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CHARACTERIZATION AND PRODUCTION OF CALCIUM COMPOUND FROM WASTE EGG SHELLS

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Abstract : The characterization and production of calcium compound from waste Egg Shell was carried out in this study. The elemental/chemical composition, the material purity as well as structural morphology of the waste Egg Shell were examined both before and after the production of the calcium compound. Calcium compound was produced from these waste eggshells while varying the concentration of acid used at a range of 0.1 mol/L to 0.5 mol/L and the ratio of weight of eggshells to volume of acid used (between 1g:100mL to 5g:100mL). The result of this shows the optimal % yield obtained to be 87.5% at 0.5 mol/L [HCl] and 5g:100mL ratio of weight of eggshells to volume of acid used. The result of the XRD obtained confirmed the processed calcium compound to be anorthite ($CaAl_2Si_2O_8$), 00-009-0464 with a couple of other impurities such as sylvite (KCl) 00-002-0412, marialite ($Na_4Al_3Si_9O_{24}Cl$) 00-002-0412 and mellite ($Cl_2Al_2O_{12}.16H_2O$) 00-042-1501, while the SEM confirms the formation of anorthite.

Keywords: Eggshells, Calcium compound, Anorthite, Chemical composition.

1.0 INTRODUCTION

Calcium is a silvery white metal (alkaline earth) with atomic number 20 and a cubic crystal structure. It is a reactive metal that forms a dark oxide-nitride layer when exposed to air. Although, calcium is the fifth most abundant element and the third most abundant metal in the earth crust by weight, however, it is mostly found in its metallic form in nature since it easily reacts with oxygen and water (Ropp and Richard, 2012). Pure calcium metals have few applications due to its high reactivity; but, in small quantities it is often used as an alloying component in steelmaking, and sometimes, as a calcium–lead alloy, in making automotive batteries. Other calcium compounds, such as quick lime (CaO), slaked lime ($Ca(OH)_2$), limestone ($CaCO_3$), gypsum ($CaSO_4.2H_2O$), anhydrite ($CaSO_4$), and hydroxyapatite ($Ca_5(PO_4)_3(OH)$), have all been known and used by human beings for several thousands of years. For instance, calcium bromide ($CaBr_2$), is a white powder which have many uses and is present in fire retardants, drilling fluids, cooling agents in freezing mixtures, food preservatives, and medication for the treatment of anxiety; quicklime (CaO) is used in water purification devices for drinking water production (Struminska, 2015). In recent past, calcium has been processed from mineral ores such as limestone, calcites, dolomite and gypsum, but other calcium containing components such as bone shells, egg shells, oysters have been neglected to constitute nuisance in the environment in this part of the world.

An eggshell is the hard, outer covering of an egg made up of mostly calcium carbonate, a common form of calcium while the rest is made up of protein and other minerals (Nakano *et al.* 2003). Currently, eggs are being used to make a variety of products such as cakes, salad dressings and quiches, whose production results in several daily tons of eggshell waste as a by-product and incur considerable disposal costs throughout the world. According to the chemical analysis, eggshell contains about 92-95 % calcium carbonate with 5-7% organic membranes (Yamamoto *et al*. 1996). Large amounts of eggshell are generated everyday around the world, most of which are passed through egg breaking industries in which eggshell are broken and converted to various liquid egg products. Eggshell powders, however, have physical, chemical and a crystalline structure similar to commercial limestone (Rovenský, 2003). Waste eggshell contain about 92-95 % limestone (Yamamoto *et al*. 1996) which is a carbonate sedimentary mineral containing impurities and is processed to produce pure limestone powder (Teir *et al*. 2005). This research work therefore aims at the production and characterization of calcium compound from waste egg shell.

2.0 MATERIALS AND METHODS

The egg shells sample used in this work was collated and collected from a small chops bakery outlet within The Federal Polytechnic Ede, Osun State, Nigeria. The eggshells obtained were washed both inside and outside to get rid of dirt and other organic materials, and then sun dried for five (5) days. The dried egg shell samples were then crushed, milled and sieved to yield fine powder. The crushed and dried eggshells were then packed and stored in air tight containers at room temperature and used as raw material for the duration of the study. The crushed and dried samples were subjected to X-Ray Diffraction **(**XRD) and Scanning Electron Microscopy (SEM) analyses to assess the material purity and structural morphology of the raw egg shells, respectively. The material purity assessment was analyzed using the reflection-transmission spinner stage employing the Theta-Theta settings and the peaks obtained from these analyses were matched with the mineral phases from the PDF 2 ICDD (International Centre for Diffraction Data) database attached to the Pan analytical Xpert Highscore Plus XRD software while, the structural morphology was assessed using the Phenom Pharos BSD 15 kV SEM machine.

