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Chemical and Microstructural Effects of Different Calcinating Temperatures on Selected Pozzolans

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Abstract

Recent researches show that agricultural wastes can be reuse as pozolans; this contributes to our environmental sustenance. The need to successful carry out proper analysis contributes significantly to improving the overall use of the discovered pozolans. Therefore, this research aims to investigate the micro-structural and chemical analysis of some selected pozzolans at different calcinating temperatures. Rich husk ash (RHA), groundnut shell ash (GSA), locust beans pod ash (LBPA) and bamboo leaf ash (BLA) were obtained; their chemical and microstructural analysis at different calcinating temperatures (500˚C, 600˚C and 700˚C) were carried out using X-ray fluorescence and scanning electron microscope. The results show that the optimum calcinating temperatures considering the microstructure and chemical composition of RHA, BLA and LBPA were 700˚C, 500˚C and 600˚C respectively. These pozzolans were also classified according to ASTM 618 requirement.

Keywords

Agro-Waste, Calcinating Temperature, Chemical Composition, Microstructure, Pozzolans

1. Introduction

Pozzolans are partial substitute for cement in the construction industries. It can partially replace cement in the production of mortar, sandcrete blocks and concrete. Pozzolans are siliceous materials which by itself possess no cementitious properties but in processed and finely divided form, react in the presence of water with lime to form compound having cementitious properties [1] [2]. The use of pozzolans in cement industries is necessary to reduce the amount of CO_2 being emitted into the atmosphere during the production of cement and the high energy needed for cement production. In addition, agro-waste pozzolans help to manage our waste and improve our ecology system. Pozzolans from agricultural waste are very reactive in their finely divinely form and their performance could be optimized when calcinated at the right condition among which we have the right calcinating temperature. Calcinating temperature in pozzolans is the temperature at which the ashes were obtained. Morales *et al.* [3] studied the effects of calcinating conditions on the microstructure of sugar cane wastes ashes and discovered that calcinating conditions influence the microstructure and pozzolanic activation of pozzolans. Nuntachai *et al.* [4] also investigated the effects of loss on ignition (LOI) on the compressive strength and sulfate resistance of mortars as another calcinating condition and reported that LOI affects these two properties of the tested mortars at different ages of the concrete. Other researchers Salau and Osemeke [5] conducted their own research on the effect of calcinating temperature on pozzolanic characteristics of calcined kaolin (metakaolin). They discovered that the silica content of metakaolin increases with increase in calcinating temperature and time of calcination. They studied a temperature range of 600˚C and 1050˚C and recommended 750˚C for metakaolin production having considered the chemical and LOI properties of the pozzolans. In view of all these, there is active research in this area and hence the need to successful carry out proper analysis and test procedure contributes significantly to improving the overall use of any discovered pozzolan. This will in turn improve the quality of life by providing necessary infrastructure and other basic enhanced facilities. The reactivity of a pozzolan depends on its chemical and mineralogical composition, the type and proportion of its active phases which also depends on temperature. This research has been conducted on four different agro-waste ashes to establish their suitability under three different calcinating temperatures and the effect of the calcinating temperature on their chemical composition and microstructure. These four agro-waste ashes namely rice husk ash (RHA), groundnut shell ash (GSA), locust bean pod ash (LBPA) and bamboo leaf ash (BLA) has been previously discovered to improve the compressive strength of concrete as well as some other properties like chloride ion resistance [6] [7] [8] [9].

2. Experimental Investigation

Rice husk ash (RHA), groundnut shell ash (GSA), locust bean pod ash (LBPA) and bamboo leaf ash (BLA) were obtained and calcinated at different temperatures of 500˚C, 600˚C and 700˚C for 1 hour at a step of 100˚C in 1 hour in a close electric furnace. The ashes were then sieved using 50 μmm (2 μ inches) sieve as shown in **Figure 1**. Their chemical and microstructural analysis at different calcinating temperature (500˚C, 600˚C and 700˚C) were obtained alongside with their loss on ignition. The chemical compositions were obtained in term of their oxide compositions using X-ray fluorescence spectrometer [10] and their

Figure 1. Ashes samples for BLA LBPA, GSA and RHA after calcinations.

micro-structures were obtained with the use of scanning electron microscope [11] [12]. The X-ray fluorescence spectrometer test was carried out on an energy dispersive X-ray fluorescence spectrometer Shimadzu EDX-702HS operated at 40 kV and 18 mA. The current was adjusted automatically with a maximum of 1 mA. The samples were loaded into chamber through the sample trays giving utmost consideration to the samples labels *i.e.* the slot numbers. The oxide compositions of the various samples were then obtained appropriately with the use of EDX Shimadzu software package. The scanning electron microscope used for this research work is shown in Figure 2, the accelerated voltage, displayed magnification and the scale are all shown in the result.

