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# Green Bio-Based CaO from Guinea Fowl Eggshells

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## Abstract

Eggshells are among the emerging hazardous waste from the food processing industry. This work sought to valorize waste guinea fowl eggshells. Guinea fowl eggshells (GFEs) were evaluated in the production of CaO for chemical and industrial application. The functionality, thermal stability, elemental composition, phase distribution and surface morphology properties of uncalcined GFEs and GFEs calcined at 700°C, 800°C, 900°C, 1000°C and 1100°C were systematically studied by FTIR, TGA, XRF, XRD and SEM-EDX respectively. The elemental analysis revealed Ca as the main element in the GFEs. The uncalcined GFEs showed intense peaks that corresponded to calcite $(CaCO_3)$ phases. These transformed into $Ca(OH)_2$ as the temperature of calcination increased and finally to CaO in the FTIR analysis. In the XRD diffractograms, the main peaks at $2\theta$ values were 29.466° for the uncalcined GFESs and at 37.377° for the sample treated at 1100°C. The phases were confirmed as CaO when compared with JCPDS files. Using the Scherer equation, the CaO crystallite size for the sample calcined at 1100°C was found to be 50.68 nm along the (2 0 0) orientation. All the samples showed multi-step decomposition patterns in the thermogravimetric analyses (TGA), with weight loss of up to 47% for the uncalcined GFEs sample, which was mainly due to the transformation of the calcite $(CaCO_3)$ phase to CaO by removal of bound water, organic components, and $CO_2$. Samples calcined at 1100°C showed mainly CaO phases in XRD analyses and fairly stable with 7% loss in weight after treatment at 800°C. SEM images of samples calcined at 900°C were irregular compared to samples treated at 1100°C. EDX data revealed that the surface structure was 100% calcium and oxygen. GFEs are a potential source of pure calcium oxide for various industrial uses.

## Keywords

Sustainable Materials, CaO, Biowaste, TGA, SEM-EDX, XRD

## 1. Introduction

To guarantee sustainable development, there is the need to consider valorization of waste to ensure zero waste standards. Again, in the wake of "green chemistry" there is the need to develop new sustainable materials that are reusable, recyclable and green [1]. In this context there is a growing interest in the use of naturally occurring and bio-based compounds as industrial feedstock.

There has therefore been a shift in attention in research into developing new clean technology and more environmentally friendly compounds to substitute the use of harmful reagents and catalysts in the chemical industry [2]. In a review published recently by Laca *et al.,* [3], egg processing waste was listed among the emerging problems in the food industry and considered as hazardous waste by UE regulations. However, waste eggshells have opportunities for the bioeconomy if new uses are ascribed to these materials. Waste eggshells are some of the useful agricultural bio-wastes that fall directly under the category of materials that are biodegradable, recyclable, and biocompatible, with a good osteoconductivity [4]. Annually, millions of tonnes of various eggshells go waste across the world, especially from the food processing industries [5]. In Brazil, 12,000 tonnes of avian eggshell waste are generated annually [6] and 15,000 tonnes in Canada annually. [5] These materials which generate $H_2S$ with associated offensive smell are usually disposed in open disposal sites or landfills.

The functional characteristics of eggshells are currently the object of intense research [7] [8]. The avian eggshell is about 11% of the egg weight [9] [10] and contains about 93% - 96% $CaCO_3$ [9] [11]. Eggshells [12] [13] and other shells from various animals, including oysters [14], mud crab [15] snails, molluscs [16] and ducks have been successfully converted into CaO. These have been used in the development of value added products [17], such as non-conventional heterogeneous catalysts for biodiesel production [18] [19] [20] as a strengthening agent in ceramic materials [6] and for the synthesis of hydroxyapatite used as scaffolds in biomedical applications [21] [22]. CaO from some eggshells have been successfully used as green, rapid and efficient catalysts in biodiesel production and other organic synthesis [23] [24] [25]. As indicated by Eletta *et al.,* [26] eggshell-CaO has been used as an adsorbent for metals in waste water treatment with negligible environmental impacts and high reusability. There is however, no report on the production, characterization and use of CaO from guinea fowl eggshells. The helmeted guinea fowl (*Numida meleagris*) accounts for almost 98% of birds kept by farmers in Northern, Upper East and Upper West Regions of Ghana and over 7% of the total poultry production in Ghana. With about 70% of the vegetation ideal for its production [27] [28], there are currently various interventions aiming at expanding production for food security and poverty reduction. Although there have been numerous studies on the socio-economics for guinea fowl production none has considered the potential uses of the waste eggshells associated with production.