2.1 Calcium compound preparation

The crushed and dried eggshell samples were weighed into five different beakers respectively, and labelled accordingly. These beakers had 1g, 2g, 3g, 4g and 5g each. The samples were mixed with the prepared hydrochloric acid solutions (concentration range of 0.1 mol/L to 0.5 mol/L) at a solid to liquid ratio of 10 g/L and stirred occasionally until no gas bubbles were observed for 3 hours. The mixture was filtered, until the supernatant was separated and then heated at 100ºC until it was dried. The same procedure was repeated for the other acid concentrations. The percent yield of eggshell calcium compound was measured for each of these parameters and the condition that gave the highest yield was chosen to be the optimal reaction condition. The calculation of percent yield is shown in equation below:

$$\%\ yield = \frac{weight\ of\ compound\ obtained\ after\ drying}{weight\ of\ crushed\ eggshell\ used}\ x\ 100$$

The calcium compound produced was subjected to XRD and SEM analyses to determine the composition and the structure of what was produced.

3.0 RESULTS AND DISCUSSION

The XRD result as shown in Figure 1 explains the crushed and dried eggshell samples to be made of calcite, $CaCO_3$ {01-072-4582}; quartz, SiO_2 {01-070-3755}; andradite, $Ca_3Fe_2^{3+}(SiO_4)_3$ {00-003-0814}; garnet, 3(Ca,Fe,Mg)O.(Al,Fe,Cr)Si_3O_{12} {00-002-0981} and lime, CaO {00-003-1123} with calcite being the prominent constituent with about 70% composition .

