₃ and Fe₂O₃ equals to 85.89 wt % which is in good agreement with the requirements (>70 wt %) stated in ASTM designation: C 618-89 for natural pozzolana [27]. The phase composition of OPC, wt % according to Bouge's calculations are; 47.78% C₃S, 23.83% β-C₂S, 10.73% C₃A, 9.68% C₄AF and 0.914% lime saturation factor (LSF).

TABLE 2. CHEMICAL COMPOSITION WT % OF OPC AND BAGASSE ASH

Oxide	OPC, wt %	Bagasse ash, wt %
SiO ₂	20.88	50.69
Al ₂ O ₃	6.08	13.56
CaO	63.00	10.57
Fe ₂ O ₃	3.18	11.78
MgO	1.50	4.24
SO ₃	1.60	0.04
Na ₂ O	0.22	3.06
K ₂ O	0.24	0.49
Cl	0.11	0.18
LOI	2.35	4.41
Total	99.16	99.02

Fig. 1 illustrates the XRD patterns of bagasse ash as well as bagasse ash calcined at 700-1000°C. Bagasse ash contains amorphous silica as indicated from the heap in the range 15-35 2θ in addition to quartz, tridymite and christobalite. Bagasse ash also contains residual amounts of calcium carbonate, calcium silicate and calcium aluminosilicate minerals which appear in form of calcite, kilchoanite and gehlenite minerals respectively. Calcination of bagasse ash at 700°C combusts unburned carbon completely as indicated from the disappearance of heap in the range 15-35 2θ. Increasing the peak intensity of quartz may be attributed to that calcination also enhances the transformation of amorphous silica into quartz. Raising the calcination temperature of bagasse ash up to 900°C enhances the transformation of quartz into tridymite as shown from increasing its peak intensity. Calcite also, decomposes into portlandite (Ca(OH)₂) with raising the calcination temperature.

Fig. 2 illustrates The TGA curve of bagasse ash. The weight loss which occurs in the temperature range 25-125°C may be due to the loss of moisture. The main weight loss (about 31%) which occurs in the temperature range 400-800°C could be due to unburned carbon. This is within the range of loss on ignition values of 16-33 wt% reported in literature for bagasse ash [28 and 29]. Raw bagasse ash also has a weight loss in the temperature range 850-1000°C could be due to the decomposition of residual calcite which originally present in bagasse ash.

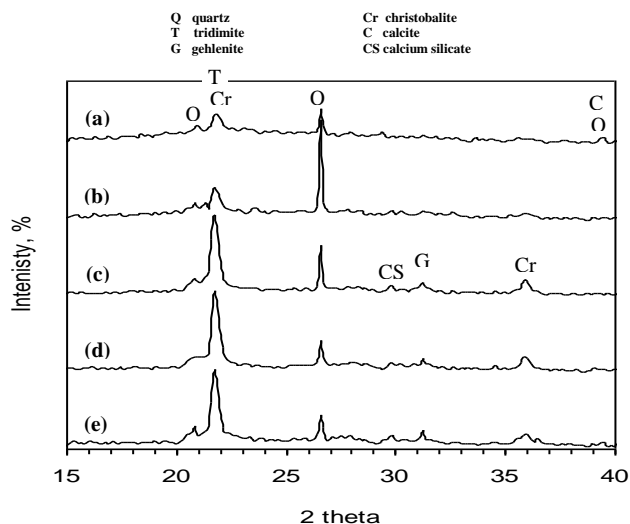


Fig. 1. XRD patterns of (a) raw bagasse ash as well as bagasse ash calcined for 3 hrs at (b) 700°C, (c) 800°C, (d) 900°C and (e) 1000°C

Fig. 3 illustrates the FTIR spectra of bagasse ash as well as bagasse ash calcined at 800°C. The most significant absorption bands of silica appear at 1101, 796 and 467 cm^{-1} which are attributed to asymmetric stretching vibration of Si-O-Si, symmetric stretching vibration of Si-O-Si and bending vibration of O-Si-O respectively [30]. The carbonate absorption band that appears around 1429 cm^{-1} attributed to asymmetric stretching vibration of carbonate (ν_3 of CO_3^{2-}). The intensities of the absorption bands of silica increase with calcination of bagasse ash. This is an indication of increasing of the degree of polymerization of silica network due to crystallization of amorphous silica and transformation of quartz into tridymite [31].