This study explored for the first time the synthesis and characterization of

guinea fowl eggshells (GFEs) as an alternative source of CaO. The focus of the work was to thoroughly characterize and compare the functional and chemical properties of untreated GFEs, synthesized CaO (from the calcined GFEs) and commercially acquired CaO using Fourier Transform infra-red spectroscopy (FT IR), thermogravimetric analysis (TGA), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and energy dispersive X-ray fluorescence spectroscopy (EDXRF).

## 2. Experimental

### 2.1. Materials

Commercial $CaCO_3$ and CaO (95%) of analytical grade were purchased from BDH, Labo Chemie (Mumbai, India). Waste guinea fowl eggs shells were collected from domestic waste, over a period of four weeks from the Sunbrungu area in the Upper East Region of Ghana.

### 2.2. Sample Preparation and Calcination of GFESs

The eggshells were washed with running water and rinsed in warm water to reduce organic materials. The sample was oven dried at 105°C for 2 hours and crushed in a stainless steel grinder. The GFEs were calcined by heating at elevated temperatures in the range of 700°C - 1100°C with 2 hours hold time. The calcined samples were stored in capped glass vials to avoid reaction with air and moisture.

### 2.3. Instrumental Methods for Characterization of Guinea Fowl Egg Shells (GFESs)-CaO

The functional groups of GFEs and calcined samples were identified by Fourier transform infrared (FTIR) analyses using a Perkin Elmer spectrum II (94133) in ATR mode, with NIOS2 at a resolution of 4 and 24 scans, at 400 - 4000 cm$^{-1}$ and with a resolution of 1 cm$^{-1}$. Background spectra were obtained before sampling. The crystalline phases and qualitative composition of the samples were studied by X-ray diffraction (XRD) using an X-ray tube with Cu k$a$ radiation ($\Lambda$ = 1.5412) source accelerated at 40 mA and 40 kV and a graphite secondary beam monochromator. The radiation was in the range of 10 - 80 2$\theta$ angle. The intensity was measured by continuous scanning with a step size of 0.033 and measuring time of 100 s. The phases were identified by comparing with Joint Committee on Powder Diffraction Standard (JCPDS) files. The thermal stability and weight loss of the uncalcined and calcined samples were evaluated by thermo-gravimetric analyses (TGA) (Mettler Toledo) in a temperature range of 30°C - 1000°C at a heating rate of 10°C min$^{-1}$ under nitrogen atmosphere and the samples held at 1000°C for 10 minutes. Elemental composition analysis was investigated by using a hand held XRF instrument. Scanning electron microscopy (SEM) images were taken at 2000x and 10000x magnification. EDX was used to characterize the elemental composition of the surface structure of the GFESs

samples. The loss in ignition of the GFEs samples was determined by calculation using the relationship: $Loi = (A_i - A_f)/A_i \times 100$; where $A_i$ is the weight of the sample before calcination and $A_f$ is the weight of the sample after calcination. Colour change analysis of samples was by visual inspection.

## 3. Results and Discussion

### 3.1. Effect of Calcination on GFEs

The calcination process was accompanied by a series of colour changes, from light brown to grey and then white as the temperature increased from 700˚C to 1100˚C. The grey colour transformation observed at both 700˚C and 800˚C was due to the combustion of egg membranes and other organic components reducing the sample to mainly calcite ($CaCO_3$). Formation of Lime (CaO), a white crystalline material produced from the decomposition of $CaCO_3$ at elevated temperatures was confirmed above 800˚C with the formation of white samples. These transformations during calcination accounted for the corresponding weight loss in the samples (Table 1).