Materials

RHA was obtained by sun drying rice husk, BLA by sun-drying fallen bamboo leaf, GSA was obtained by sun-drying ground shell while LBPA was obtained by sun-drying locust beans pods. Their ashes were then obtained by calcinating in an electric oven at different temperatures.

3. Experimental Results and Discussion

3.1. Test Result from X-Ray Fluoresce Spectrometry

The results of chemical analysis of pozzolanic materials samples use for this experiment carry out with x-ray fluorescence spectrometry are shown in Tables 1-4 for RHA, BLA, GSA and LBPA respectively.

The result for RHA in Table 1 shows the sum of S_1O_2 + AL_2O_3 + Fe_2O_3 at 500˚C, 600˚C and 700˚C calcinating temperature to be 76.6%, 77.8% and 79.77% respectively. This implies that calcinating RHA at any of this temperature will fulfilled the minimum required sum of the above oxides and other requirements for class N and F pozzolan according to ASTM 618 [1]. Among the three calcinating temperature under investigation, 700˚C is the optimum temperature for

Figure 2. Scanning electron microscope used for the research.

Table 1. Chemical composition of RHA at different calcinating temperature.

Chemical Composition	% Composition of RHA at 500˚C Calcinating Temperature	% Composition of RHA at 600˚C Calcinating Temperature	% Composition of RHA at 700˚C Calcinating Temperature
Silicon Oxide	71.80	74.50	74.48
Aluminum Oxide	3.28	2.34	3.47
Ferric oxide	1.72	1.08	1.82
Titanium oxide	0.30	0.29	0.31
Calcium oxide	4.84	2.84	3.62
Lead oxide	9.61	7.63	8.03
Magnesium oxide	0.58	0.62	2.10
Manganese oxide	0.16	0.51	0.81
Chromium oxide	0.41	0.32	0.32
Sodium Oxide	0.11	-	0.03
Potassium Oxide	5.20	3.65	3.67
Sulphide	0.18	0.38	0.18
Loss on Ignition	-	0.82	3.24

obtaining RHA if the chemical composition and all other requirement in ASTM 618 [1] is to be considered. For BLA, Table 2 shows the sum of S_1O_2 + AL_2O_3 + Fe_2O_3 for 500˚C, 600˚C and 700˚C calcinating temperature to be 84.53%, 81.32%, 81.89% respectively. Similarly the three calcinating temperatures satisfy all the required for class N and F which make them suitable for use as pozzolans of either class N or F. Considering the chemical composition the optimum calcinating temperature is at 500˚C. The result in Table 3 shows the chemical composition of GSA with the sum of S_1O_2 + AL_2O_3 + Fe_2O_3 at 500˚C, 600˚C and 700˚C calcinating temperatures to be 32.75%, 29.18% and 33.26% respectively. None of these chemical composition results fall into any class of pozzolans, this

Table 2. Chemical composition of BLA at different calcinating temperature.

Chemical Composition	% Composition of BLA at 500˚C calcinating temperature	% Composition of BLA at 600˚C calcinating temperature	% Composition of BLA at 700˚C calcinating temperature
Silicon Oxide	75.80	75.90	78.27
Aluminum Oxide	3.75	4.15	1.69
Ferric oxide	4.98	1.27	1.63
Titanium oxide	1.54	0.20	2.02
Calcium oxide	6.49	8.02	5.98
Lead oxide	0.21	2.19	0.81
Magnesium oxide	3.70	2.06	1.65
Manganese oxide	0.014	0.02	0.02
Chromium oxide	0.32	0.12	0.14
Sodium Oxide	0.16	0.24	0.28
Potassium Oxide	0.40	5.62	3.16
Sulphide	1.20	1.08	3.01
Loss on Ignition	1.12	0.09	-

Table 3. Chemical composition of GSA at different calcinating temperature.

