ISSN 2229-5518

SEM-EDS Analysis of Portland Cement and Sugarcane Bagasse Ash Collected from Different Boilers of Sugar Industry

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Abstract— When sugar cane is crushed in the milling plants of the sugar factories for extracting its juice, the fibrous residue left over after the extraction of juice is known as bagasse. Bagasse is a mixture of hard fibre, with soft and smooth parenchymatous (pith) tissue, having hygroscopicity, soil, wax, sugar etc. When bagasse is used as fuel in the combustion boiler of cogeneration plant under controlled burning, a reactive amorphous silica, known as sugarcane bagasse ash (SBA) is formed. In the present study, micro structural observations and elemental analysis of Portland cement (OPC) and sugarcane bagasse ashes collected from four different sugar factories are carried out. Out of these four factories, three are located in the region of north Karnataka state (Chikodi, Vijayapur, M.K.Hubli) and one in Maharashtra state (Ichalakaranji) are studied. These ashes are represented as BA-C (Chikodi), BA-V (Vijayapur), BA-M (M.K.Hubli) and BA-I (Ichalakaranji). The microstructural observations were made using Scanning Electron Microscope (SEM) and elemental compositions were analyzed by Energy dispersive spectroscopy (EDS). As indicated by SEM results, the OPC and all the sugarcane bagasse ashes presents different microstructure. On the other hand EDS map shows calcium as predominate element in OPC and silicium in all the bagasse ashes.
Index Terms— Sugarcane bagasse ash, microstructure, elemental analysis, Portland cement, SEM-EDS, Temperature

1 INTRODUCTION

In recent years, the research related to agricultural wastes is intensifying with the aim of evaluating their potential for recycling as well as the elimination of the landfills. In this research li ne, the works are focused mainly on the sugar cane wastes. Sugarcane is the most profitable crop as compared to the crops like wheat, paddy and cotton. This has led to expansion of sugarcane in Maharashtra and Karnataka despite these states being water stressed. When sugar cane is crushed in the milling plant of sugar factories for extracting juice, the fibrous residue left-over after the extraction of juice is known as bagasse and in some countries is known as migasse.

The term bagasse is derived from the French and Spanish word "bagazo" given to the husks of grapes or olives (waste materials) left over after extracting their contents by pressing [1]. All industry like sugar, pulp and paper, fertilizers, textiles etc. require large quantities of electric power and economics of cogeneration depends on the availability of cheap source of fuel. All cogeneration activities in sugar factories have evolved methods of using bagasse more effectively to generate steam and consequently, electric power more efficiently and economically. Bagasse, being a sulphur free fuel (unlike coal, petroleum fuels which contain lot of sulphur) its usage as fuel is an environmental friendly technology. The capital cost of power generation in sugar factories is much lower than power generation in conventional power stations.

Sugarcane bagasse has an intricate structure, and is basically made out of 25% lignin, 25% hemicellulose and 40–50% cellulose [2]. Sugarcane bagasse is utilized as fuel in the cogeneration procedure to create steam and power in sugar businesses. At the point when bagasse is singed in ignition evaporator under controlled burning, amorphous silica is formed in the remaining ash. In the wake of consuming, bagasse fiery remains is gathered as a result from cogeneration evaporator [3]. Quantity of ash generated is of the order of 0.3% of the weight of sugarcane crushed in the factory i.e. if 500000 tonnes of sugarcane is crushed in a season, the total ash produced is of the order of 1500 tonnes [1].Table 1 presents state wise number of factories operation in India.

Table 1: State wise number of factories operation in India

States	2010-2011	2011-2012	2012-2013
Andhra Pradesh	37	37	36
Bihar	10	11	11
Chhattisgarh	3	3	3
Gujarat	19	19	18
Haryana	14	14	14
Karnataka	59	58	60
Goa	1	1	1
M.P	13	13	12
Maharashtra	167	170	172
Punjab	16	17	16
Rajasthan	1	1	1
T.N and Pondicherry	46	45	45
U.P Central	49	49	49
U.P East	42	42	40
U.P West	34	33	33
Uttarakhand	10	10	9
W.B	6	6	6
All India	527	529	526
Duration of crushing season (days)			
All India	136	137	126
Production of sugar (Lakh Tonnes)			
All India	243.94	263.42	251.41

