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Green Synthesis of Zinc Oxide and Composite Zinc Oxide Nanoparticles Using Cassava Leaves Extract

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ABSTRACT

The demand for commercial nanoparticles are on the increase due to their wider applications in many fields, the biological synthesis provides a cost-effective, more stable materials using eco-environmental friendly routes to reduce health risk and hazardous waste into the environment. Nanoparticles are products of a rapidly growing technology which is widely applied in many fields. The use of toxic organic solvents and strong reducing agents are not environmental friendly which results in producing hazardous wastes that poses serious threat on the environment. The aim of this study is to synthesize zinc oxide nanoparticles (ZnO NPs) and composite ZnO nanoparticles (CZnO NPs) using cassava leaf extract. Chemical precipitation method was used to synthesize ZnO NPs and CZnO NPs. Both nanoparticle samples were characterized using analytical techniques such as Fourier Transform Infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF) and Thermogravimetric analysis (TGA). The XRD results revealed the 6 mineral phases in cassava leave, 7 mineral phases in composite ZnO NPs and 7 mineral phases in ZnO NPs. SEM morphology revealed large, rough, irregular shaped particle sizes which looks like particle agglomeration. The FTIR results showed the broad band at 3276.33 cm^{-1}, 2046.31 cm^{-1}, 1606.48 cm^{-1}, 1158.2 cm^{-1}, 868.47 cm^{-1} stretching which indicate O-H, C=O, C-O, and ZnO stretch respectively. The TGA results showed thermal stability of 100 g of ZnO samples up to over 300^{o}C before decreasing. In conclusion, the ZnO NPs and its composite ZnO NPs were successfully synthesized and exhibited close similar qualities.

Introduction

Green synthesis are usually eco-friendly in recent years, we have seen improvements in the field that deals with the nature, composition, analysis, and use of nanoparticles [1]; [2]. Nanoparticles are small particles that ranges between 1 and 100 nanometers in size. Nanoparticles can occur naturally, be created as the by-product of combustion reactions or be produced to perform special functions [3]. Due to their size they deal with materials with atypical physical, chemical and biological properties that occur at a nanoscale in contrast to their bulk materials such as large surface area [4], high electrical and thermal conductivity and high tensile strength which has led to the application in various fields such as medical sciences, electronics and devices, food industries, textile and fabrics and in the environment [1]; [5]; [6]. ZnO is a well-known n-type semiconductor in materials science and as a result of defects such as oxygen vacancies and zinc interstitials [7]. Semiconductor compounds [8]; [9]. It has a broad band gap of 3.37 eV, high bond strength, with a high binding energy (60 meV) at room temperature. Zinc oxide exhibit some chemical properties such as Zinc oxide can be found as mineral zincite, a white powder and if it contains manganese impurities, it will change from orange or red in colour [10]. Crystalline zinc oxide is thermochromic in nature, and changes from white to yellow when heated and back to white when cooled, the colour change is due to a slight loss of oxygen at high temperatures [11]. Because of its electrical, optical, and structural characteristics, ZnO has attracted substantial interest as a wide band gap semiconductor in many fields of research and in the development of lasers as well as solid state blue to ultraviolet optoelectronics. ZnO breaks down into zinc vapor and oxygen, exhibiting high stability and when heated in the presence of carbon, it forms more volatile Zinc. When ZnO is exposed to air, it absorbs water vapor and carbon dioxide to form zinc carbonate which can be attributed to its amphoteric nature. The ZnO interacts with both acids and alkalis [12]; [13]. The benefits of ZnO are that it has a large band gap with higher voltage breakdown, to maintain high electric fields, high temperature and high power operations [14].