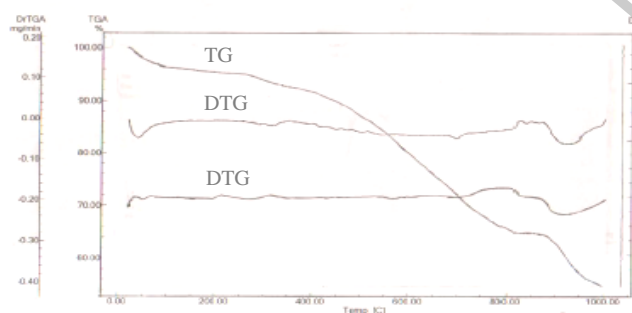


Fig. 2. TGA curve of raw bagasse ash

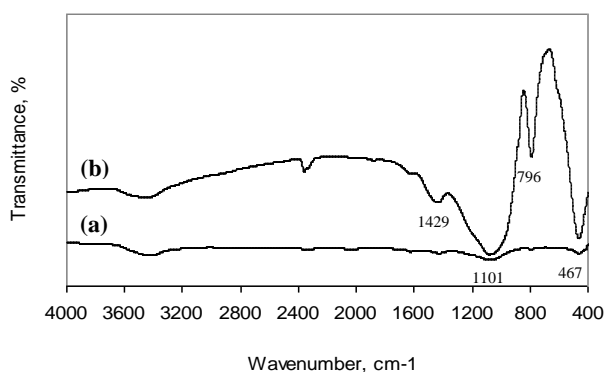


Fig. 3. FTIR spectra of (a) raw bagasse ash as well as (b) bagasse ash calcined at 800°C

Fig. 4 illustrates the SEM micrograph of raw bagasse ash as well as bagasse ash calcined at 700 and 800°C. Unburned carbon appears as black flaks (U) while silica grains appear as whitish peaces (S) in case of raw bagasse ash. Unburned carbon combusted and disappeared while silica aggregate concentrated in case of bagasse ash calcined at 700°C. Calcination temperature of bagasse ash at 800°C affects the morphology of silica grains i.e. sintering of silica.

B- Pozzolanic activity of calcined bagasse ash

Fig. 5 illustrates the fixed lime content (mmole/l) of bagasse ash calcined at 700-1000°C up to 28 days. The fixed lime content of all calcined bagasse ash samples increases with curing time due to the progress of pozzolanic reaction between active silica present in calcined bagasse ash and saturated lime solution, hence, the amount of pozzolanic reaction products and fixed lime content increase with curing time. The fixed lime content of calcined bagasse ash samples decrease with increasing the calcination temperature. Increasing the calcination temperature lead to the conversion of quartz into tridymite which have lower specific gravity than quartz because the atoms are less closely packed in tridymite than in quartz [32], hence, it may be has a higher chemical stability, i.e. lower pozzolanic activity towards saturated lime solution than quartz.

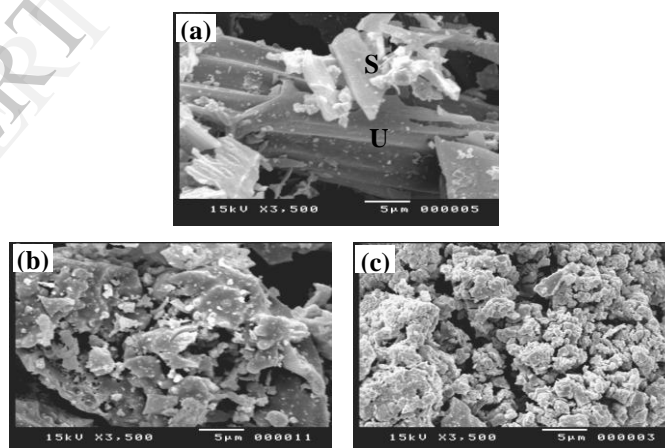


Fig. 4. SEM micrograph of (a) raw bagasse ash as well as bagasse ash calcined at (b) 700°C and (c) 800°C