### 3.2. Thermal Stability and Decomposition Pattern of GFES-CaO Studied by Thermogravimetric Analysis

Thermal decomposition of GFES-CaO was investigated by TGA (Figure 1). The thermograms of samples depict a multi-step decomposition. Minor weight losses were observed between 0˚C - 300˚C which may be due to removal of moisture and organic components, with major loss occurring between 300˚C and 800˚C (decomposition of $CaCO_3$). TGA revealed weight loss of about 47% in the uncalcined GFESs mainly due to the decomposition of $CaCO_3$ and formation of CaO and $CO_2$. This weight loss was similar to those observed for samples calcined above 800˚C in Table 1. The sample treated up to 1100˚C was fairly stable after a temperature of 450˚C with about 7% loss in weight. No weight loss was observed after about 850˚C for the uncalcined GFESs in TGA, which implies that complete transformation had occurred at this temperature; thus optimum. Previous studies by Singh and Sharma [29] indicated that when calcination temperature is

**Table 1.** GFESs weight loss at varying temperature treatments with corresponding colour changes (in muffle furnace).

| Temperature treatment (˚C) | Loi (%) | Colour of sample |
| --- | --- | --- |
| 105 (Uncalcined GFESs) | 0.98 ± 0.05 | LB |
| 700 | 12.93 ± 0.67 | DG |
| 800 | 46.58 ± 0.15 | LG |
| 900 | 47.65 ± 0.01 | W |
| 1000 | 47.73 ± 0.19 | W |
| 1100 | 48.48 ± 0.24 | W |

Loi—weight loss on ignition, LB—light brown, DG—dark grey, LG—light grey, W—white.
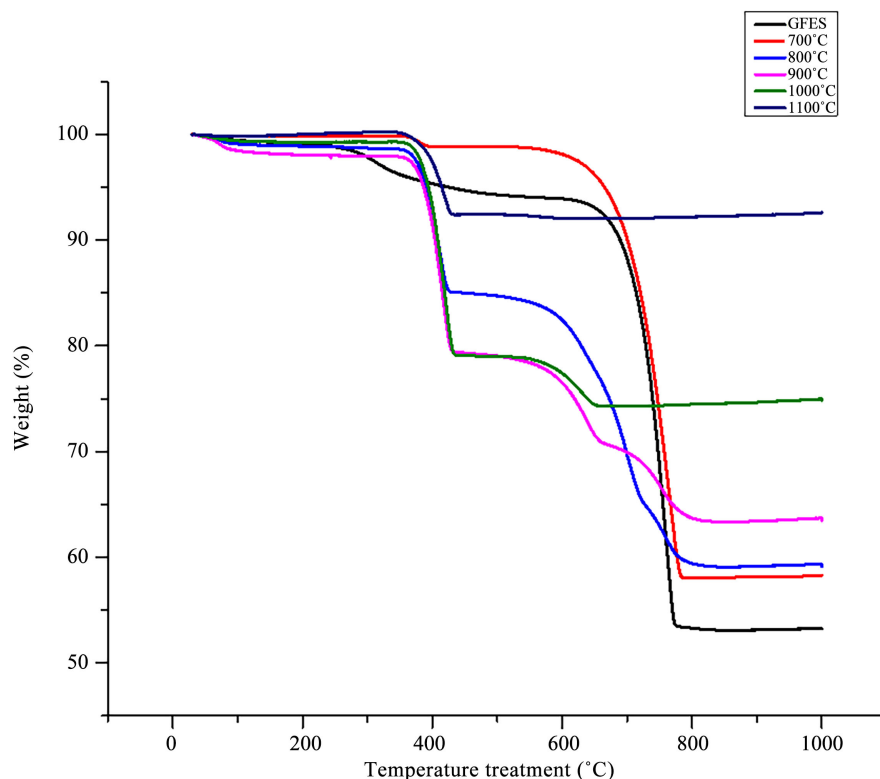
**Figure 1.** TGA analysis of uncalcined and calcined GFESs.

beyond 1000˚C it increases sintering effect. This phenomenon reduces the catalytic activity of CaO due to reduced surface area. This could have accounted for the slight increase in masses observed in the TGA analysis of the samples after 900˚C. Optimum temperature for calcination of was chosen above 800˚C in earlier works reported by Birla *et al.*, [30] and Mosaddegh and Hassankhani [24]. Again, in a study by Viriya-empikul *et al.*, [13] [16] a temperature of 800˚C was selected as the suitable temperature for the calcination of waste mollusks shells to obtain CaO; Wei *et al.*, [12] used temperatures above 800˚C to produce CaO from eggshells while Nakatani *et al.*; [14] it also reported 700˚C and above for the transformation of oyster shells to CaO.