Chemical Composition	% Composition of GSA at 500˚C calcinating temperature	% Composition of GSA at 600˚C calcinating temperature	% Composition of GSA at 700˚C calcinating temperature
Silicon Oxide	28.42	22.08	25.00
Aluminum Oxide	6.65	2.24	2.24
Ferric oxide	0.68	4.86	6.02
Titanium oxide	8.02	0.93	2.16
Calcium oxide	12.81	16.42	27.24
Lead oxide	1.79	2.12	1.06
Magnesium oxide	5.48	2.06	4.31
Manganese oxide	0.32	0.21	0.56
Chromium oxide	0.012	0.02	0.01
Sodium Oxide	1.08	-	-
Potassium Oxide	22.65	40.28	24.25
Sulphide	1.82	1.29	1.32
Loss on Ignition	4.52	8.37	5.29

is an indication that GSA is not a pozzolan or the calcinating temperature could be increased to further observe the trend. The result for LBPA in **Table 4** shows that the sum of S_1O_2 + AL_2O_3 + Fe_2O_3 at 500˚C, 600˚C and 700˚C calcinating temperatures are 65.43%, 66.41% and 62.03% respectively which satisfies the requirement for class C pozzolan. All the three calcinating temperatures are good

Table 4. Chemical composition of LPBA at different calcinating temperature.

Chemical Composition	% Composition of LBPA at 500˚C calcinating temperature	% Composition of LPBA at 600˚C calcinating temperature	% Composition of LBPA at 700˚C calcinating temperature
Silicon Oxide	39.24	42.30	39.21
Aluminum Oxide	14.08	10.15	9.58
Ferric oxide	12.11	13.96	13.24
Titanium oxide	0.61	0.87	0.58
Calcium oxide	17.43	13.82	12.85
Lead oxide	6.81	8.36	7.90
Magnesium oxide	2.01	2.35	2.43
Manganese oxide	1.23	2.08	1.26
Chromium oxide	0.20	0.20	0.19
Sodium Oxide	1.40	0.96	1.05
Potassium Oxide	7.83	4.82	5.12
Sulphide	1.31	1.61	1.56
Loss on Ignition	6.30	6.43	6.08

but the optimum for LBPA is 600˚C considering the chemical composition. Therefore each of these pozzolans can be calcinated at their optimum temperature so as to maximize their chemical properties as well as preventing energy and performance loss through over calcination. LOI obtained also differs for all the ashes at different calcinating temperatures. RHA and BLA satisfy the requirement on LOI in accordance with ASTM 618 [1].

3.2. Test Result from Scanning Electron Microscopy

Result of the scanning electron microscope for GSA at 50 and 100 magnifications are shown in **Figures 3-8**. These images for GSA at 500˚C, 600˚C and 700˚C calcinating temperatures show changes in its microstructure in term of sizes, packing and interlocking spaces. The microstructure improves in term of these features as the calcinating temperature increases having the optimum at 700˚C. Better interlocking and packing of the microstructure particles is visible in **Figures 3-8**. Using the optimum calcinating temperature with improved microstructural properties increase will increase the durability and other properties of such concrete [13] [14].

Figures 9-14 show the SEM image for BLA, the best improve microstructure being at 600˚C. Comparing the SEM images for RHA to BLA shows a difference in the structure shape. RHA exhibits a needle-like shape while BLA exhibits a robust-like shape for all the calcinating temperatures. The optimum temperature of 600˚C and not 700˚C obtained for BLA shows that pozzolans can be over calcinated thereby loosing its properties [15] [16]. Therefore, proper calcinations at the right temperature are necessary in production of pozzolans to allow for the

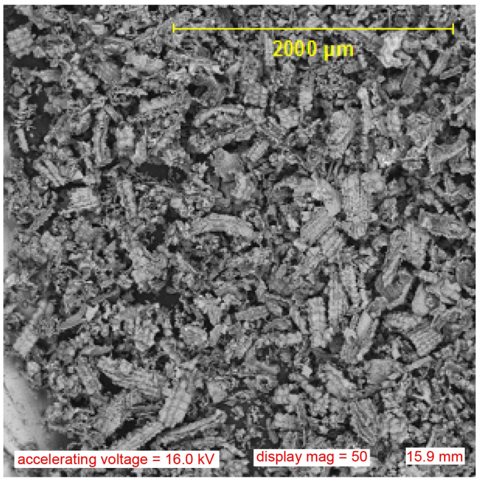

Figure 3. SEM image for RHA at 50 display mag. at 500˚C.

