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CHARACTERIZATION OF WHEAT HUSK ASH AND CALCINED EGGSHELL AS POTENTIAL GLASS FORMER

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***Abstract*—Numerous agricultural byproducts, such as rice husk and straw, bagasse from sugar cane, palm kernel shell, wheat husk and straw, corn cobs, etc, are highly desired for the production of renewable energy and are seen as potential raw materials for high-value products. Because they can be used to extract quality silica and Calcium oxide for borosilicate glass production, this research has demonstrated that these wastes have a significant end value. X-ray diffraction (XRD) spectroscopy, Fourier transform infrared spectroscopy (FT-IR), and X-ray fluorescence spectroscopy (XRF) were used to characterize the calcined waste eggshell and wheat husk ash for crystal type, compound identification, and chemical composition. The findings demonstrated that the amount of silica and calcium oxide obtained from agricultural waste could be a suitable alternative source for making glass, with calcined eggshells having a calcium oxide content of 91.7% and wheat husk ash having a silica content of 71.3%. The potential for utilizing the CaO and amorphous silica in the formation of glass is thus intriguing.**

Keywords— Borosilicate, Wheat husk, Calcination, Leaching, Agrowaste, Quartz.

I. INTRODUCTION

Glasses are a special category of materials containing essential elements like network formers, modifiers, and intermediates, giving each type of glass its unique properties. Glasses with a silica structure have long been used in places like breweries, bottling plants, window panes, cars, buildings, etc. Since silicate glasses have extraordinary physicochemical properties and can be used in optical components like fiber optics[1], lamp envelopes, mirror lenses, prisms, and selective absorption at specific wavelengths are just a few of the technological uses of borosilicate (BS) glasses [2]. They have recently drawn significant attention for their potential use in a variety of industrial fields. These applications result from the materials' high purity, mechanical strength, thermal stability, and resistance to chemical attack [3], as well as their cost-effectiveness, wide range of high optical transparency, high bond strength, and small cation extent [4]. Industrialization and diversification are crucial when taking into account the need for glass raw materials in the nation, especially in the regional markets. A significant consumer of glass products currently meets their needs through imports due to a limited supply [5]. This practice restricts the production schedule of businesses that use glass products as feedstock in their manufacturing, which leads to high production costs for consumers to purchase such products. Agricultural waste has the potential to replace imported chemicals very effectively while also reducing the environmental damage brought on by careless waste disposal [6]. Due to the easy availability of key raw materials used in production, the high market demand for these products has been easily met. Several useful materials have been made from agricultural wastes to alleviate the difficulties associated with their disposal [7]. However, if new applications are found for these wastes, there may be a chance for a society that is more biodegradable.

A useful inorganic chemical compound with many uses is silicon dioxide, also known as silica (SiO_2). It is present in large quantities in the crust of the earth and is a naturally occurring form of quartz sand that can be amorphous, crystalline, or gel. Agricultural waste products such as palm kernel shell ash [8], wheat husk ash (WHA) [9], sugar cane bagasse ash [10], rice husk ash [11], etc. also contain silica. Due to its high silica content derived from the earth during growth, wheat husk (WH) has recently been used as a novel silica source among wastes. The majority of this waste could be turned into a new source of silica for this country. Silica-rich wheat husk ash is produced when the organic material in WH burns in the air and decomposes [12]. Due to silica content, wheat husk ash is valuable. The only renewable source that is frequently mentioned in the literature for producing value-added silicon materials is rice husk ash. [13]. Silicon dioxide can be extracted properly through leaching. Leaching refers to the procedure of removing a substance from a solid using a liquid extraction medium. The desired element diffuses into the solvent while it is still in its natural solid state [11,5]. Temperature, contact time, and solvent choice are three crucial variables in the leaching process.

Mass transfer and solubility can be improved by adjusting the temperature.