## 3.3. Phase Transformation of Calcined GFEs by X-Ray Diffraction (XRD) Analysis

XRD patterns gave information on the crystalline phases in the samples. The XRD patterns of the calcined and untreated GFEs are presented in **Figure 2**. The untreated sample and sample treated at 700˚C temperature showed similar peaks which are characteristic of crystalline phases of $CaCO_3$ and corresponds to $CaCO_3$ phases as reported by Jazie *et al.* [31] and Tangboriboon *et al.* [32]. This data indicated that a temperature of 700˚C and hold time of 2 hours in the furnace was not sufficient to decompose $CaCO_3$. The samples calcined at 800˚C, 900˚C and 1000˚C for hold time of 2 hours also had similar peak patterns corresponding mainly to $Ca(OH)_2$ phases mixed with some $CaCO_3$ phases. This can be attributed
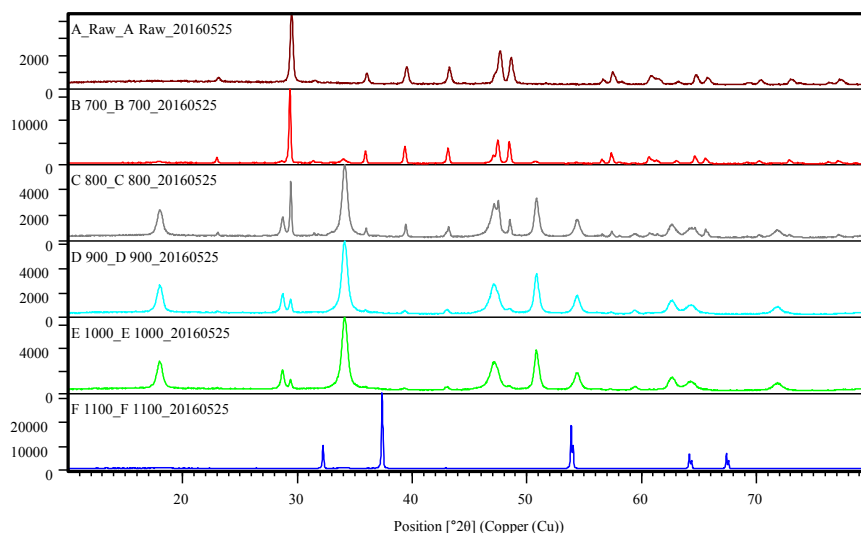
**Figure 2.** Stacked plot of XRD spectra of raw and calcined GFESs, (treated in the temperature range of 700°C, 800°C, 900°C, 1000°C and 1100°C respectively).

to phase transformation of calcite to lime. However, lime could have absorbed atmospheric moisture resulting in the formation of $Ca(OH)_2$. The major peaks associated with $CaCO_3$ reduced in intensity at elevated temperatures from 800°C to 1000°C. At 1100°C calcite peaks were absent; sharp peaks of CaO phases were visible at $2\theta$ values of 32.23°C, 37.37°C, 53.87°C, 64.37°C and 67.60°C, which corresponded to the (111), (200), (220), (311) and (222) planes. In previous studies by Cree and Rutter [5] formation of CaO started at about 500°C. However, concentrations below 1% are not detectable in XRD due to detection limits of the equipment but became visible when concentration was amplified at higher temperature. The fore mentioned could be a probable explanation for the absence of CaO phases in the XRD patterns for samples below 1100°C. The crystallite size of the CaO produced at 1100°C was calculated using the Scherer equation. The GFEs produced CaO with average crystallite size of 50.68 nm, along the (2 0 0) plane.