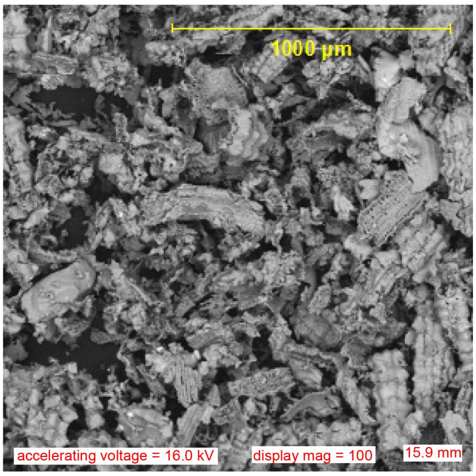

Figure 4. SEM image for RHA at 100 display mag. at 500˚C.

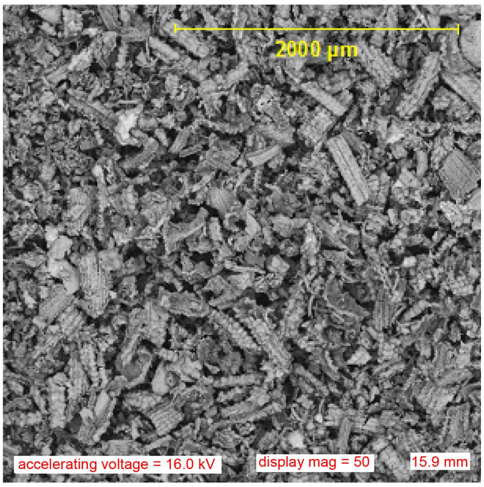

Figure 5. SEM image for RHA at 50 display mag. at 600˚C.

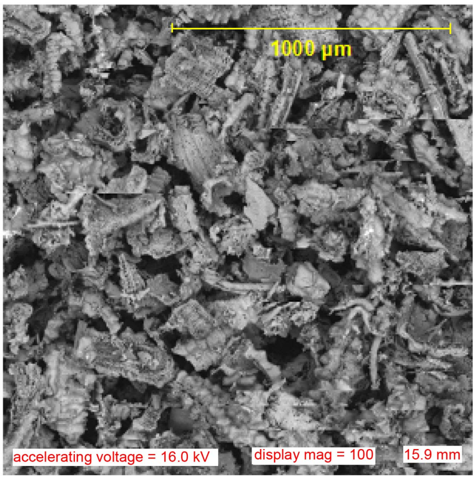

Figure 6. SEM image for RHA at 100 display mag. at 600˚C.

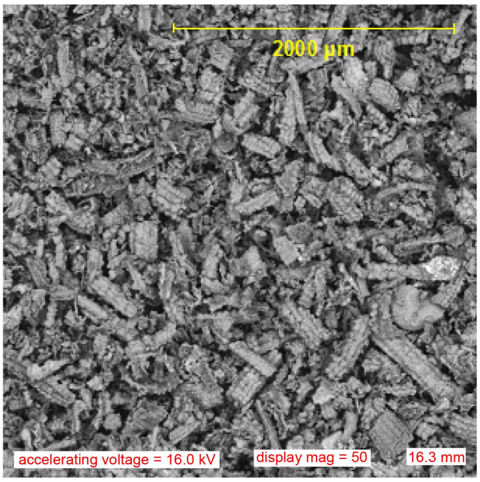

Figure 7. SEM image for RHA at 50 display mag. at 700˚C.

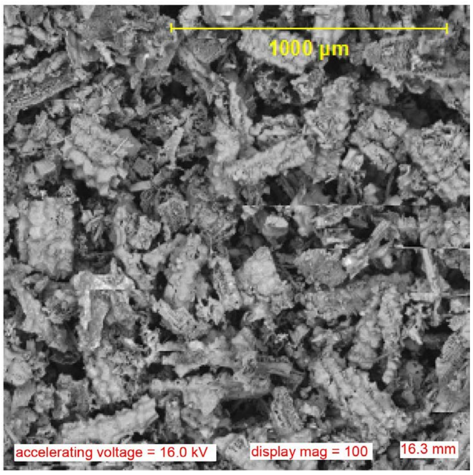

Figure 8. SEM image for RHA at 100 display mag. at 700˚C.