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nanoZnO used in electronics and optoelectronics will be enhanced by successful doping of p-type semiconductor [15]. ZnO is transparent when it comes to visible light, and it absorbs ultraviolet light < 3655 Å. ZnO appears white in the range of visible wavelength but titanium dioxide rutile and anatase have a reactive index and higher degree of opacity. ZnO can be synthesized physical, chemical and biological methods. Physical methods of synthesizing ZnO nanoparticles are called top-down technique which is expensive and time-consuming. Physical methods are: Laser ablation: [16]; [17]; [18]. ball milling [19]; [20], spray pyrolysis (Al- [21]; [22], vapour phase [23], ion implantation [24], mechanical grinding, and electron beam lithography [25]. Chemical method is the bottom-up technique and it is not expensive for synthesizing nanoparticles at a high temperature. ZnO materials can be synthesized in different particle sizes and shapes using the following chemical methods such as: sonochemical [25]; [26]; [27], Precipitation method [4] precipitation [28]; [29], solvothermal [30]. Chemical reduction method [16], Sol-Gel [31], ultrasonic [32]; [29], micro-emulsion [33], microwave [34] [35]. Biological method of synthesizing ZnO NPs has gained a lot of attention because it is more environmentally friendly and economical due to the absence of harmful chemicals or large amounts of energy used [9]. Green synthesis is a biological technique which involves the use of microorganisms such as fungi, yeast, plant extracts or the usage of templates such as DNA, membranes, and viruses to replace chemical solvents and stabilizers to decrease the toxicity of both product and process [29]. ZnO NPs can cause toxicity and other negative effects such as fibrosis, oxidative damage, inflammation and the emission of pro-inflammatory mediators could arise [16]. The aim of this research is to synthesize zinc oxide and composite zinc oxide nanoparticles using cassava leaf extract.

2. Materials and Method

2.1. Study Area

Ojo is a town and a Local Government Area (LGA) in Lagos State, Nigeria. It is located on the eastern axis of the Trans–West African Coastal Highway, about 37 km west of Lagos. It is part of the Lagos Metropolitan Area located at coordinates: 6°28′N 3°11′E, Population (2006 census), Total 609,173, Estimate (2016) 838,900, Density, 8,700/Sq mi (3,300/km^2). Ojo is primarily a residential township, although, it has some major markets such as Alaba International market, Alaba livestock market (Alaba Rago), the old Lagos International Trade Fair complex, Ojo market and Iyana-Iba market. It also houses the divisional headquarters of 81 division Nigerian Army and Navy Town. South of the town (across Badagry creek), the rest of the local government is sparsely populated and consists of mangrove swamps and sandy beaches. Some of these beaches are holiday spots in the festive season. Wildlife mostly consists of reptiles, rodents and birds including crocodiles, iguanas, monitor lizards and squirrels. Whales and dolphins have been known to visit the coastal areas.

2.2. Samples and Sampling

The samples were purchased from a local market and chemical store in Lagos State at Ojo local government area. The cassava leaves were collected from a farm inside Lagos state university, Ojo Campus, Lagos State.

2.3. Chemicals/Apparatus Used

The apparatus are: beakers, measuring cylinder, analytical weighing balance, filter paper, funnel, conical flask, watch glass, water bath, pH meter, magnetic stirrer, oven, thermometer, mortar and pestle. Chemicals used are: Zinc Chloride ($ZnCl_2$), NaOH., Ethanol, Distilled water, Cassava leaf extract

Figure 1: Map of Ojo Area, Lagos State.

2.4. Synthesis of Nanoparticles

2.4.1 Reagent Grade Synthesis of ZnO Nanoparticles

27.3 g of $ZnCl_2$ was dissolved in 100 mL of distilled water under vigorous stirring at room temperature and 5 M of NaOH solution was added drop wise into the $ZnCl_2$ solution until it attains a pH of 12 to form a pale white precipitate solution. The mixture was stirred on a magnetic stirrer for 2 hours and later filtered to obtain a pale white precipitate. The precipitate was washed repeatedly with distilled water followed by ethanol, filtered and dried in the oven. The dried nanoparticle was cooled at room temperature and grinded with mortar and pestle into fine particles.

2.4.2 Cassava Leaf Extract Synthesis of Composite ZnO Nanoparticles

27.3 g of $ZnCl_2$ was dissolved in 100 mL of distilled water under constant stirring at room temperature. 20 mL of cassava leaf extract as added into the $ZnCl_2$ solution and heated up to 50 °C, 50 mL of 5 M NaOH was added drop wise into the solution and stirred continuously for 2 hours on a magnetic stirrer turn to a pale yellow precipitate. The precipitate was filtered and washed repeatedly with distilled water followed by ethanol, filtered and dried in the oven. The dried nanoparticle was cooled at room temperature and grinded with mortar and pestle into fine particles.