ISSN 2229-5518

Sugarcane bagasse ash (SCBA) is created in huge amounts (67,000 tons/day) in India, on account of the widely created sugar industry. Transfer of bagasse ash remains is a basic issue for sugar ventures in light of ecological limitations and land prerequisite. Fast execution of new cogeneration plants and development of cogeneration limit of existing plants in sugar businesses are additionally anticipated that would build bagasse fiery debris age altogether in the significant sugar producing nations [3]. Moises Frias et.al.(2011) reported that morphology of ashes directly depends on the process and burning temperature. EDX analysis shows that the coarse particles present in ashes are formed by silica which corresponds to the quartz particles. The fine bagasse ash forms prismatic particles with high contents of Si, Al, Ti and Fe [4]. Baharudeen et.al. (2014) utilized sugarcane bagasse as a pozzolonic material and studied its compatibility with super plasticizers. It was observed that as bagasse ash replacement increases in cement the water content also increases in cement paste. This is due to the irregular structures of fine burnt particles observed in the raw bagasse ash. Cardeiro et.al. (2009) studied effect of calcination temperature on the pozzolonic activity of SBA. The results shows that calcination temperature is an important parameter for the production of SBA with pozzolonic activity. The SBA produced with air calcination at 600°C for 3 hrs presents amorphous silica and low carbon content. EDS analysis of the SBA burnt at 600°C presents C=11.71%, O=51.41%, Na=0.5%, Si=19.71%, P=0.4%, K=2.6% by mass.

In previous research studies, many researchers have reported that the microstructure and chemical composition of sugarcane bagasse ash depends on burning temperature of sugarcane bagasse in the boilers of sugar factories, soil where sugarcane is grown, variety of sugarcane, inputs (fertilizer and irrigation water) applied to the sugar cane crop etc. Scanning electron microscopy (SEM) and Energy dispersive spectroscopy (EDS) was used in the analysis of microstructure and chemical composition of Ordinary Portland Cement (OPC) and sugarcane bagasse ash (SBA). In the current research, microstructure and elemental characterization of highly reactive sugarcane bagasse ash obtained from the different cogeneration boilers located in different regions of the Karnataka and Maharashtra state, India are studied. The results obtained are compared with microstructure and elemental composition of Ordinary Portland Cement.

2.2 Sugarcane Bagasse Ash (SBA)

Sugarcane bagasse ash used in the present study was collected from different cogeneration boilers located in different regions of the Karnataka and Maharashtra state. Three factories are located in the northern region of Karnataka state (Chikodi, Vijayapur, M.K.Hubli) and one in Maharashtra state (Ichalakaranji). These 4 ashes are symbolized as BA-C, BA-V, BA-M and BA-I respectively.

2. Materials

2.1 Ordinary Portland cement (OPC)

OPC of 43 grade was used to study the microstructure and elemental composition under SEM images coupled with EDS analyzer.

3. Methodology

Scanning electron microscopy (SEM) and Energy dispersive spectroscopy (EDS) was used in the analysis of microstructure and chemical composition of OPC and SBA. SEM images were taken on the OPC and SBA to examine the microstructure properties. Before SEM and microanalysis, the samples were coated with a layer of carbon in argon gas atmosphere at a high vaccum of 5.0X10-6 Torrs, in order to make them electrically conductive in nature. SEM (JSM-IT300) accompanied by an EDS analyzer, supplied by JEOL Ltd. was used for the image processing. The JSM-IT300 provides a large specimen chamber which can accommodate specimens containing multiple samples at a time.

EDS analysis was also carried out to determine the elemental composition of OPC and SBA. EDS spectra were acquired with the help of INCA systems software that is used by the Oxford spectrometer. The working distance was maintained at 13mm and the probe currents (64.8µA-67µA) in order for the EDS analyzer to work properly.