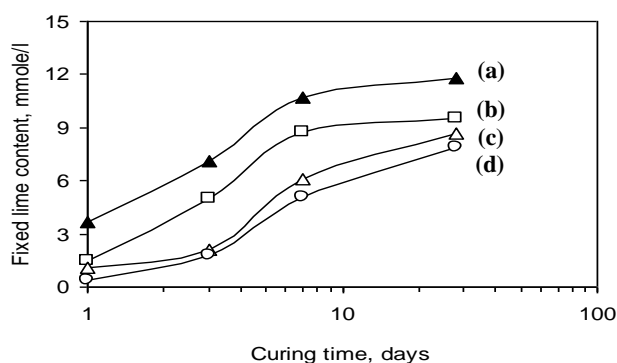


Fig. 5. Fixed lime content (mmole/l) of bagasse ash calcined at (a) 700°C, (b) 800°C, (c) 900°C and (d) 1000°C up to 28 days

Fig. 6 illustrates the XRD patterns of the 28 days pozzolanic activity test residues of bagasse ash calcined at 700-1000°C. It was observed that peak intensity of quartz and CSH decrease whereas those of tridymite and christobalite increase with increasing calcination temperature of bagasse ash. This is due to that quartz transforms into tridymite with increasing calcination temperature after 870°C. Quartz reacts with saturated lime solution to form CSH while tridymite is less reactive than quartz in the pozzolanic activity towards saturated lime solution.

Table 3 illustrates the weight loss, in mg units for CSH and CH phases derived from the TGA of 28 days pozzolanic activity test residue of bagasse ash calcined at 700, 800 and 1000°C shown in Fig. 7. It was observed that bagasse ash sample calcined at 700°C have the higher weight loss values in the temperature range 125-275°C corresponding to the decomposition of CSH. This is an indication for its pozzolanic activity. Also, bagasse ash sample calcined at 700°C have the higher weight loss values in the temperature range 450-550°C corresponding to the decomposition of CH [33]. This is an indication for its higher adsorption capacity to saturated lime solution due to presence of amorphous silica. Calcination of bagasse ash at higher temperature enhances the crystallization of silica and reduces the pozzolanic activity as well as the adsorption capacity of calcined bagasse ash.

TABLE 3. WEIGHT LOSS, MG FOR CSH AND CH PHASES DERIVED FROM THE TGA OF 28 DAYS POZZOLANIC ACTIVITY TEST RESIDUE OF BAGASSE ASH CALCINED AT 700, 800 AND 1000°C SHOWN IN FIGURE (10).

Phases	Weight loss, mg	
	CSH	CH
Sample	Temperature range	
	125-275°C	450-550°C
700°C	-0.071	-0.038
800°C	-0.043	-0.020

Q quartz
T tridymite
G gehlenite
H CSH
Cr christobalite
CS calcium silicate
P portlandite

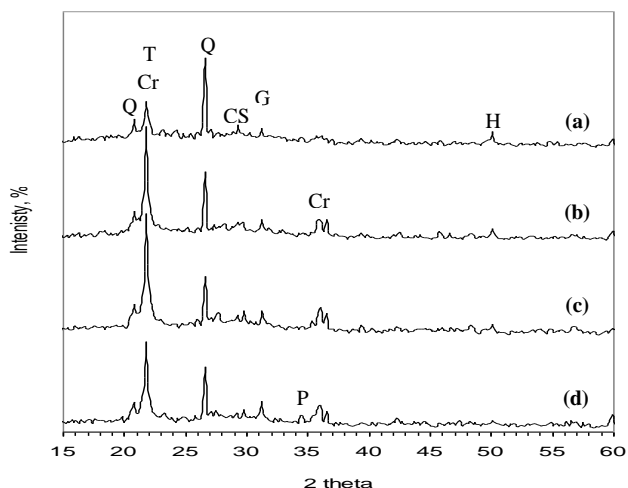


Fig. 6. XRD patterns of the 28 days pozzolanic activity test residues of bagasse ash calcined at (a) 700°C, (b) 800°C, (c) 900°C and (d) 1000°C