Calcium oxide (CaO) is an amorphous substance that is used as a glass modifier and comes in crystalline or powdery solid form. Li, Na, K, Ca, and Mg are "alkali or alkali-earth cations" that can be used to modify silicate glasses to change their properties in a variety of applications, including laser optics for starting fusion reactions, and medical and dental implants [14]. CaO can be made using either natural or inorganic raw materials as the starting point [15]. Many scientists have investigated various inexpensive natural biological sources, including plants, fish bones, seashells, eggshells [16], etc. About 98% of eggshell is calcium carbonate, with traces of phosphorus and magnesium [17]. In order to prepare calcium oxide, chicken eggshell offers a desirable and affordable calcium source. The calcination procedure can transform $CaCO_3$ into calcium oxide (CaO) as shown in (1).

$$CaCO_3 \rightarrow CaO + CO_2 \quad (1)$$

CaO, which was the active phase in the eggshells, required a temperature above 800ºC for the calcination process to take place [16,17,18].

The main goal of this research is to synthesize calcium oxide from eggshells and bio-silica from wheat husks, as well as examine the characteristics of calcined eggshells and wheat husk ash for the production of borosilicate glass. This study's methodology is cost-effective, environmentally friendly, and it offers the chance to use agro-waste for technological applications.

II. MATERIALS AND METHODS

A. Material Collection

The wheat husk used to make bio-silicate was obtained from a farm in Maiduguri, Nigeria, and the chicken eggshells waste from restaurants in Suleja, Nigeria.

B. Preparation of sample

The used chicken eggshells were washed to get rid of any dirt or other debris that had adhered to the shell's surface. After removing the internal white membrane, the shell underwent a thorough distilled water rinse. then dried for three hours at 105°C in the oven to remove any remaining water from the eggshell. The dried eggshells were blended until smooth. The eggshell was heated in a furnace at 800, 900, and 1000ºC for 5 hours. In order to produce pure CaO compounds and remove impurities associated with the chicken eggshell, high temperatures are used during the calcination process.

The wheat husk was cleaned thoroughly to remove all remaining trash and extra-biological material. 1.0 M hydrochloric acid (HCl) was used to prepare the wheat husk, the mixture was then heated for 2 hours at 100ºC for about an hour. Following a distilled water rinse to remove any remaining hydrochloric acid, the leached wheat husk was then dried for 3 hours at 105°C. Finally, the dried wheat husk was burned for 5 hours at a temperature of 700 °C in an alumina crucible to produce a pure wheat husk ash (Bio-silicate). These outcomes will be used later for the production of borosilicate glass.

C. Characterization Method

1) X-Ray Fluorescence (XRF) analysis

XRF model of "PanAlytical Epsilon 4" was used to determine the percentage of CaO in the sample by analyzing the elemental composition of calcined eggshells and wheat husk ash. Using an agate pulverizing device (planetary micro mill pulverisette 7), the sample was ground into powder. A 150μ mesh sieve press was used to press the sample. This was done to guarantee the samples' homogeneity. The outcomes are given in Tables I and II.

2) X-ray Diffraction characterization

Phase identification and quantification, as well as calculating the percentage of crystallinity of calcined chicken eggshell and wheat husk ash, were done using the Rigaku Miniflex XRD apparatus. The X-ray diffractometer has an acceleration voltage of 30 kV (fixed), a current of 15 mA (fixed), a scan range of $2\theta = 2^o - 145^o$, and a scan speed of 0.01-100 º/min. The X-ray source is a copper anode (**λ** Cu Kα, radiation = 1.5406Å). The diffraction patterns were analyzed using the Joint Committee on Powder Diffraction Standards (JCPDS).

3) Fourier Transform-Infrared (FTIR) Characterization

Agilent Technology CARY 630 Fourier Transform-Infrared Spectrometer (FTIR) from Umar Musa Yar'dua University (UMYU) Katsina, Nigeria, was used to identify the functional groups in the WHA and the calcined chicken eggshell samples. Each sample was finely powdered for sample preparation. Figure 2 (a) and (b) show the FTIR spectra of the wheat husk ash and calcined eggshells, respectively.