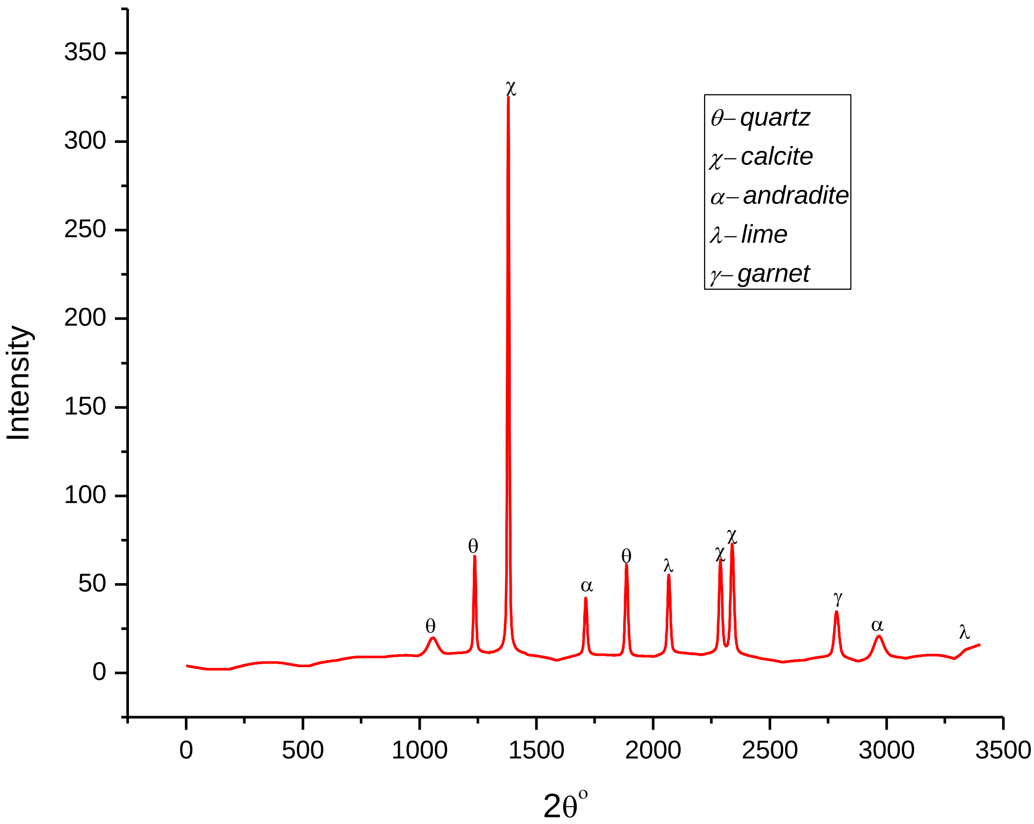

Figure 1: XRD pattern of the raw crushed eggshell with their respective International Centre for Diffraction Data (ICDD) file number for peaks assignments showing Calcite ($CaCO_3$) 01-072-4582; Quartz (SiO_2) 01-070-3755; Andradite ($Ca_3Fe_2^{3+}(SiO_4)_3$) 00-003-0814; Garnet (3(Ca, Fe, Mg)O.(Al, Fe, Cr)Si_3O_{12}) 00-002-0981 and Lime (CaO) 00-003-1123.

The morphological study on the other hand (Figure 2) shows the presence of shining particles which could be attributed to the dominant presence of $CaCO_3$. This is corroborated by the values as examined by the EDS (Figure 3) where Ca has a weight concentration of about 62.50%, C as 8.67% and O as 3.09%. Other portions of the micrograph can be attributed to the presence of mixture of organic matters and magnesium carbonate (Baba et al., 2013; Schwetman and Cornell, 1991).

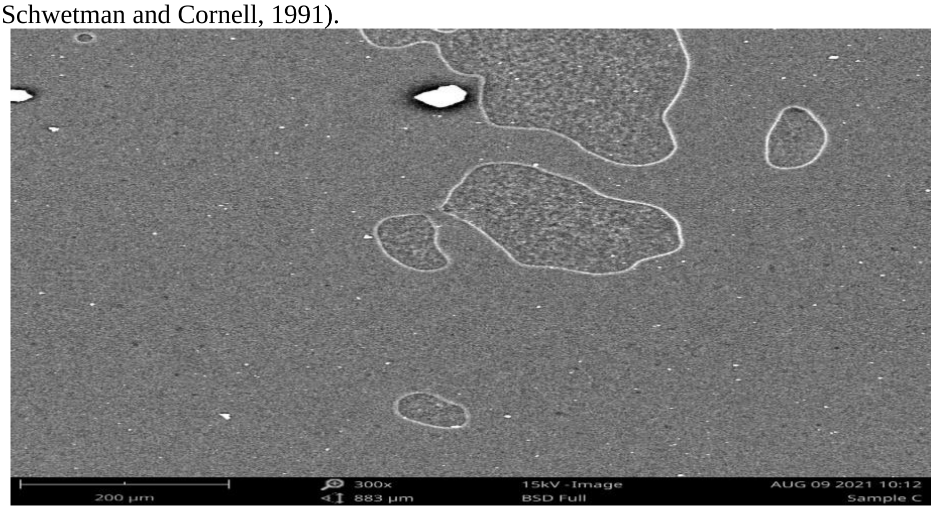

Figure 2: Scanning Electron Image of raw egg shell.