4. Results and discussion

For identification and characterization of mineral phases and morphology of OPC and SBA particles, SEM-EDS analysis is the best and most widely used method. OPC and SBA (BA-C, BA-V, BA-M and BA-I) samples were analyzed to obtain the microstructural observation. The samples were also analyzed by EDS to study the chemical composition. A typical SEM image of OPC is presented in Fig.1(a). Microstructural examination of OPC did not indicate the presence of ettringite but OPC particles shows angular and irregular. EDS analysis of selected area (see rectangle in Fig. 1(a)) shows that the OPC particles contains predominately calcium, oxygen, silicium and trace amounts of aluminium, iron, potassium, sodium as shown in Fig. 1(b) and elemental composition is presented in Table 2. The elemental composition of OPC is reported in Table 2 presents Ca as major element due to the utilization of limestone (contains CaO) as a raw material during manufacturing of cement.

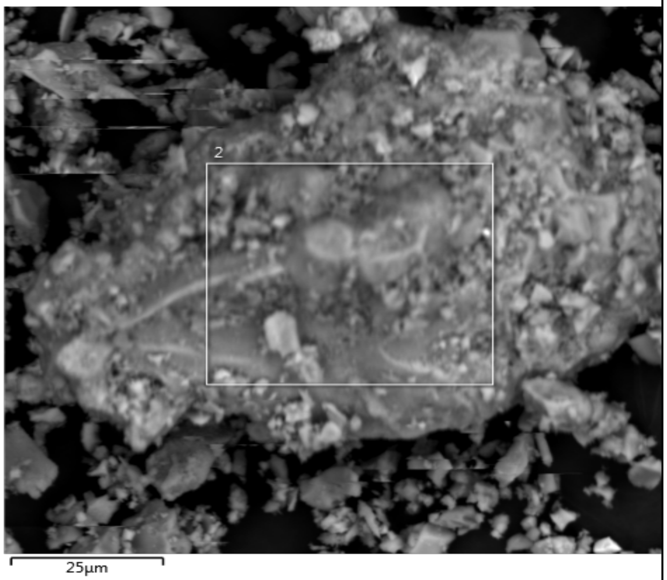

Fig. 1(a): SEM image of OPC

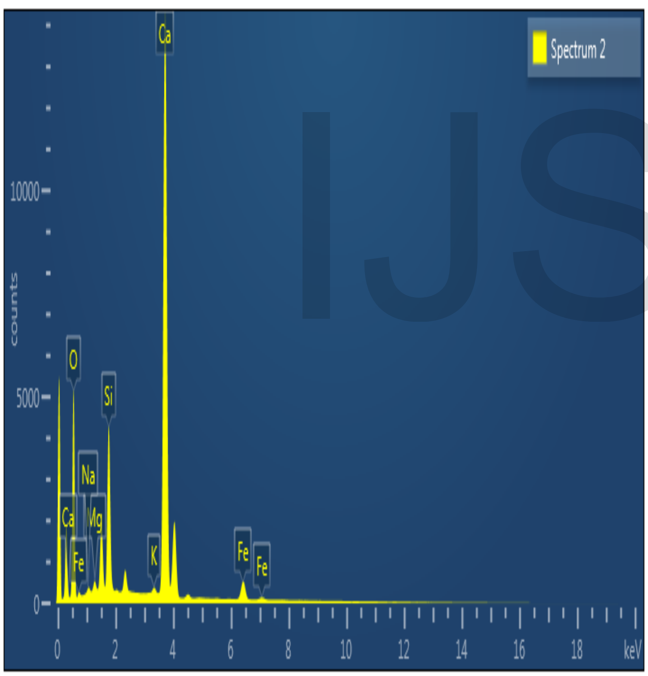

Fig. 1(b): EDS analysis of OPC

Table 2: Elemental composition of OPC

Element	*OPC*	
	Weight (%)	*Atomic (%)*
O	*34.65*	*55.23*
Si	*7.92*	*7.19*
S	*1.25*	*0.99*
Ca	*46.70*	*29.71*
Fe	*4.32*	*1.97*
Al	*3.61*	*3.42*
K	*0.41*	*0.27*
Mg	*0.62*	*0.65*
Na	*0.51*	*0.57*