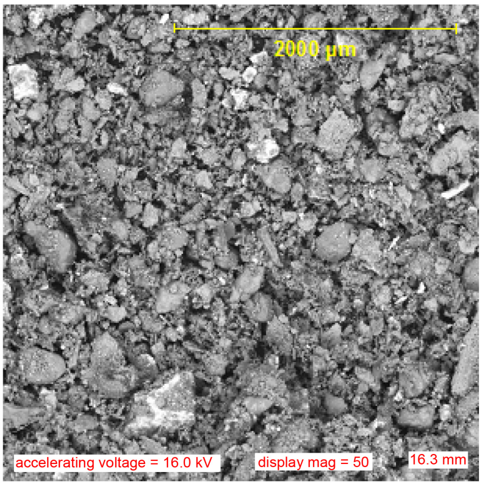

Figure 9. SEM image for BLA at 50 display mag. at 500˚C.

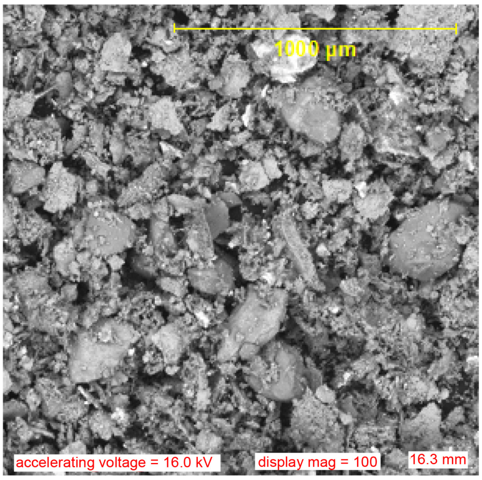

Figure 10. SEM image for BLA at 100 display mag. at 500˚C.

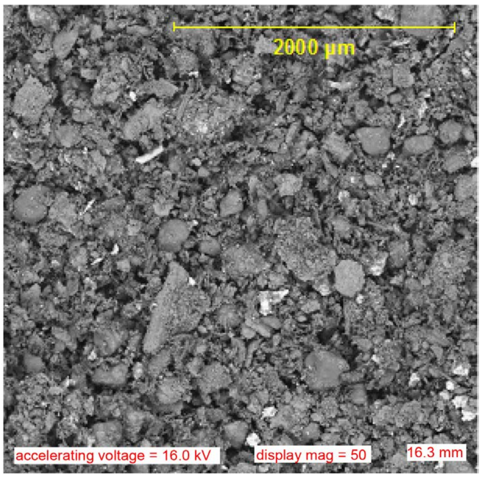

Figure 11. SEM image for BLA at 50 display mag. at 600˚C.

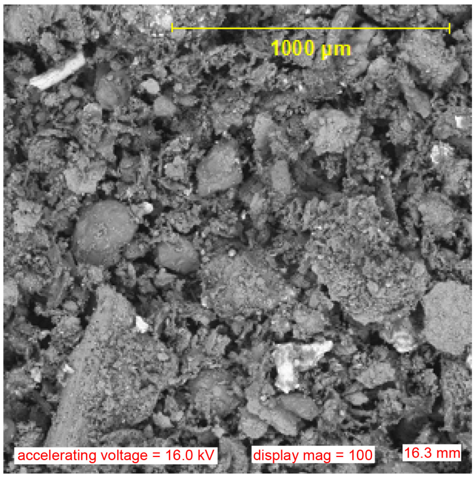

Figure 12. SEM image for BLA at 100 display mag. at 600˚C.

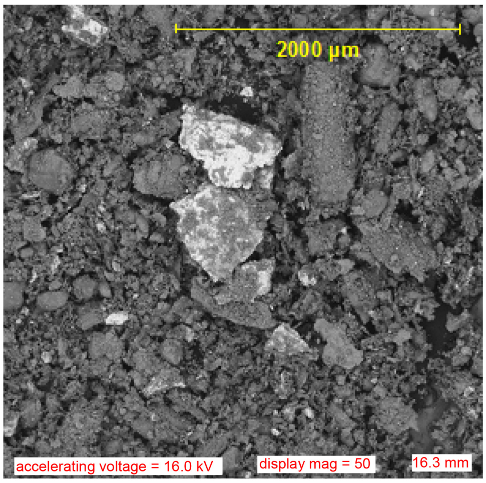

Figure 13. SEM image for BLA at 50 display mag. at 600˚C.

