Green Fabrication and Characterization of Zinc Oxide Nanoparticles Using Celery Leaf Extract

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ABSTRACT

The development of novel theoretical and experimental techniques in nano-science always lead to proactive opportunity for innovative nano-scaled materials. Materials with nano-structure are known to bridge the smallest atoms and the largest molecules of bulk materials through which many enhanced properties can be attained. In an attempt to minimize environmental hazards, reducing the use of toxic non-degradable products and moving towards developing an environmentally safe material. This paper will present the synthesis of bio-based nano-sized ZnO particles prepared from celery leaf plant extract as reducing/stabilizing agent and zinc chloride metal salt as metal ion precursor that chemically altered into metal oxides nanoparticles. "Co-precipitation" method was used to synthesize the ZnO nanoparticles at room temperature. To properly characterize the synthesized ZnO nanoparticles, four different tests were conducted namely, XRD, FE-SEM, EDS and AFM. The characterization analysis for the synthesized ZnO nanoparticles indicated average particles size of 35 nm. Also, the morphology analysis indicated the formation of ZnO nanoparticles having shape of hexagonal wurtzite nanocrystals compatible with the Joint Committee on Powder Diffraction Standards (JCPDS) card no. (36-1451) for pure ZnO nanoparticles. The produced ZnO nanoparticles from green synthesis process can be directly used as an eco-friendly additive in many industrial applications.

Keywords: ZnO Nanoparticles, plant extract, green synthesis, XRD analysis, morphology analysis, AFM test.

1. Introduction

Nanotechnology is the conquering term for every revolutionary technical development. It deals with developing and managing materials at nano-scale that is one billion times smaller than a meter. The implementation of nanotechnology in real world practice comprehends the fabrication and application of materials ranging from individual molecules to submicron dimensions into many systems [1]. Nanoparticles are considered the building blocks in the production process of nano-materials [2]. The ability of nanotechnology to adapt certain changes to produce customed properties has a leading role to overcome environmental and technical challenges [3]. The unique properties of nanocrystalline metal oxides gained the attention in industrial applications like photo-catalysts, pigments, and fillers [4]. Furthermore, nanoparticles were proved to be efficient as an additive to diesel fuel that improved engine performance by lowering gas emissions such as CO, NOx and HC compounds [5]. Recent work showed that nanotechnology can be infused with green bio-based materials resulting in more environmentally friendly biodegradable nanoparticles. Plant and food waste which are biodegradable materials can be a potential substitute for many chemical additives, also from financial viewpoint, food wasters are cost-saving materials along with being more eco-friendly products [6]. In general, there are two approaches for synthesizing nanomaterials either "Top down" or "Bottom up"[7]. In the approach of top-to-bottom there are several methods applied including milling, sputtering, etching, laser ablation, etc. where in the approach of bottom-to-up, nanoparticles are built up from simpler molecules and allowing nucleation growth of particles to nanoscale, such methods include chemical vapor deposition, co-precipitation, sol-gel process, atomic/molecular condensation and laser pyrolysis [8].

In the past decade, the arising environmental concerns urged the researchers to look for innovative substitute process through which nanoparticles can by synthesized without using toxic chemicals. Biological methods have been suggested as an ecofriendly alternative to chemical and physical methods. Consequently, it paved the way for "green synthesis" of nanoparticles using enzymes, plant extracts and even microorganisms [9]. Gold and silver metal nanoparticles were among the first metals to be synthesized using plant extracts from various plants such as oat, aloe vera, coriander, lemon grass and alfalfa (flowering plant). Zinc Oxides is one of the most desirable materials as semiconducting photocatalyst owing to its cost saving production and excellent optical properties having large band gap and high excitation binding energy like UV filtering properties and high catalytic activity [10]. ZnO nanoparticles is a registered material recognized by the United State Food and Drug Administration as safe zinc component [11]. In recent work, zinc Oxide (ZnO) nanoparticles were prepared from variety of plant leaf extracts such as crown flower, green tea, copper leaf and coriander [8]. In this paper, “bottom-up” approach was utilized to synthesize ZnO nanoparticles using zinc chloride ($ZnCl_2$) salt as metal ion precursor and celery leaf extract as the reducing/stabilizing agent. “co-precipitation” method was applied for the nucleation and growth of ZnO nanoparticles. As for the characterization of the synthesized ZnO nanoparticles, four different tests were conducted which are X-ray diffraction (XRD), Field Emission Scanning Electron Microscope (FE-SEM), Energy Dispersive Spectroscopy (EDS) and Atomic Force Microscope (AFM), each test evaluated different aspect of the nanoparticles and shall be discussed thoroughly.