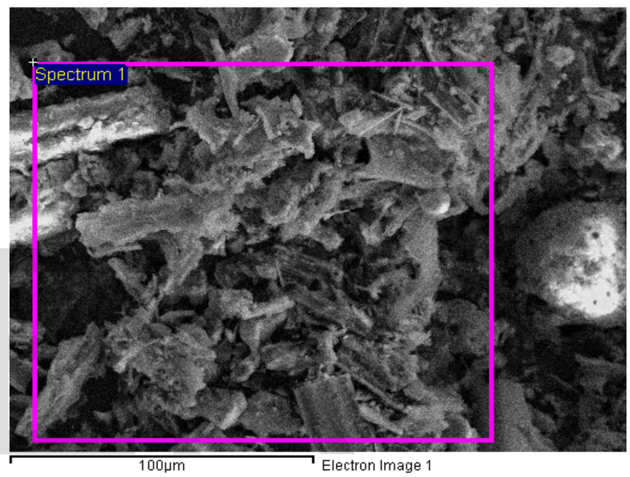

Fig. 2(a): SEM image of BA-C

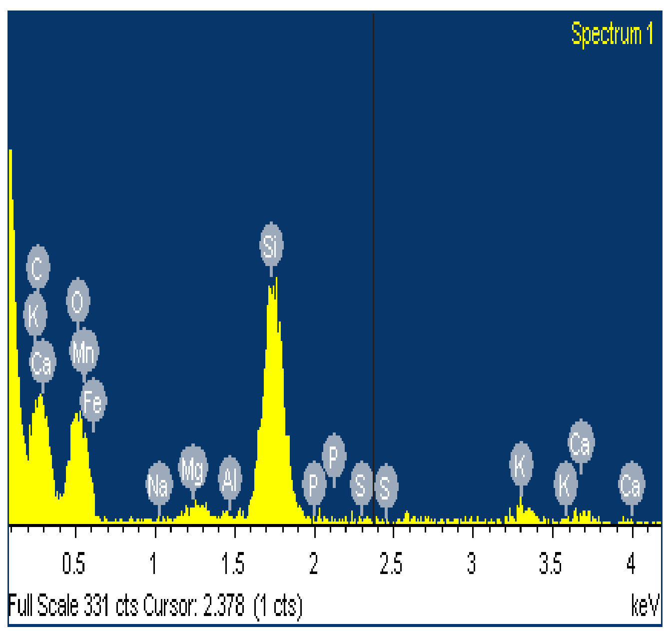

Fig. 2(b): EDS analysis of BA-C

The morphology of sugarcane bagasse ash collected from the Chikodi region (BA-C) is as shown in Fig. 2(a). BA-C presents particles of varied shapes and sizes, less dense as compared to OPC. SEM image of BA-C shows crystalline and prismatic particles with well defined edges. The crystalline microstructure shows that BA-C particles contain predominately Si in the ash. Spherical shaped particles are also identified in the micrograph. The marked rectangle in Fig. 2(a) was selected for EDS analysis. EDS analysis of BA-C presents higher masses of O and Si compared to K, Ca, and Mg. The masses of O, Si and Ca elements were 55.43%, 35.03% and 2.95% respectively and trace elements are Mg and K are observed as shown in Fig. 2(b).The elemental composition of BA-C is presented in Table 3.

Table 3: Elemental composition of BA-C ash

Element	*BA-C*	
	Weight (%)	*Atomic (%)*
C	*0*	*0*
O	*55.43*	*69.46*
Si	*35.03*	*25.01*
S	*0*	*0*
Ca	*2.95*	*1.48*
Fe	*0*	*0*
Al	*0*	*0*
K	*4.40*	*2.26*
Mg	*2.19*	*1.81*
Na	*0*	*0*
P	*0*	*0*
Mn	*0*	*0*