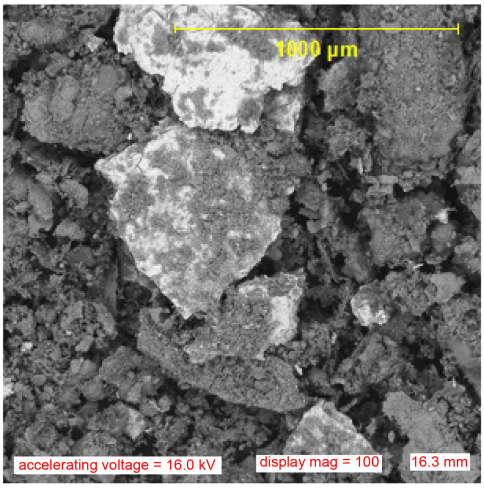

Figure 14. SEM image for BLA at 100 display mag. at 600˚C.

efficient use of their chemical and microstructural properties among others [16].

GSA shows a very good microstructure at 700˚C calcinating temperature than the remaining calcinating temperatures as shown in **Figures 15-20**. It exhibits both needle-like shape and robust-like shape.

For LBPA SEM images shown in **Figures 21-26** show the best microstructure to be at 600˚C calcinating temperature. The shape is cluster. All these results show that pozzolans exhibit different microstructure at different calcinating temperatures. This features should be considered alongside with the chemical composition and other criteria when selecting the optimum calcinating temperature for any kind of pozzolans.

The SEM images shown in **Figures 3-26** for RHA, BLA, GSA and LBPA further revealed that different pozzolans exhibits different shapes, RHA shows needle like shapes, BLA shows robust-like shape, GSA combines the two shapes

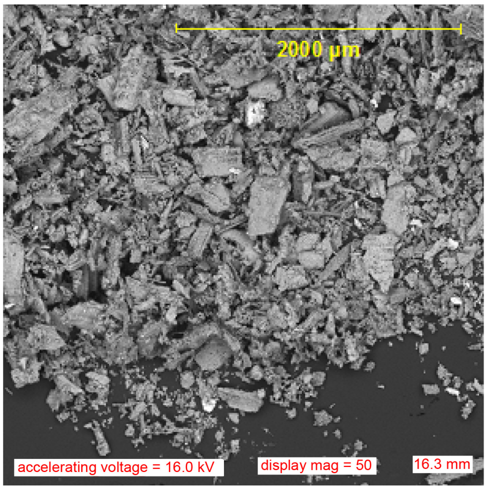

Figure 15. SEM image for GSA at 50 display mag. at 500˚C.

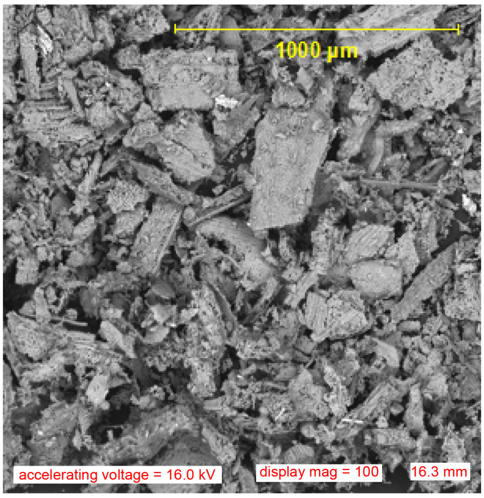

Figure 16. SEM image for GSA at 100 display mag. at 500˚C.

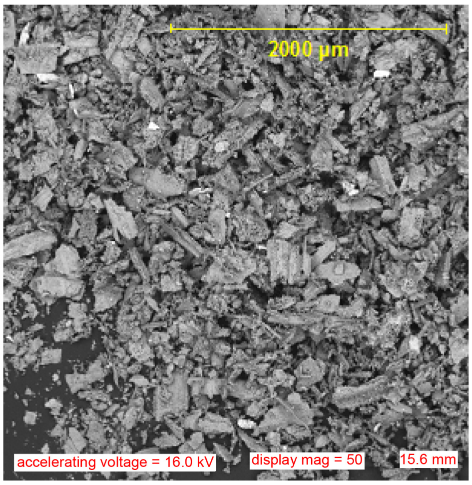

Figure 17. SEM image for GSA at 50 display mag. at 600˚C.