Fig. 8 illustrates the FTIR spectra of 28 days pozzolanic activity test residue of bagasse ash calcined at 700, 800 and 1000°C. The spectra show the absorption bands of silica appear at 1101, 796 and 467 cm^{-1} as well as the absorption bands of carbonate appear around 1429 cm^{-1} . The presence of CSH is indicated from its characteristic absorption band centered at 976 cm^{-1} (ν_3 of Si-O) [34 and 35]. The decrease of intensities of absorption bands of silica is an indication of lowering the degree of polymerization of silica network due to formation of hydration products (C-S-H) because of the pozzolanic activity of bagasse ash [31]. On contrast, the intensities of the absorption bands of silica increase with calcination temperature. This may arise from transformation of quartz into tridymite and decreasing the pozzolanic activity of bagasse ash with calcination temperature.

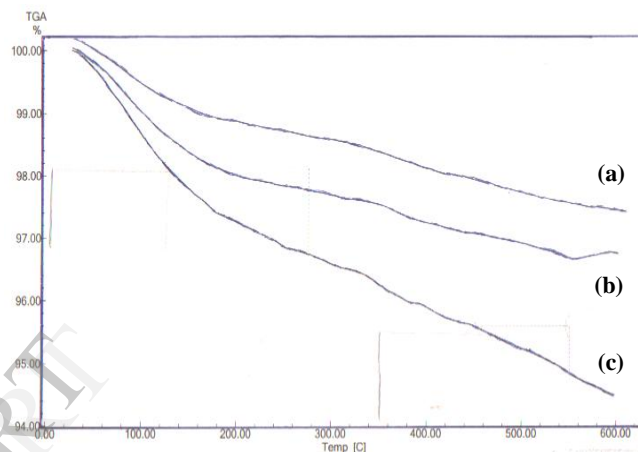


Fig. 7. TGA curves of 28 days pozzolanic activity test residue of bagasse ash calcined at 700, 800 and 1000°C

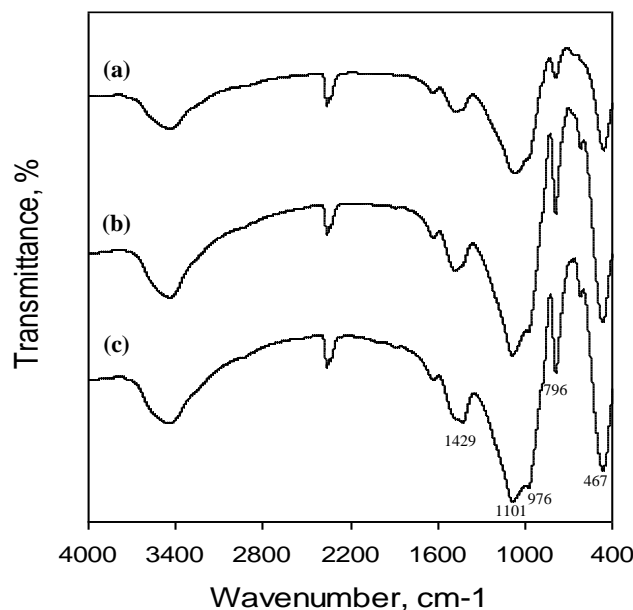


Fig. 8. FTIR spectra of 28 days pozzolanic activity test residue of bagasse ash calcined at (a) 700, (b) 800 and (c) 1000°C

Fig. 9 illustrates the SEM micrograph of 28 days pozzolanic activity residue of bagasse ash calcined at 700-1000°C. It was clearly shown that sample calcined at 700°C contains rough silica grain surfaces impregnated with much of the dense calcium silicate hydrates web structure. The amount of calcium silicate hydrates markedly diminish at samples calcined at 800 and 900°C. this indicates the lowering of pozzolanic activity of bagasse ash with calcination beyond 700°C. Bagasse ash sample calcined at 1000°C show vitrified cracked silica grain surfaces with very small amount of calcium silicate hydrates.