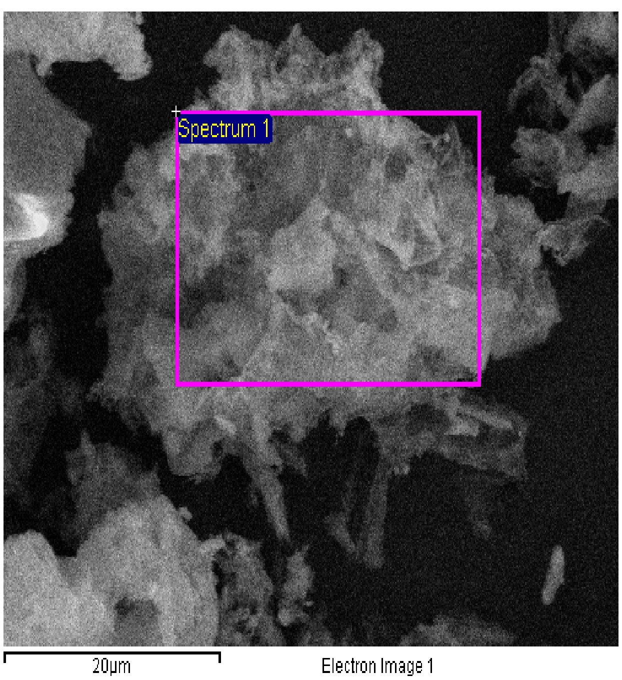

Fig. 3(a): SEM image of BA-V

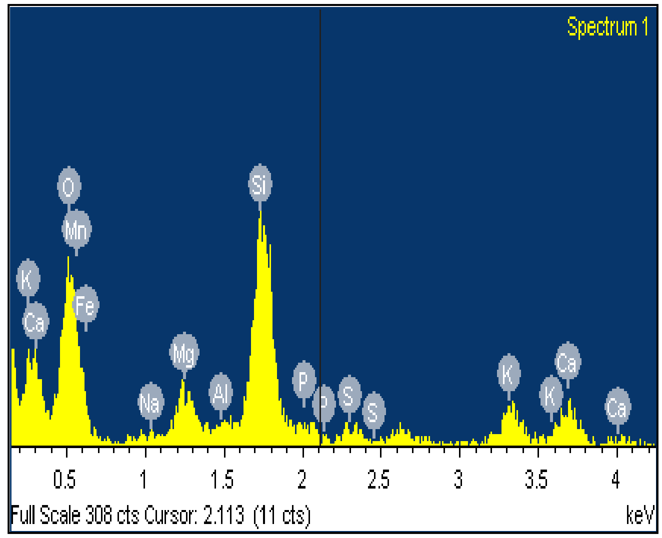

Fig. 3(b): EDS analysis of BA-V

The morphology of sugarcane bagasse ash collected from the Vijayapur region (BA-V) is as shown in Fig. 3(a). The microstructure of BA-V is very different from those observed in OPC and BA-C. BA-V particles indicate denser and less crystalline as compared to OPC and BA-C particles. According to EDS analysis (marked rectangle area in Fig. 3(a)) shows BA-V particles are composed of Oxygen (O), silicium (Si), calcium (Ca) and potassium (K) as major elements and the other trace elements are magnesium (Mg), aluminium (Al), phosphorus (P) and sulphur (S) as shown in Fig. 3(b). The Si concentration in the BA-V is lower than that registered in BA-C, while Ca concentration is higher than the BA-C particles. EDS map shows presence of P and S elements in trace amounts may be possibility due to application of fertilizer to the sugarcane crop and presence of soil particles in the ash. The elemental composition of BA-V is presented in Table 4.

Table 4: Elemental composition of BA-V ash

Element	*BA-V*	
	Weight (%)	*Atomic (%)*
O	*56.55*	*71.22*
Mg	*4.72*	*3.9*
Al	*1.86*	*1.38*
Si	*20.20*	*14.47*
P	*2.60*	*1.69*
S	*2.03*	*1.27*
K	*5.59*	*2.88*
Ca	*6.35*	*3.18*
Mn	*0*	*0*
Fe	*0*	*0*
Na	*0*	*0*