2. Theoretical Background

It has been showed that various biological systems including plants and algae, fungi, yeast and bacteria have the ability to transform inorganic metal ions into metal nanoparticles. This ability is attributed to the reductive capability of proteins, effective phytochemicals (terpenoids, polyphenols, sugars, alkaloids, amides, flavones and phenolic) present in plant extracts and metabolites present in biological organisms (V. V. Makarov, et. al., 2014).

Among available green methods of producing metal nanoparticles is utilizing plant extracts, plants used as biological precursors are able to reduce metal salt ions, soluble salts and have strong metal ion hyperaccumulation which can be harvest via sintering methods [13](V. V. Makarov, et. al., 2014). For instants, when using silver nitrates are substrate with mustard greens and alfalfa (flowering plant) they produced 50 nm silver nanoparticles. The process of utilizing plant extracts to produce nanoparticles is considered simple and easy compared to bacterial and fungi synthesis [13]. The hypothesis presented in literature work regarding the synthesis of metal oxide nanoparticles via plant leaves extracts encompasses three phases [8]:

- The activation process during which metal ions from salts are bio-reduced and nucleation process of reduced metal ions occur.
- The growth phase during which tiny particles combine with greater ones in spontaneous matter via a process called Ostwald ripening.
- The termination phase during which final shape of nanoparticles is defined

The nucleation and growth of the nanoparticles starts with a reaction between Zn^{2+} and hydroxide ions which is followed by the aggregation process. The general chemical reactions are as follows:

$$ZnCl_2 + 2NaOH \rightarrow Zn(OH)_2 + 2NaCl \quad (1)$$

$$Zn(OH)_2 + \text{Phytochemical from plant leaf extract} \rightarrow ZnO + H_2O \quad (2)$$

$$ZnO + H_2O + \text{drying and calcination} \rightarrow ZnO\ NPs \quad (3)$$

The previous reaction occurs in sequent steps during which the zinc hydroxide, $Zn(OH)_2$ is an intermediate product. The hydrogens are then reduced by the phytochemical components from the plant extract which upon drying, final product of nanopowder zinc oxide, ZnO is formed.

3. Methodology & Experimental Work

3.1. Celery Leaf Extract Preparation

About 2 Kg of Celery leaves were soaked for about an hour and pre-washed with tap water to remove dirt and residual undesired contaminants. The leaves were rinsed with de-ionized water several times. After that, 1.5 L of de-ionized water was added to a pot where celery leaves were transferred into. The leaves were left to simmer on a very low heat for about two and half hours. Eventually, the leaves were left to cool off and let to extract more of the biological plant components into the aqueous plant extract for about 24 hours. Lastly, the plant extract was filtered using filter cloth/fabric, conical funnel and beaker. Finally, the celery leaf extract was preserved in a glass container as shown in figure 1.

Figure 1. Celery leaf plant extract

3.2. Co-Precipitation of ZnO Bio-Based Nanoparticles

ZnO nanoparticles were synthesized chemically using co-precipitation method by reacting the metal salt $ZnCl_2$, NaOH aqueous solution and celery leaf plant extract. The reaction produced a viscous, slimy precipitate from which ZnO nanopowder was obtained. The synthesis process started by filling a 250 ml beaker with 50 ml de-ionized water, weighing 0.6815 g of $ZnCl_2$, then added it to the de-ionized water and let the solution stir on magnetic stirrer. While stirring, 20 ml of plant leaf extract was being added to the solution via pipette drop by drop. At first the zinc salt solution is white, but the addition of plant extract changed the solution color to yellowish – faint green color as shown in figure 2. It is noteworthy to mention that the chemical reactions led to formation of ZnO nanoparticles precipitate which occurred at room temperature, no heating applied but only continuous mechanical agitation via magnetic stirring. Lastly, 2.5 ml of NaOH aqueous solution was added to former solution using dropper and let it stir for 45 minutes. Once the NaOH solution is added, very tiny white particles start to form immediately indicating the nucleation and growth of ZnO nanoparticles.

The solution is then transferred into centrifuge and run the samples for 30 minutes to separate the nanoparticles from the aqueous solution. The precipitate is then washed with ethanol for several times to get rid of undesired by-products, dried using incubator for two hours, and grinded using pestle and mortar till fine powder is obtained. Lastly, the nanopowder was calcinated in an oven at 600^{o}C for 6 hours as shown in figure 3. The final product is grey powder form of ZnO nanoparticles as shown in figure 4.