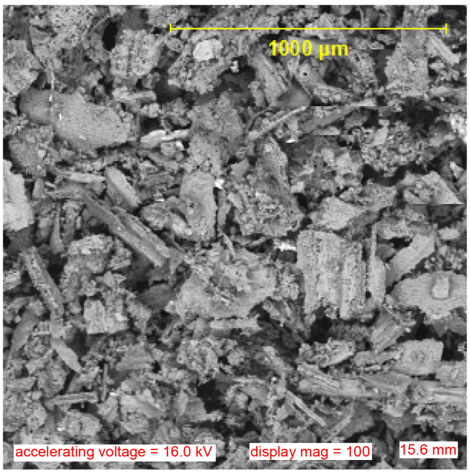

Figure 18. SEM image for GSA at 100 display mag. at 600˚C.

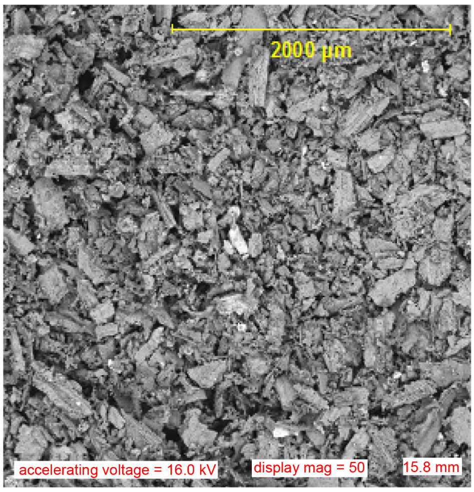

Figure 19. SEM image for GSA at 50 display mag. at 700˚C.

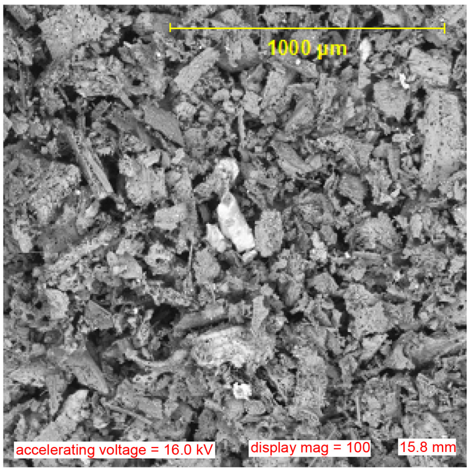

Figure 20. SEM image for GSA at 100 display mag. at 700˚C.

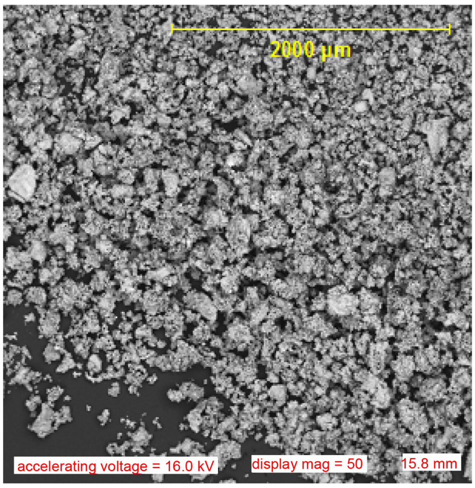

Figure 21. SEM image for LBPA at 50 display mag. at 500˚C.

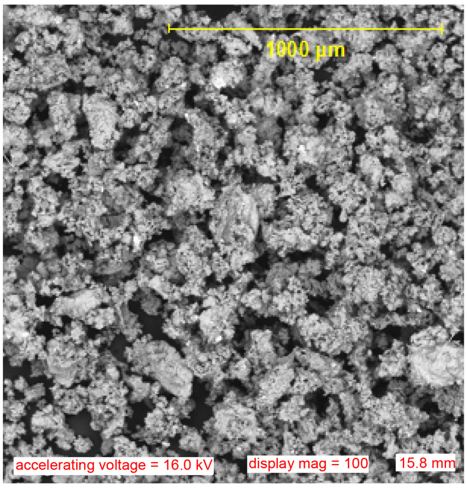

Figure 22. SEM image for LBPA at 100 display mag. at 500˚C.