### 3.4. FT-IR Analysis of GFE-CaO

Fourier transform spectra were used to confirm the functional groups present in the GFE-CaO (Figure 3). The uncalcined GFEs exhibited medium absorption peak at frequency band of 1415 cm$^{-1}$, one broad band at 1082 cm$^{-1}$ and two sharp peaks at 873 and 712 cm$^{-1}$. These peaks are associated with vibration bands for carbonate $CO_3^{2-}$ ions. The sharp peaks observed at about 3641 cm$^{-1}$ in the FT-IR spectra are characteristic of –OH groups of $Ca(OH)_2$ formed from absorption of water by CaO. This absorption band was observed in all the calcined samples. The intensities of the characteristic peaks related to $CaCO_3$ at 1406 cm$^{-1}$ and 1052 cm$^{-1}$ decreased as calcination temperature increased, since peak size is linked with concentration of the compound. It implied that the amount of $CaCO_3$ reduced as it transformed into CaO. Engin *et al.* [33] detected
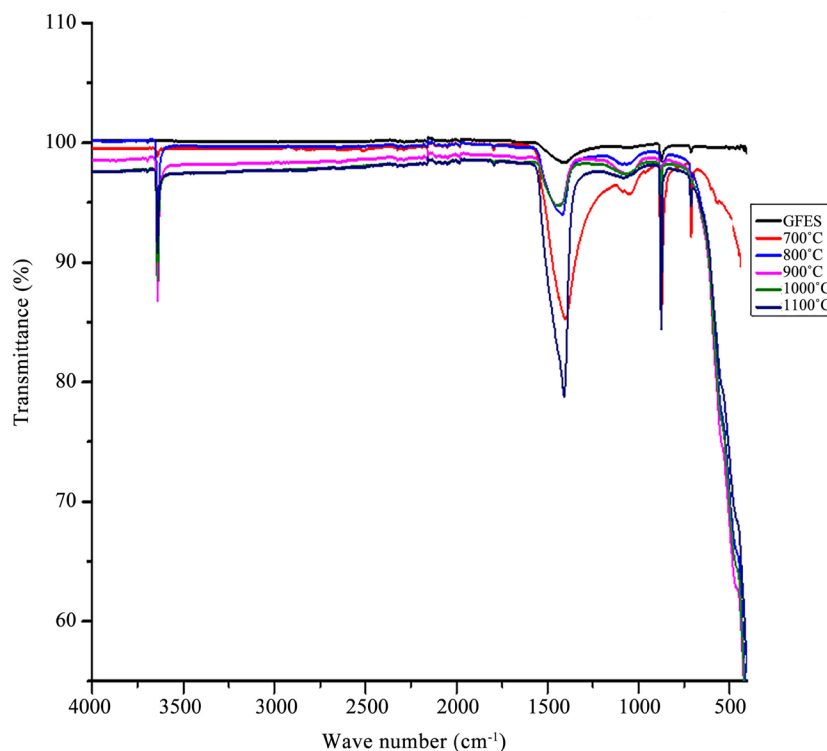
**Figure 3.** FTIR of uncalcined GFEs and calcined GFEs (treated in the temperature range of 700˚C, 800˚C, 900˚C, 1000˚C and 1100˚C respectively).

that less intense peaks of –C-O groups were observed as temperature increased. Also similar trends were reported in previous studies by Cree and Rutter [5], and Jazie *et al.*, [31] who attributed the shift in absorption bands to higher energy to the reduction in $CaCO_3$ during decomposition.

## 3.5. Elemental Composition of GFES-CaO by XRF Analysis

The elemental composition of the samples was determined by XRF data in Table 2. Calcium was identified as the major element is the samples, with about 47.91% Ca in the uncalcined GFEs and this increased to about 61.86% after the removal of organic materials at 700˚C. Moradi *et al.* [34] found about 50.85% Ca in XRF analysis of calcined mussel shells. Commercial CaO and $CaCO_3$ in this study had lower amounts of calcium compared with the calcined GFEs in Table 2. The GFEs samples also contained trace amounts of Ba, Fe, K, S and Sr. However, Ba, K and S concentrations were below detectable limits in commercial CaO sample. Oxides of elements like Ba and Sr have been used to enhance the catalytic activity of CaO in literature. However, the levels recorded in this study are negligible compared to Ca and may not have impact on the CaO produced.

## 3.6. Surface Morphology and Elemental Composition of GFEs CaO Studied by SEM and EDX

The morphology of the GFE-CaO particles was observed by Scanning Electron Microscopy (SEM).