Figure. 2. – The nano-solution final color after addition of plant extract

Figure. 3. Oven used for calcination process

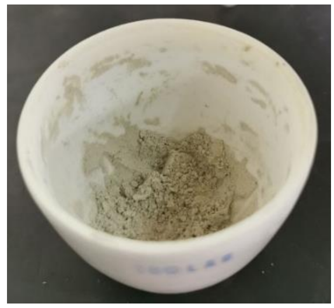


Figure. 4. Final form of ZnO nanopowder after calcination

4. Results and Discussion

The trick in obtaining nano-sized particles is the conditions at which the synthesis process was performed. Ideally, the slower the reaction, the smaller the particles being formed. The magnitude of stirring speed is also a factor to be taken in consideration. The speed magnitude is recommended to be strong stirring opposite to the slow addition of aqueous solutions. Another factor affecting the size of synthesized nanoparticles is the duration for complete chemical reaction. The longer the solutions are let to react on the stirrer the more ZnO particles are expected to be formed, as most of the Zn^{2+} ions will be reduced by the plant extract [14]. The role of NaOH aqueous solution was being a precipitate agent aid to precipitate the nanoparticles from the aqueous nano-solution.

4.1. XRD Analysis

XRD is a non-destructive test used for phase identification of crystallographic structure, chemical composition and physical properties of a crystalline material [7]. Diffracted rays are generated when the nano-sample interacts with incident rays, the detected rays are then counted. The diffraction pattern is displayed measuring the intensity of diffracted rays at different angles of the tested nano-materials [15]. The phase purity and crystallinity of pure ZnO nanoparticles examined by X-ray diffraction technique is shown in figure 5.

From figure 5, sharp diffraction peaks are clearly observed located at 31.674º, 34.423º, 36.158º, 47.571º, 56.647º, 62.94º, 67.859º and 69.174º corresponding to (100), (002), (101), (102), (110), (103), (112) and (201) respectively. The diffraction peaks positively match with the JCPDS card no. (36-1451) indicating the formation of well crystalline ZnO nanoparticles with hexagonal wurtzite nanocrystals structure and lattice constants a = b = 3.249 Å and c = 5.206 Å. The consistency of the diffraction peaks with the ZnO card of JCPDS confirms that the synthesized nanopowder is free of impurities as no other characteristics XRD peaks are observed. Furthermore, the difference in intensities of the diffraction peaks indicates that the growth rate is different in various directions. the growth rate affects the surface area of the plane in a way that smaller surface area is obtained at higher growth rate which gives lower intensity [16].

XRD analysis also gives indication on the crystalline average size for the tested materials. The crystalline size for the synthesized ZnO nanoparticles can be calculated using Debye-Scherrer formula, equation (4) [17];

$$d = \frac{0.89\,\lambda}{\beta \cos\theta} \tag{4}$$

Where d is the crystalline size in nm, 0.89 is constant Scherrer shape factor, $\lambda = 0.1541$ nm is the wavelength of Cu Kα radiation, β is the full width at half maximum (FWHM) of the diffraction peak (radians), θ is Bragg diffraction angle (peak position in radians). Therefore, the crystalline size for the corresponding plane of (101) at θ = 36.15º with FWHM equals to 0.2402º is found to be 34.39 nm.

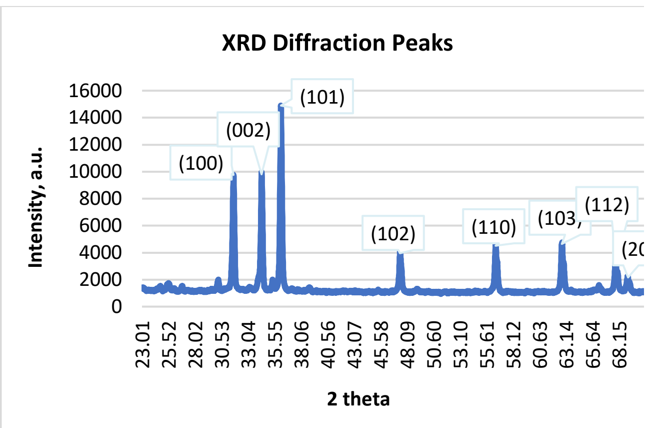


Figure. 5. XRD pattern for synthesized ZnO nanoparticles

4.2. Morphology Analysis – FE-SEM & EDS Tests

FE-SEM stands for Field Emission Scanning Electron Microscope. EDS stands for Energy Dispersive Spectroscopy. These two tests are essential for characterization purposes and are usually integrated together serving as imaging tool for nanoparticles and giving details on their surface morphology, particle sizes and shapes, imperfections, contamination sports and other metallographic information FE-SEM data is usually matched with EDS data, because FE-SEM capture the microstructure image of the materials and EDS identify the elemental composition of that material [18].