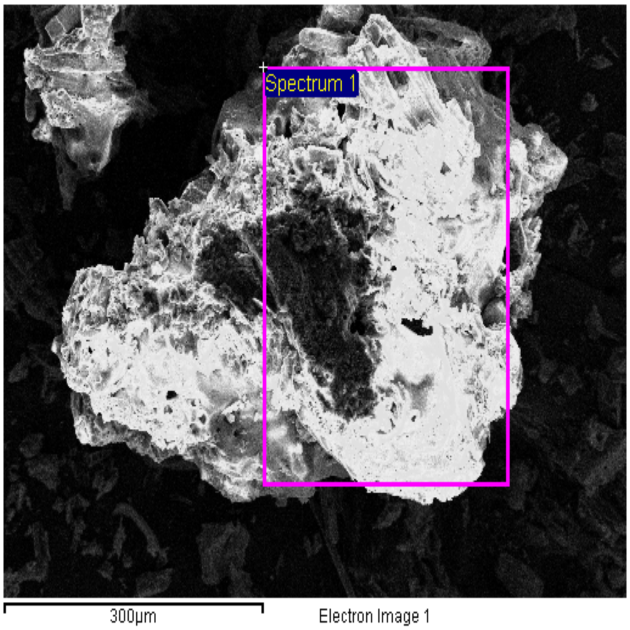

Fig. 4(a): SEM image of BA-M

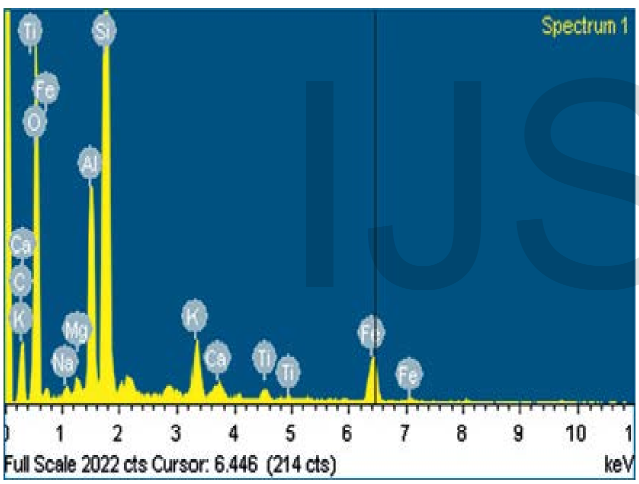

Fig. 4(b): EDS analysis of BA-M

The morphology of sugarcane bagasse ash collected from the M.K.Hubli region (BA-M) is as shown in Fig. 4(a). SEM image of BA-M shows dark in color due to temperature maintained in the boiler varying from 600 to 650□C. The dark color also indicates that the bagasse contains some carbon due to incomplete calcinations of sugarcane bagasse as corroborated by the EDS elemental analysis as shown in Fig. 4(b) According to EDS analysis (marked rectangle area in Fig. 4(a)) shows BA-M particles are composed of O, silicium (Si) and Carbon (C), as major elements and the other trace elements are magnesium (Mg), aluminium (Al), calcium (Ca), potassium (K) and titanium (Ti) as shown in Fig. 4(b). From the previous studies, it has been reported that large quantities of carbon may slow down the pozzolonic activity as they decrease the amount of contact between CH and silica rich grains [5]. The elemental composition of BA-M is presented in Table 5.

Table 5: Elemental composition of BA-M ash

Element	*BA-M*	
	Weight (%)	*Atomic (%)*
O	*56.19*	*62.90*
C	*12.17*	*18.08*
Mg	*1.39*	*1.29*
Al	*4.88*	*3.23*
Si	*16.43*	*10.44*
P	*0*	*0*
S	*0*	*0*
K	*1.92*	*0.88*
Ca	*1.52*	*1.23*
Mn	*0*	*0*
Fe	*4.50*	*1.44*
Na	*0.39*	*0.30*
Ti	*0.61*	*0.23*