$ZnCl_2 + H_2O \longrightarrow Zn^{2+} + Cl^-$ ……………………………1

$NaOH + H_2O \longrightarrow Na^+ + OH^-$ ……………………………2

$2Zn^{2+} + 4OH^- \longrightarrow Zn(OH)_4$ ………………………….. 3

$Zn(OH)_4^{2-} \longrightarrow ZnO\ (s) + H_2O + 2OH^-$ …………… 4

3. Results and Discussion

3.1 Characterization

The pulverized cassava leaves, synthesized zinc oxide and composite zinc oxide NPs were characterized using SEM, FTIR, XRD, TGA and XRF analysis.

3.1.1 SEM Analysis

The result of SEM in Figure 2 revealed the morphology of pulverized cassava leaf at x700 magnification were irregular shape particle sizes and the PLC and CZnO NPs revealed more agglomeration with larger particle sizes than the ZnO NPs [36]; [37].

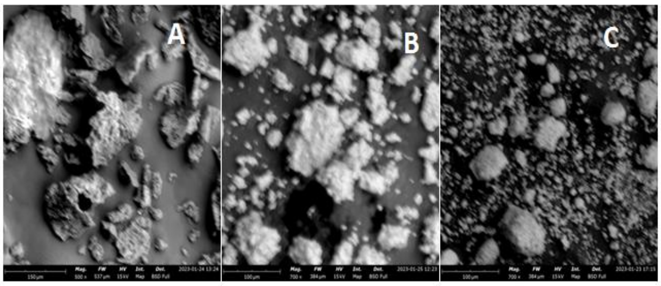

Figure 2: SEM morphology of pulverized cassava leaves (PLC) (A), composite ZnO NPs (B) and ZnO NPs (C) at x700 magnification.

3.1.2. FTIR

The FTIR analysis was used to determine the functional groups and the chemical composition of the synthesized samples measured within the range of 400–4000 cm^{-1} [38]. The results of the synthesized PCL, CZnO and ZnO NPs presented in Figure 3A revealed a broad O-H stretching band in PCL, and CZnO NPs at 3174-2860 cm-1 and 3637 cm^{-1} respectively indicates little trace of water while the sharp N-H stretching peak for ZnO NPs at 3276.33 cm^{-1}, stretching band peaks at 2046.31 cm^{-1} and 1606.48 cm^{-1} indicates CO2 and C=O stretching respectively corresponds to result reported in previous study (Wahab et al., 2007, 2008)[39], [40] while bands around 1159.2 – 1085 cm^{-1}, indicated C-O stretching [41];][42]; [43] and stretching bands identified at 868.47 cm^{-1} and 738.01 cm^{-1} indicates ZnO and CZnO NPs respectively [42]; [44]; [45].

3.1.3. TGA

Thermogravimetric analysis (TGA) was carried out to the find out the decomposition process of the materials. The results of TGA for the PCL, CZnO and ZnO materials presented in Figure 3B revealed the weight loss of all the materials over the temperature range from 100 ºC to above 900 ºC and the materials were thermally stable before the weight began to decline with increasing temperature and this trend was observed in previous studies [46].