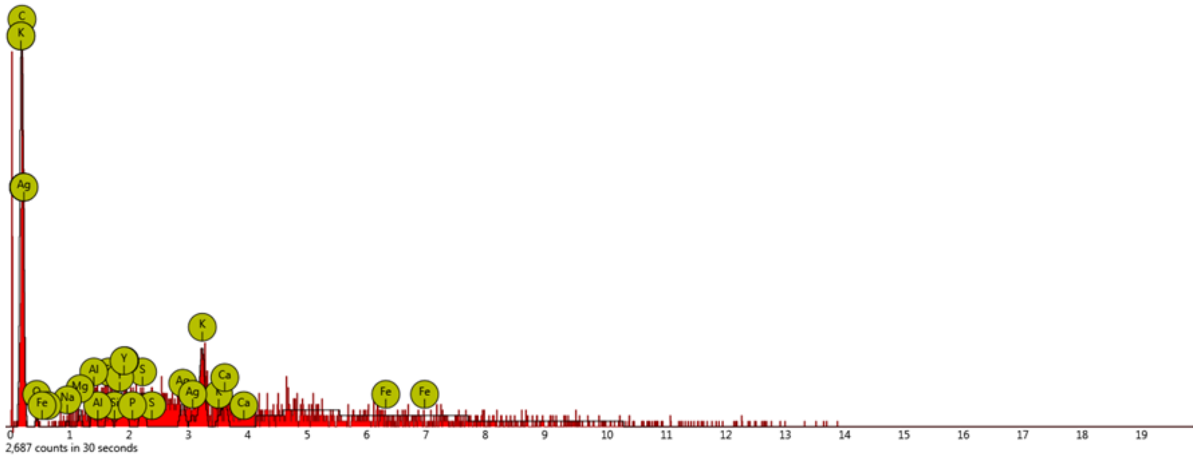

Figure 3: Energy Dispersive Spectra of raw egg shell.

The treatment of eggshell with the different concentrations of HCl and the ratios of weight of eggshell versus volume of acid used gave the table as summarised below.

Table 1: % yield of HCl treated eggshell calcium compound

Treatment	Conc. of acid used (mol/L)	Ratio of wt of eggshell to vol. of acid used (g:mL)	% yield
1	0.1	1:100	10.0
2		2:100	15.8
3		3:100	21.6
4		4:100	30.5
5		5:100	41.0
6	0.2	1:100	14.8
7		2:100	22.3
8		3:100	28.2
9		4:100	39.0
10		5:100	47.4
11	0.3	1:100	23.8
12		2:100	33.0
13		3:100	38.7
14		4:100	51.3
15		5:100	59.6
16	0.4	1:100	29.3
17		2:100	39.6
18		3:100	49.5
19		4:100	63.7
20		5:100	74.5
21	0.5	1:100	40.4
22		2:100	52.8
23		3:100	65.6
24		4:100	76.7
25		5:100	87.5

From the table, treatment 1 to 5 had a significant increase as the ratio of weight of eggshell to volume of acid increased; this showed a spike in % yield from 10.0 % to 41.0%. Similar trends were obtained when the concentration of acid used increased. At 0.2 mol/L of HCl, 47.4% yield was observed as the optimal yield, while at 0.3 mol/L, 0.4 mol/L and 0.5 mol/L, the optimal % yield obtained were 59.6%, 74.5% and 87.5%, respectively. This shows that as the concentration of acid used increases, the % yield increases.

Treatment numbers 25 and 20 with the highest yield percentages were then subjected to XRD and SEM analyses to ascertain the purity and morphological composition of the processed calcium containing powder.