The images showed irregular clusters of spherical particles for both samples calcined at 900˚C and 1100˚C at high magnification (10,000×) (**Figure 4(a)** and **Figure 4(b)**) and low magnification (2000×) ((**Figure 4(c)** and **Figure 4(d)**) respectively. The resulting spectra from the inspection fields of the energy dispersive X-ray spectroscopy (EDX) (**Figure 5(a)** and **Figure 5(b)**) suggest that the calcined samples were mainly calcium and oxygen as indicated in **Table 3**. According to Viriya-empikul *et al.,* [13] the surface area and calcium oxide content of eggshells are the key parameters that specifically affect the catalytic activity. This implies that the GFE-CaO will have high catalytic activity since the surface composition is mainly calcium and oxygen.



(a)

(b)

(c)

(d)
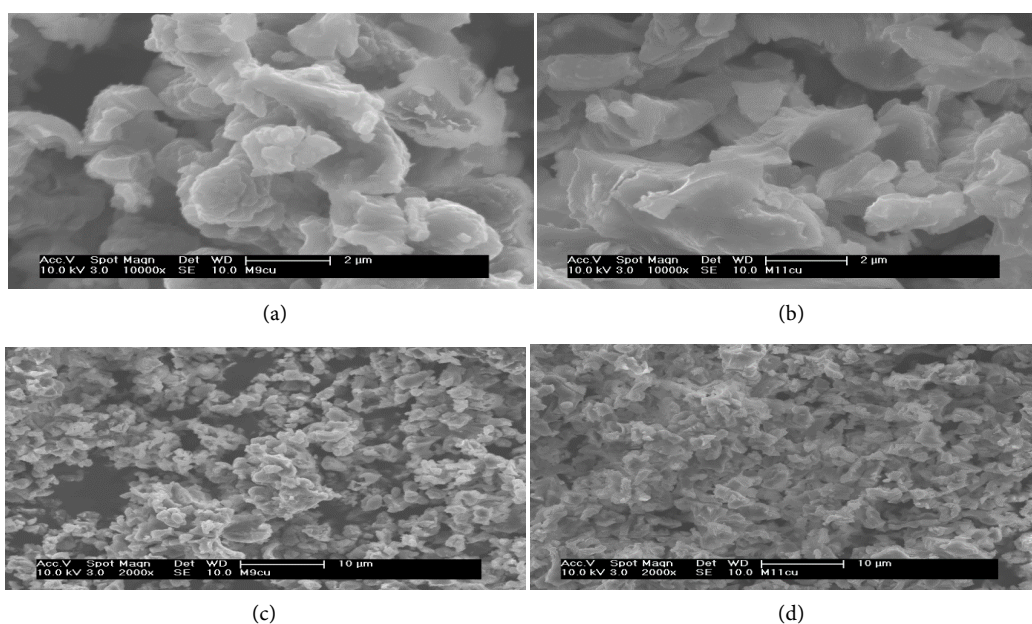
**Figure 4.** SEM and EDX of GFEs samples calcined at 900˚C and 1100˚C. (a) SEM of sample treated at 900˚C (magnification of 10,000×, 2 μm); (b) SEM of sample treated at 1100˚C (magnification of 10,000×, 2 μm); (c) SEM of sample treated at 900˚C (magnification of 2000×, 10 μm); (d) SEM of sample treated at 1100˚C (magnification of 2000×, 10 μm).
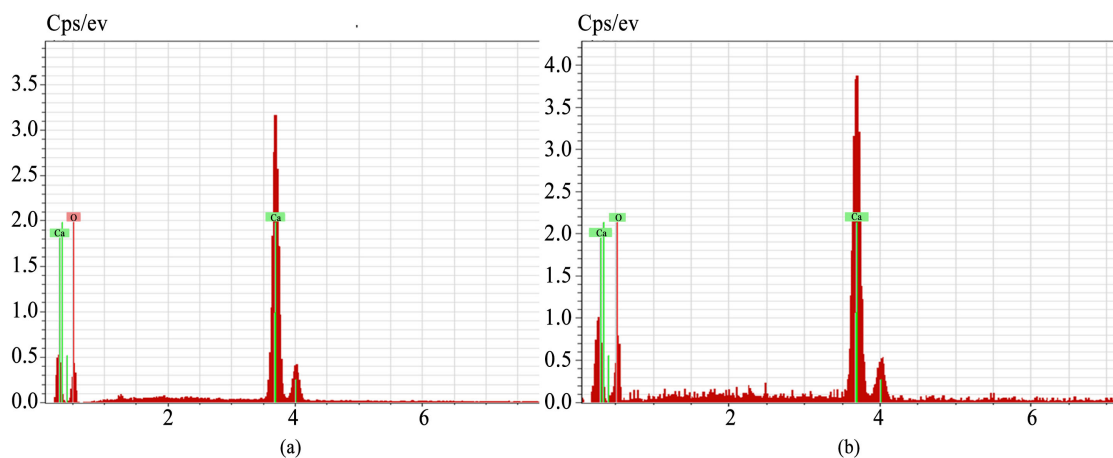