3.1.4. XRD

Figure 4 presents the results of XRD spectrum and mineral composition of cassava leaves, ZnO and Composite ZnO nanoparticles which that it consist of 7 mineral components for CZnO cassava and ZnO NPs while the PLC contains 6. The CZnO NPs and ZnO NPs consist one different minerals are Souconite-15A and Zinc Chloride H respectively. The CZnO NPs and ZnO NPs revealed they are crystalline while the PLC was amorphous. These XRD diffraction pattern showed sharper peaks of the synthesized ZnO at reflection angle of the spectral peaks which appears at angle 2θ indexed at 11.82◦, 22.52◦, 25.18◦, 28.56◦, 56.78◦, 32.24◦, 32.34◦, 33.42◦, 38.24◦, 45.86˚, 54.34◦, 58.58◦ and CZnO NPs at 2θ of 11.8, 22.6◦, 25.22, 28.56, 32.18, 32.36, 34.1, 38.28,45.92, 54.3, 58.64 [32–34](Estrada-Urbina et al., 2018; Cao et al., 2019). The crystalline average particle size was calculated by using the Debye-Scherrer equation on the diffraction peaks for PCL, CZnO and ZnO NPs particles was found to be 1.6 nm, 6.2 nm and 8.9 nm respectively. The sharp and narrow peaks' intensity confirmed that the synthesized ZnO nanoparticles are fine grain size particles with good crystallinity and high quality. All the indexed peaks of the spectrum confirmed that the synthesized ZnO NPs are crystalline particles except PCL which was amorphous with a broad peak [47].

3.1.5. XRF Analysis

XRF elemental composition results presented in Figure 5 revealed that the PLC consist of 27.45 % SiO2, 46.71 % CaO, 1.41 % Fe2O3, 2.26 % SO3, 9.30 % while CZnO NPs consist of 24.51 % Cl and K2O 11.96 % MgO. The 24.51 % Cl revealed the addition of to the CZnO NPs Table 1: XRF elemental composition of pulverized cassava leaves (PCL), synthesized ZnO and composite ZnO NPs.

4. Conclusion

The results of this study showed that cassava leaf extract can be used to synthesize zinc oxide nanoparticle which revealed the morphology with irregular particle sizes. The XRD of the synthesized of the NPs were crystalline have long sharp peaks for CZnO NPs, and ZnO NPs while the peak of PCL was amorphous and particle size was calculated using Debye-Scherer's equation to be 1.6 nm, 6.4 nm and 8.4 nm for PCL, CZnO NPs and ZnO NPs respectively. The XRD revealed the composition of the mineral phases presents in the PCL, CZnO NPs and ZnO NPs were 6, 7 and 7 respectively. The ZnO NPs particle size was reduced from 8.4 nm to 6.4 nm when pulverized cassava leaf extract was used to synthesize the CZnO NPs. The FTIR absorption bands revealed some of the functional groups stretching of the three samples: N-H, O-H, =C-H, C=O, C-O and Zn-O at absorption bands.

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Conflicts of Interest

The finance or funding of this research is self-sponsored by the authors' collaboration and we declared that there is no conflict of interest

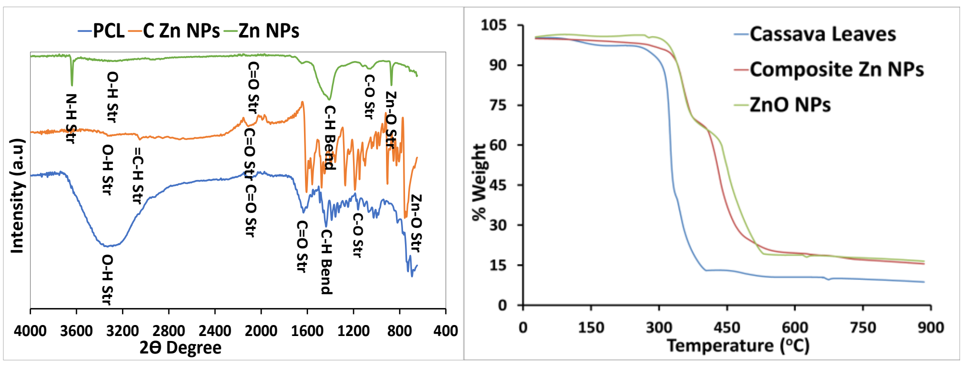

Figure 3: FTIR spectrum (A) and TGA analysis (B) of pulverized cassava leaves (PLC), synthesized ZnO (Zn NPs) and composite ZnO NPs (CZn NPs)