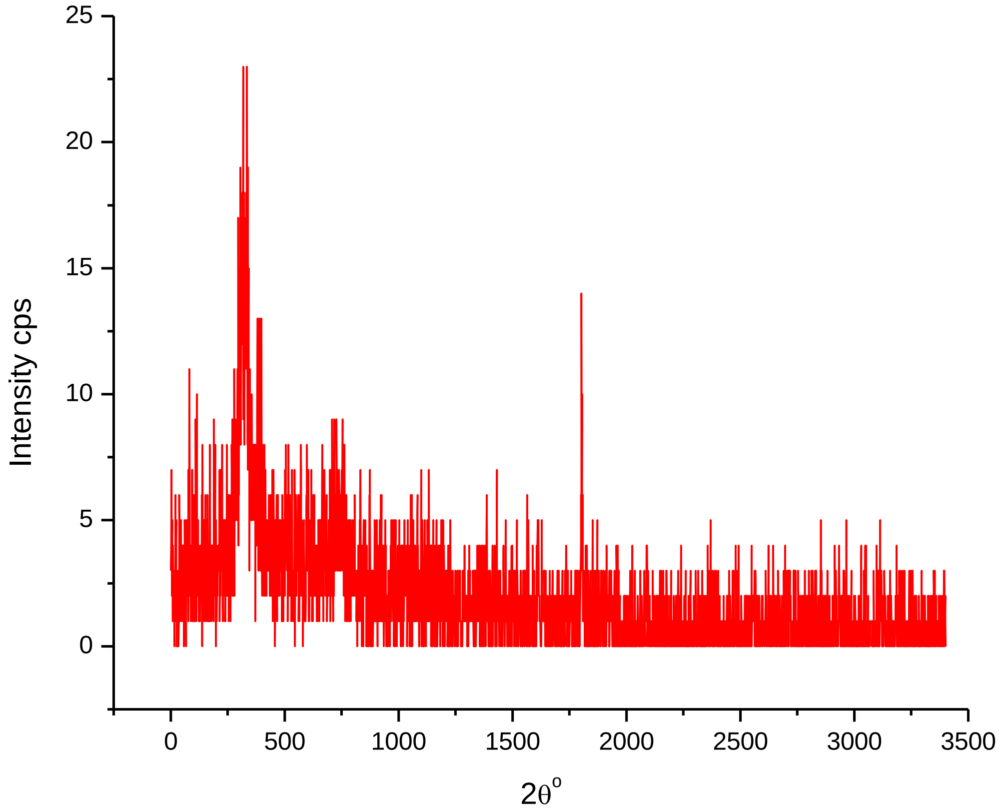

Figure 4: XRD spectra of processed calcium compound

Figure 4 displaying the XRD spectra shows the processed calcium compound to be majorly made up of Anorthite ($CaAl_2Si_2O_8$), ICDD file number 00-009-0464 and a couple of impurities such as Sylvite (KCl) 00-002-0412, Marialite ($Na_4Al_3Si_9O_{24}Cl$) 00-002-0412 and Mellite ($Cl_2Al_2O_{12}.16H_2O$) 00-042-1501.

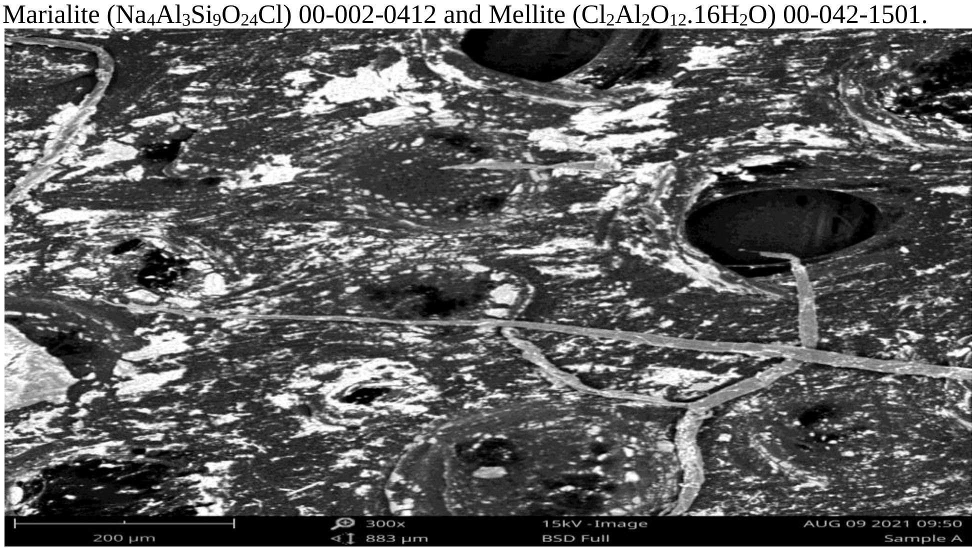

Figure 5: SEM image of processed anorthite

The morphological structure as shown in Figure 5, however, displays dense grains that are distributed throughout the matrix phase to form an interconnected network with pores essentially located around the matrix phase (Traore et al., 2003). This is evident of the formation of anorthite as predicted by the EDXRD result above.

4.0 CONCLUSION

The production and characterization of calcium compound from waste egg shells sourced around The Federal Polytechnic Ede were studied. Mineralogical characterization such as XRD and SEM were carried out on the samples so as to ascertain the elemental/chemical composition, the material purity as well as the structural morphology of the waste eggshell. Calcium compound was produced from these waste eggshells while varying the concentration of acid used at a range of 0.1 mol/L to 0.5 mol/L and the ratio of weight of eggshells to volume of acid used (between 1g:100mL to 5g:100mL). The result of this shows the optimal % yield obtained to be 87.5% at 0.5 mol/L [HCl] and 5g:100mL ratio of weight of eggshells to volume of acid used. The result of the XRD obtained confirmed the processed calcium compound to be anorthite ($CaAl_2Si_2O_8$), 00-009-0464 with a couple of other impurities such as sylvite (KCl) 00-002-0412, marialite ($Na_4Al_3Si_9O_{24}Cl$) 00-002-0412 and mellite ($Cl_2Al_2O_{12}.16H_2O$) 00-042-1501, while the SEM confirms the formation of anorthite.

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