**Figure 5.** (a) EDX of sample treated at 900˚C; (b) EDX of sample treated at 1100˚C.

**Table 2.** Elemental composition of uncalcined and calcined GFESs form XRF analyses.

| SAMPLE | Ba (% w/w) | Ca (% w/w) | Fe (% w/w) | K (% w/w) | S (% w/w) | Sr (% w/w) |
|---|---|---|---|---|---|---|
| Commercial CaO | <LOD | 60.35 ± 0.38 | 0.07 ± 0.04 | <LOD | <LOD | 0.02 ± 0.00 |
| UGFEs | 0.10 ± 0.00 | 47.91 ± 1.91 | 0.04 ± 0.01 | 0.18 ± 0.01 | 0.76 ± 0.03 | 0.13 ± 0.01 |
| 700˚C | 0.12 ± 0.01 | 61.86 ± 1.32 | 0.05 ± 0.01 | 0.10 ± 0.01 | 0.15 ± 0.01 | 0.21 ± 0.00 |
| 800˚C | 0.19 ± 0.01 | 70.83 ± 1.86 | 0.05 ± 0.02 | 0.11 ± 0.02 | 0.15 ± 0.02 | 0.32 ± 0.02 |
| 900˚C | 0.19 ± 0.02 | 71.04 ± 0.96 | 0.06 ± 0.02 | 0.08 ± 0.02 | 0.15 ± 0.03 | 0.31 ± 0.01 |
| 1000˚C | 0.19 ± 0.00 | 73.70 ± 0.73 | 0.04 ± 0.02 | 0.06 ± 0.02 | 0.14 ± 0.05 | 0.34 ± 0.01 |
| 1100˚C | 0.20 ± 0.00 | 71.65 ± 0.67 | 0.06 ± 0.03 | 0.06 ± 0.03 | 0.16 ± 0.02 | 0.34 ± 0.01 |
| Commercial CaCO₃ | <LOD | 55.48 ± 0.96 | 0.07 ± 0.04 | 0.04 ± 0.00 | 0.15 ± 0.02 | 0.03 ± 0.00 |

UGFEs: unclaimed guinea fowl egg shells, LOD: lower than detectable concentration.

**Table 3.** Surface composition of GFE-Cao synthesized at 900˚C and 1100˚C by EDX.

| Sample | Element | Weight (%) |
|---|---|---|
| 900˚C | calcium | 55.50 |
| 900˚C | oxygen | 44.50 |
| Total | | 100 |
| 1100˚C | calcium | 58.64 |
| 1100˚C | oxygen | 41.36 |
| Total | | 100 |

## 4. Conclusion

In this study, the most suitable temperature for the conversion of waste guinea fowl eggshells (GFEs) to calcium oxide (CaO) was investigated. The characterization techniques employed confirmed that GFEs were mainly calcite. Both FTIR and XRF data indicated the presence of CaO in all the calcined GFE samples. However, CaO crystallite phases were more prominent in the XRD analysis of the sample calcined at 1100˚C. SEM showed bulky particles, which were more regular in the sample calcined at 1100˚C. EDX data also confirmed the surface elemental composition of the calcined GFE samples at 900˚C and 1100˚C to be 100% calcium and oxygen. Therefore, at temperatures above 900˚C and a hold time of 2 hours GFEs were successfully converted to pure CaO. The results suggest that GFEs are promising sources of pure bio-based $CaCO_3$ for CaO production with potential chemical and industrial applications.

## Acknowledgements

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