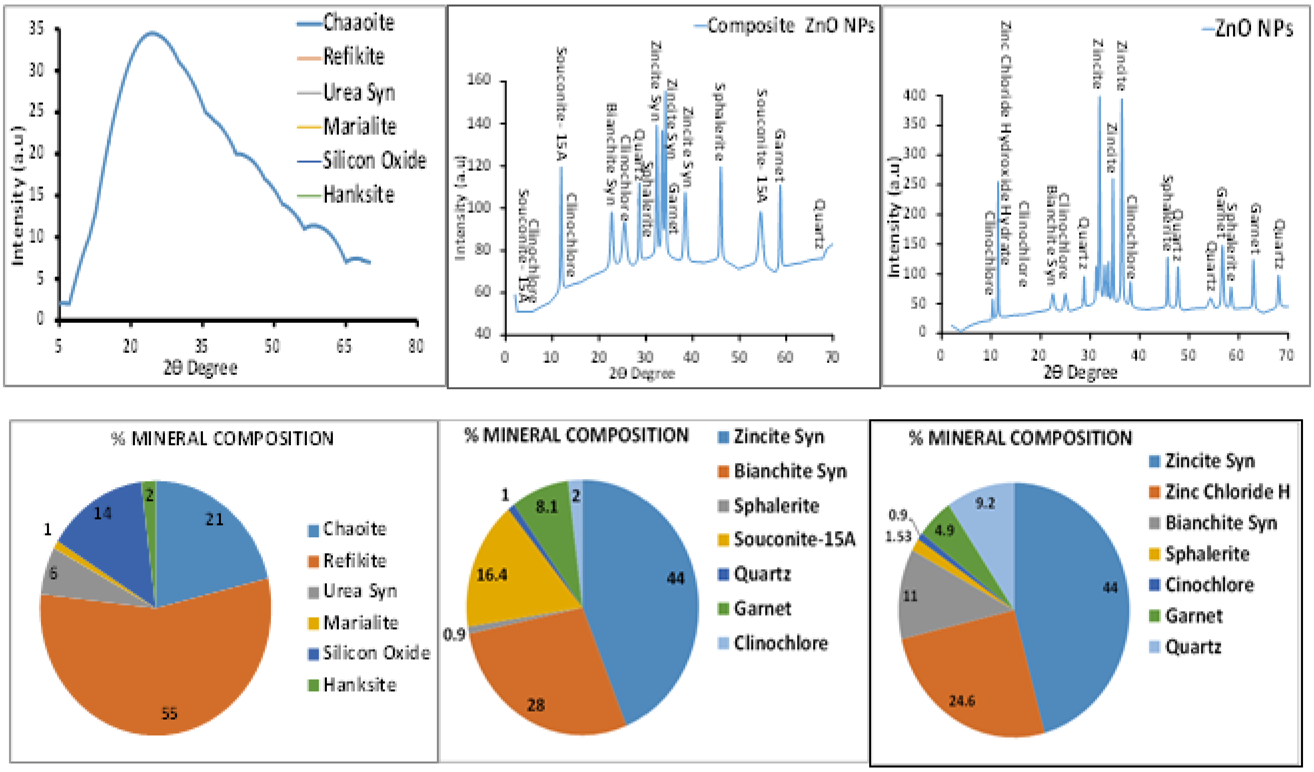

Figure 4: XRD % mineral phase composition of cassava leaves, composite Zn NPs, and Zn NPs.

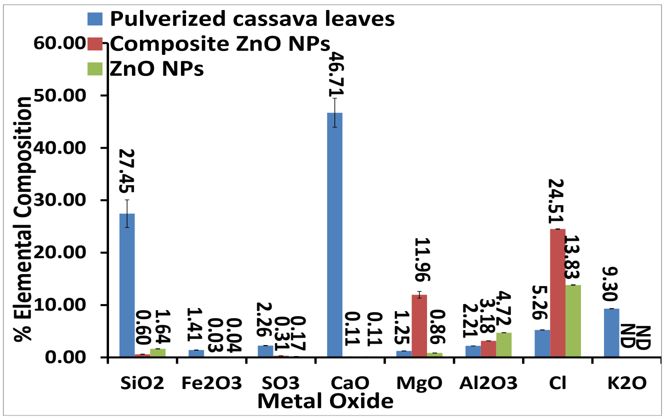

Figure 5: Elemental Composition of pulverized cassava leaves (PLC), ZnO NPs and composite ZnO NPs.

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