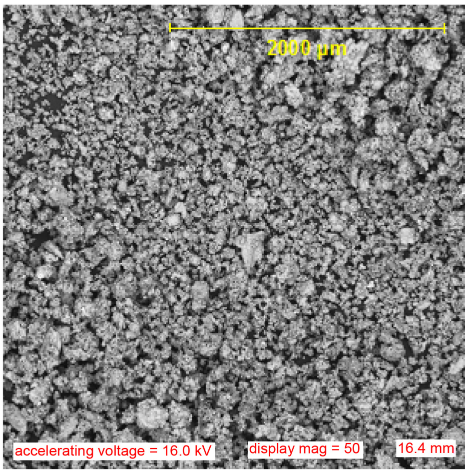

Figure 23. SEM image for LBPA at 50 display mag. at 600˚C.

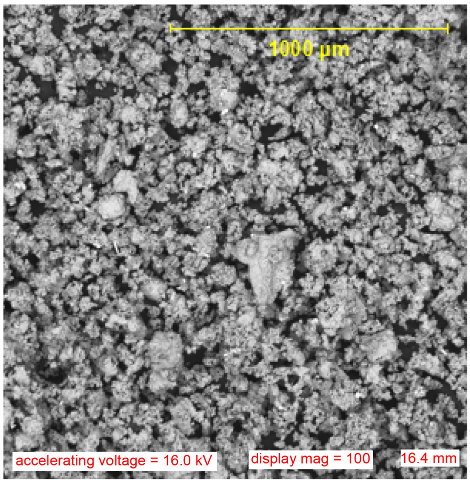

Figure 24. SEM image for LBPA at 100 display mag. at 600˚C.

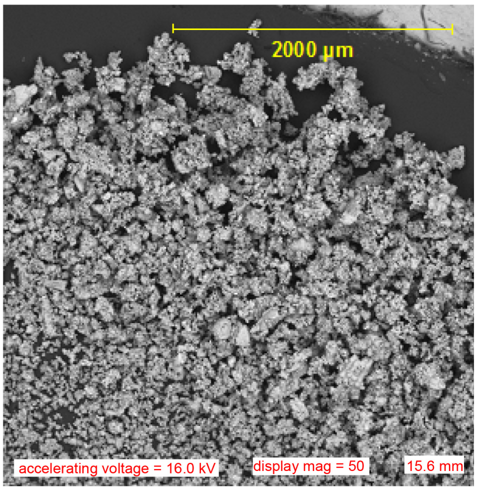

Figure 25. SEM image for LBPA at 50 display mag. at 700˚C.

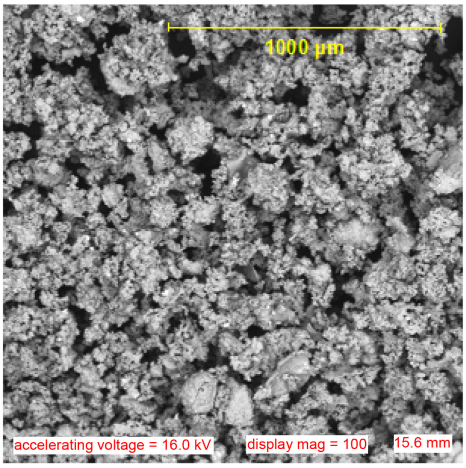

Figure 26. SEM image for LBPA at 100 display mag. at 700˚C.

while LBPA shows minute clustering shapes.

4. Conclusions

Considering the result of this research work, the following conclusion can be drawn:

1) RHA, BLA and LBPA are pozzolanic materials with RHA belonging to class N or F while LBPA belongs to class C according to ASTM 618.

2) Calcinating temperatures affect the chemical composition of different pozzolans as well as their microstructure. Different calcinating temperature shows variation in chemical composition, microstructure as well as loss on ignition.

3) In determining optimum calcinating temperature for any pozzolans their chemical composition, microstructure and loss on ignition should be properly considered among other criteria to prevent under utilisation of such pozzolans.

4) Different pozzolans exhibits different shapes after calcination.

Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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