C- Hydration characteristics of blended cement pastes

Fig. 10 illustrates the water of consistency, initial and final setting times of calcined bagasse ash blended cement pastes. The water of consistency increases linearly with bagasse ash content may be due to that bagasse ash markedly increases the amount of surface water which related to the specific surface of the system [36]. The setting time elongates with calcined bagasse ash content may be due to increasing the amount of mixing water [37] as well as decreasing clinker content which has shorter setting time [38]. The elongation of setting time also may be due to the formation of a layer of calcined bagasse ash particles around anhydrous cement grains which delays the hydration of C₃S. The thickness of this layer increases with calcined bagasse ash content hence the rate of hydration of cement decreases [39].

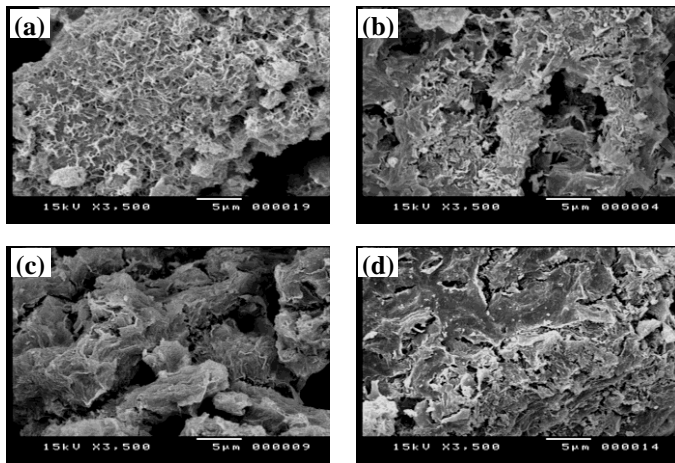


Fig. 9. SEM micrograph of 28 days pozzolanic activity residue of bagasse ash calcined at (a) 700, (b) 800, (c) 900 and (d) 1000°C

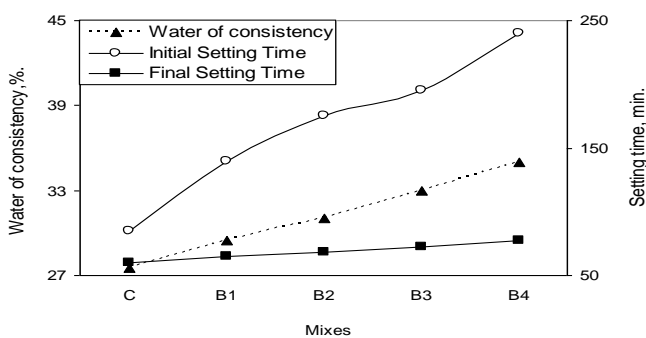


Fig. 10. The water of consistency, initial and final setting times of calcined bagasse ash blended cement pastes

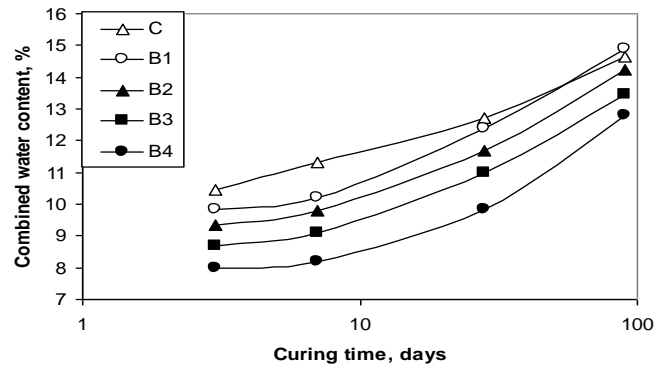


Fig. 11. The combined water content of bagasse ash blended cement pastes hydrated up to 90 days

Fig. 11 represents the combined water content of bagasse ash blended cement pastes hydrated up to 90 days. The combined water content decreases with bagasse ash content may be due to that bagasse ash at lower substitution level acts as a nucleating agent for hydration products enhancing the cement hydration while increasing bagasse ash content make a dilution effect.