III. RESULTS AND DISCUSSIONS

A. Elemental Composition Of WHA And Calcinated Eggshells

XRF analysis revealed that the CaO content in calcined eggshells increased from 73.3% to 91.7% from raw eggshells. According to this finding, $CaCO_3$ had roughly converted to CaO. Additional chemical compositions found in the calcined eggshell were shown in Table I at different temperature as well. Since $CaCO_3$ predominated in the eggshell, CaO was the element that was present in the highest concentration. According to Prayitno et al. [19] and Razali et al. [20], the same outcome was reported. Calcined eggshell contained 91.7% CaO, indicating that a rough conversion of the eggshell to CaO had taken place. The eggshell may be a naturally occurring carbonate-based material despite trace amounts of other compositions present [16].

Table II provides the analysis results of wheat husk ash chemical composition. WHA was found to have a 71.3% silica content. At 700 ºC, the WHA in this study had a higher SiO_2 content than the silica (49.17%) found in Terzioglu et al. [23] and (50–55%) Patel et al [21]. Different grades of silica with various yields can be obtained based on the temperature and length of combustions in various treatment temperatures. The quantity of silica reported in this study is consistent with the findings of Hamza et al., [22] who used rice husk as a source of silica for the synthesis of bio-silicate borotellurite (BSBT) glass, Since so much silica was successfully extracted from WH, this points to the material's tremendous potential as a source of silica precursor for glass production.

TABLE I. CHEMICAL COMPOSITION OF RAW AND CALCINED EGGSHELLS

Chemical composition	*Raw chicken eggshells (%)*	Calcined chicken eggshell (%)		
		800 °C (%)	*900 °C (%)*	*1000 °C (%)*
MgO	0.0	0.3	7.6	0.0
Al_2O_3	0.1	0.1	0.1	0.2
SiO_2	0.3	0.4	0.3	0.4
P_2O_3	0.7	0.5	0.6	0.6
CaO	73.3	79.1	91.3	82.9
CuO	0.02	0.03	0.0	0.4

TABLE II. CHEMICAL COMPOSITIONS OF WHEAT HUSK ASH (WT %)

Chemical composition	Weight (%)
SiO_2	71.3
Fe_2O_3	6.5
Al_2O_3	9.7
P_2O_5	6.3
CaO	1.0
K_2O	2.2
TiO_2	1.7
Others	3.6

B. *The X-Ray Powder Diffraction (XRD) Result Of WHA And Calcined Eggshell.*

X-ray diffraction (XRD) patterns for WHA and calcined chicken eggshell samples and the standard JCPDS reference were recorded using the Rigaku Miniflex diffractometer in Figure1a and 1b. It can be inferred from the WHA spectrum in Figure 1(b) that WHA exhibits some amorphous characteristics despite having some degree of crystallinity [23]. The WHA used in this study contains quartz and in accordance with Joint Committee on Powdered Standard (JCPDS) data number (00-003-0419). The X-ray powder diffraction (XRD) pattern of the spectrum also displayed a high intensity peak (Figure 1b), while the low intensity peaks can be attributed to additional oxides found in the WHA [22,23] between 26° and 30°, an amorphous hump was seen; this might be because amorphous glassy material is present. A high intensity peak was seen at 26.9° that corresponds to the silicates present in WHA [24]. X-ray diffraction phase analysis reveals that the predominant form of quartz (SiO_2), kaolinite (K) and Muscovite (M) (00-002-0467) in WHA exist as crystalline.