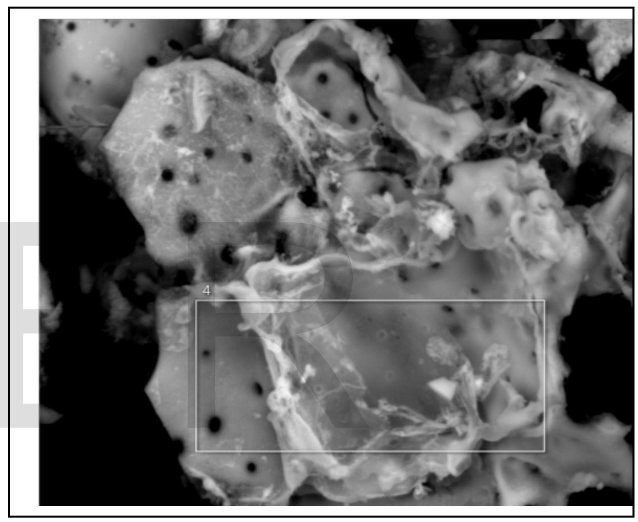

Fig. 5(a) SEM image of BA-I

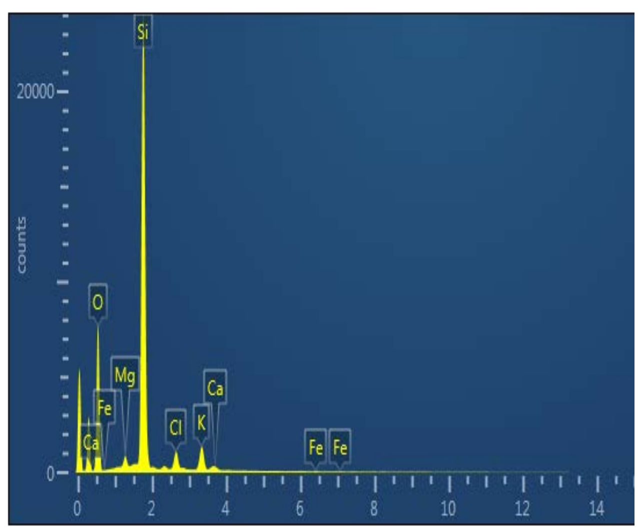

Fig. 5(b) EDS analysis of BA-I

The microstructure of sugarcane bagasse ash collected from the Ichalakaranji region (BA-I) is as shown in Fig. 5(a). SEM image of BA-I shows smooth surface with high porosity i.e spongy in nature and large surface areas. EDS analysis of BA-I (see rectangle in Fig. 5(a)) shows that the selected particles contains predominately silicium and oxygen with lesser amount of potassium, calcium, iron, chloride, phosphorus and magnesium as shown in Fig. 5(b). BA-I reported highest percentage of Si content among all the ashes and trace amount of Cl, which is not observed in any other ashes used in this study. The presence of Cl may be due to the salt content in the soil and Si is due to sugarcane grown on the black cotton soil as this soil is rich in Si concentration. The elemental composition of BA-I is presented in Table 6.

Table 6: Elemental composition of BA-I ash

Element	*BA-I*	
	Weight (%)	*Atomic (%)*
O	*49.5*	*65.02*
Si	*41.16*	*30.74*
S	*0.45*	*0.35*
Ca	*0.53*	*0.66*
Fe	*0.19*	*0.05*
Al	*0*	*0*
K	*4.20*	*1.59*
Mg	*1.22*	*1.21*
Na	*0*	*0*
Cl	*2.75*	*0.43*
P	*0*	*0*
S	*0*	*0*

5. Conclusion

This article presents the link between microstructure and elemental composition of OPC and sugarcane bagasse ash collected from different boilers of sugar industry. Based on the results and discussions the following conclusions are drawn.

- It is observed that the microstructure of OPC particles is angular and irregular and contains predominately Ca, O and Si with trace amounts of Al, Fe, and Na. The morphology of BA-C shows crystalline and prismatic particles with well defined edges. EDS analysis of BA-C particles presents higher masses of O and Si with K, Ca and Mg as trace elements.
- The morphology of BA-V particles indicate denser and less crystalline as compared to OPC and BA-C particles.
- EDS map of BA-V particle presents O, Si, Ca, and K as major elements and Mg, Al, P and S as trace elements.
- SEM-EDS analysis of BA-M particles shows presence of carbon content due to incomplete calcination of sugarcane bagasse.
- SEM image of BA-I shows spongy in nature with high porosity and elemental composition of BA-I particles presents Si and O as major elements with lesser amounts of K, Ca, Fe, Cl, P and Mg. BA-I particles consists of highest concentration Si compared to other ashes and cement used during the study period.

From EDS analysis of Portland cement and sugarcane bagasse ashes shows similar chemical composition but variation in proportion is observed. Hence SBA can be used as supplementary cementitious material in the production of concrete.

Acknowledgment

The authors gratefully acknowledge to Mr. Arif, NIO, Dona Paula, Goa, for conducting SEM-EDS analysis of the materials.

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