Fig. 12 represents the bulk density and total porosity of bagasse ash blended cement pastes hydrated up to 90 days. The bulk density decreases while the total porosity increases with bagasse ash content due to the dilution effect of bagasse ash by reducing the amount of OPC, hence the amount of hydration product reduces and as a result the bulk density decreases and the total porosity increases. The bulk density decreases with bagasse ash content may also due to that bagasse ash have lower specific gravity than OPC. Fig. 13 shows the compressive strength of bagasse ash blended cement pastes hydrated up to 90 days. Blended cement pastes have low compressive strength at early ages of hydration then the rate of strength development enhanced at later ages. Bagasse ash at the early ages of hydration acts as a filler then at later ages of hydration it acts as pozzolana and reacts with portlandite CH (accumulated in the pore of hydrated cement pastes because of hydration of clinker phases) giving additional calcium silicate hydrates.

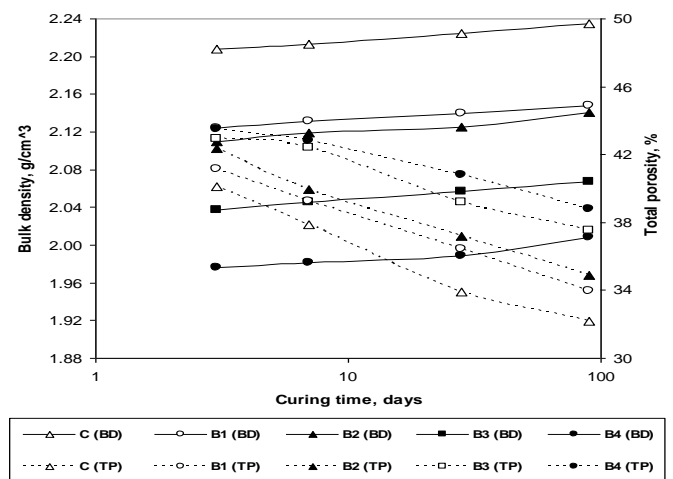


Fig. 12. The bulk density and total porosity of bagasse ash blended cement pastes hydrated up to 90 days

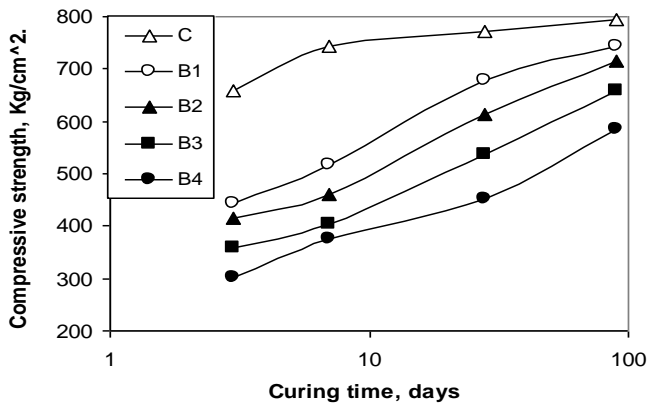


Fig. 13. The compressive strength of bagasse ash blended cement pastes hydrated up to 90 days

The compressive strength decreases with bagasse ash content because of the dilution of hydrated cement pastes by decreasing the OPC content. Fig. 14 illustrates the FTIR patterns of bagasse ash blended cements hydrated for 90 days. The absorption bands of carbonate increase with bagasse ash content may be attributed to that total porosity of hydrated cement pastes increase with bagasse ash content, which in turn increases the rate of carbonation of hydrated cement pastes. The absorption band of C-S-H at 978 cm^{-1} (stretching vibration ν_3 of SiO_4^{4-}) shifts to higher wavenumber values with increasing bagasse ash content. This may be due to that C-S-H formed by the pozzolanic action of bagasse ash differ (in its nature and water content) from that formed by hydration of OPC. The absorption band of silica appears at 462 cm^{-1} corresponding to bending vibration of O-Si-O arises from the replacement of OPC with bagasse ash. The ettringite band at 1124 cm^{-1} disappeared due to that SO_4^{2-} ions were probably replaced by CO_3^{2-} ions in case of bagasse ash blended cement pastes. The absorption band observed at 3640 cm^{-1} which is due to O-H stretching of $\text{Ca}(\text{OH})_2$ increases at high bagasse ash content. This may be attributed to that bagasse ash have a low pozzolanic activity because higher amount of portlandite accumulated although the dilution effect of bagasse ash.