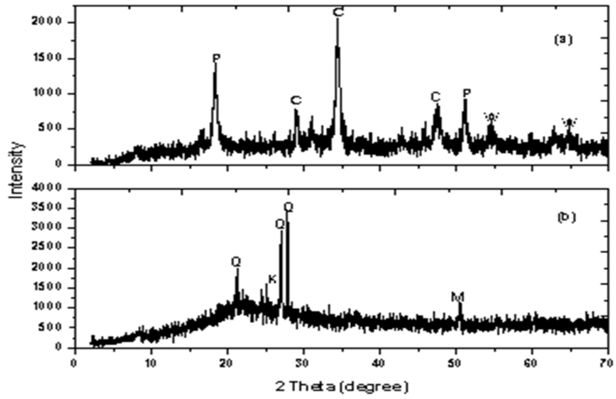

Fig. 1. XRD pattern of wheat husk ash and calcinated eggshells

The eggshell was calcined at 900 °C for 5 hours and is depicted in Figure 1a as an X-ray diffraction spectrum. At 2ϴ =34.37, the principal peak became visible. Additionally, the spectrum of this substance exhibits a number of peaks at 2ϴ = 18.31, 28.98, 34.37, 47.32, 50.99, 54.69, and 63.02. Calcium hydroxide ($Ca(OH)_2$ portlandite) with JCPDS data number (04-014-7723) is produced by the longer calcination time at 900 °C for 5 hours as a result of a reactive moisture adsorption [16,24]. Also, the phase transformation is dependent on the calcination temperature and time. Wollastonite (W), calcite (C), and portlandite (P) are all indicated by the main peak of the XRD phase analysis.

C. Compound Identification

The functional group of various parameters in the calcined eggshells and wheat husk ash is identified using FT-IR. In Figure 2a, it was observed that the characteristic peaks at 1058cm^{-1} was ascribed to the asymmetric stretching of O-Si-O [26,27] and Si-OH stretching vibration of silanol group at around 782 cm^{-1} consistent with the result obtained by Tezgiolu et al.[27], Tezgiolu et al.[23] and Gouran et al.[22]. No broad band is seen at 3300 cm^{-1}, which is assigned to O-H and denotes the presence of water. These demonstrate that the combustion process was successful. These findings highlight the prospect of wheat husk as a silica source.

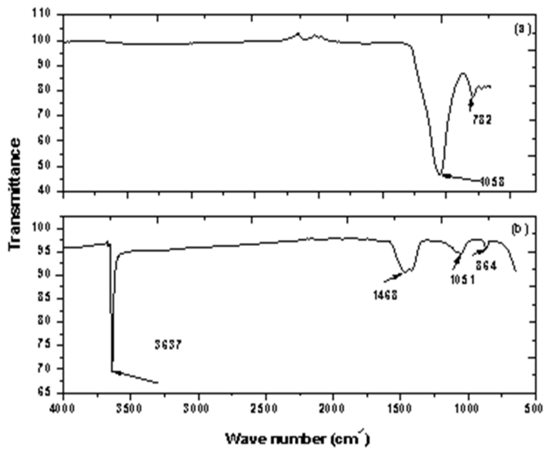

Fig. 2. FTIR spectra of (a) wheat husk ash and (b) calcined eggshells

IR spectra of calcined eggshells show distinctive bands that demonstrate the presence of OH in $Ca(OH)_2$ as shown in Figure 2b, peak at about 3600 cm^{-1}. The presence of $Ca(OH)_2$ in the calcined sample may have resulted to CaO absorbed moisture when packing and analysis [27,28]. The inorganic compound's CO_3^{2-} functional group at 1468 cm^{-1} and 1058 cm^{-1} indicated that it has not yet completely broken down into its most basic form [20], and 864 cm^{-1} (Ca–O) consistent with the result obtained by Goh et al. [16]. The transformation of $CaCO_3$ into CaO in the calcined eggshell can be used as a modifier in the production of glass.

IV. CONCLUSION

The ash from wheat husk and calcined eggshell was found to contain, respectively, 71.7% SiO_2 and 91.7% CaO using XRF spectroscopy. The XRD analysis of WHA indicates amorphous characteristics due to the broad spectrum observed and some degree of crystallinity due to the additional oxides found in the WHA. It also reveals the predominant form of quartz (SiO_2) among the phases. $CaCO_3$ had converted to CaO, as shown by the calcined eggshell's XRD and FTIR results. The results from this agricultural waste allow for potential use in the production of borosilicate glass.

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