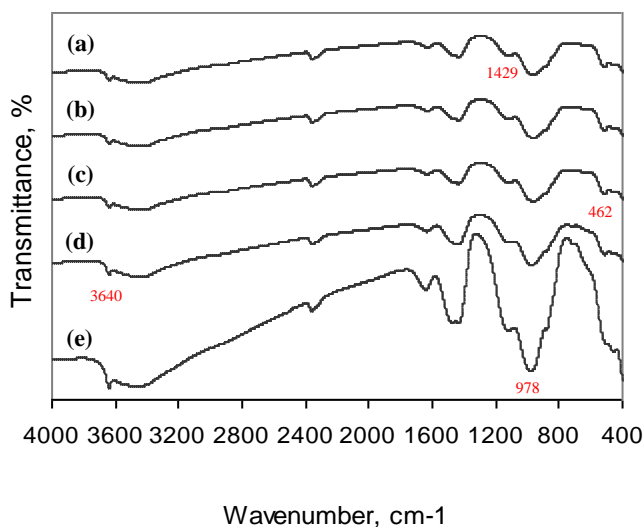


Fig. 14. The FTIR patterns of (a) OPC as well as bagasse ash blended cements (b) B1, (c) B2, (d) B3 and (e) B4 hydrated for 90 days

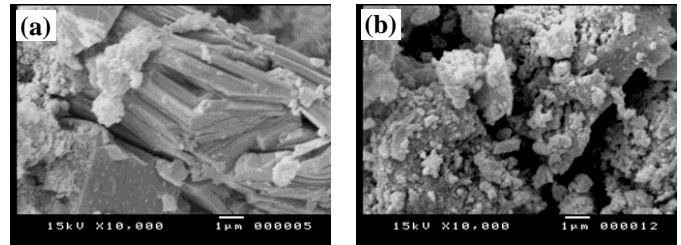


Fig. 15. SEM micrographs of (a) OPC and (b) B2 hydrated for 28 days

Fig. 15 shows the SEM micrographs of OPC and blended cement paste containing 15 % bagasse ash hydrated at 28 days. The SEM results illustrate the formation of hydration products C-S-H and CH, which fill some pores of hydrated OPC paste. In case of bagasse ash blended cement paste, excess bagasse ash forms a coating layer around anhydrous cement grains, hence, the degree of hydration and formation of hydration products decrease and as a result, the total porosity of bagasse ash blended cement paste increases.

CONCLUSIONS

The main conclusions of this investigation are:

- 1- Raw bagasse ash contains unburned carbon as well as amorphous silica, crystalline silica in form of quartz, tridymite and cristobalite. Hence, it does not act like a reactive pozzolana.
- 2- To be utilized as a supplementary cementitious material, bagasse ash must be calcined under controlled conditions to removing unburned carbon, preserve the amorphous silica and improve its pozzolanic properties.
- 3- Calcination of raw bagasse ash at high temperature enhances the crystallization of amorphous silica and transformation of quartz into tridymite. Hence, the pozzolanic activity of calcined bagasse ash samples decreases with calcination temperature.
- 4- It can be recommended that calcination of bagasse ash at 700°C up to 3 hrs is the optimum conditions required to produce calcined bagasse ash with high pozzolanic activity to be utilized as a supplementary cementitious material.
- 5- At the early ages of hydration bagasse ash acts as a filler whereas at later ages it acts as pozzolana. Increasing bagasse ash content make a dilution effect, require higher water demands and forms a layer of bagasse ash particles around anhydrous cement grains which delays the hydration of cement.

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