Figure 6 – shows images for the synthesized ZnO nanoparticles. The images clearly show the formation of hexagonal wurtzite nanocrystals structure with particles diameter ranging from 26 to 57 nm, which is compatible with the calculated crystalline size from XRD and demonstrate good quality of the ZnO nanoparticles. Hence, the average particle size calculated from FE-SEM test is 35.7 nm, as shown in figure (6-A). Some rough edges are observed as shown in figure (6-B & C) which are accounted for the layers of crystal structure of wurtzite. Additionally, it is hypothesized that the fast nucleation growth of the nanoparticles during the synthesis process contribute to formation of needle-like structures on the outside of the wurtzite particles during the layering process as shown in figure (6-D). Furthermore, in recent literatures researchers revealed that the low temperature conditions at which the nanoparticles were synthesized promote the formation of nanorods like structures scattered among the spherical or wurtzite particles.

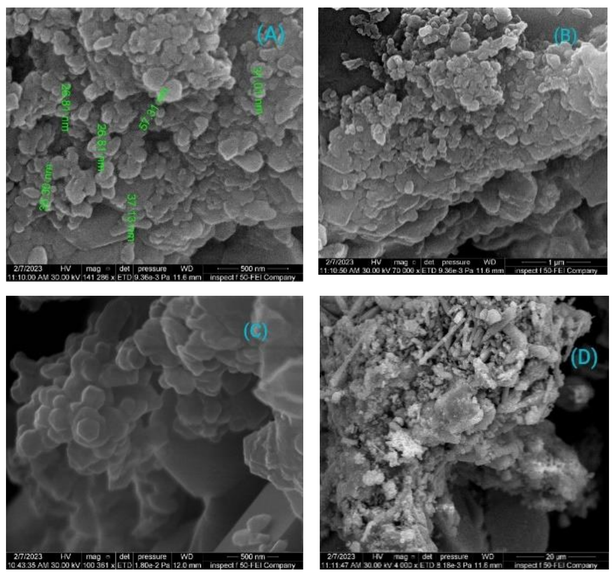
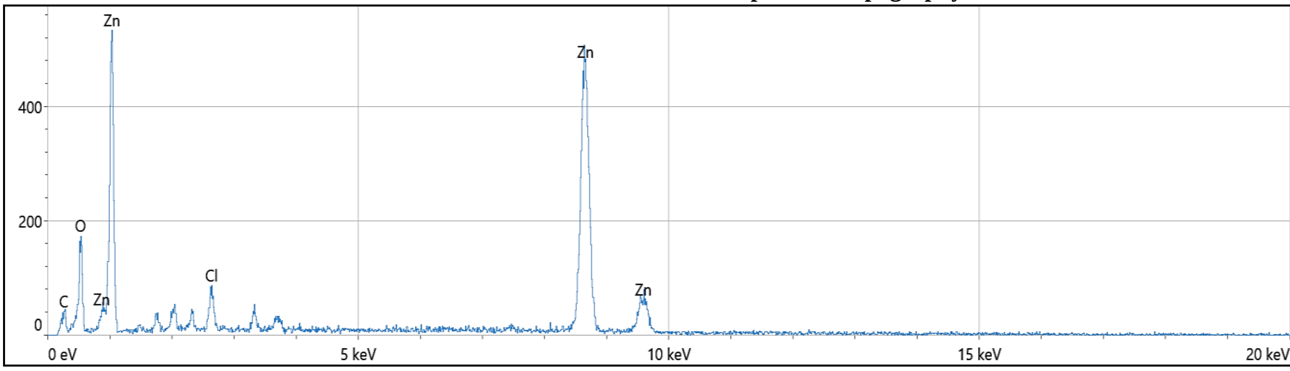


Figure. 6. FE-SEM morphology for ZnO Nanoparticles

Furthermore, in order to confirm the formation of ZnO nanoparticles, EDS analysis was performed. During the EDS measurement different areas were focus and the corresponding peaks are shown in figure 7, also table 1, shows both atomic and weight percentage for each element composing the synthesized ZnO nanoparticles. EDS results show weight percent of 69.6% for Zn-element and 24.2% for O-element. Adding both elements result in percentage purity up to 93.8% ZnO with impurity up to 6.2% represented by the presence of carbon and chlorine elements.

Table 1. EDS data for the elemental composition for the synthesized ZnO nanoparticles

EDS data		
Element	**Atomic %**	**Weight %**
C	27.7	3.9
O	41.4	24.2
Cl	3.1	2.3
Zn	27.7	69.6



4.3. AFM Analysis

AFM stands for Atomic Force Microscope. AFM test allows for 3D characterization of nanoparticles with sub-nanometer resolution. AFM test is considered among the most powerful characterization tools for surfaces scaled in micro and nano levels due to the superior resolution of the instrument [19]. This test provides both quantitative and qualitative information for many physical properties including surface texture, morphology and most importantly is nanoparticles average particles diameter [20]. The unannealed ZnO nanoparticles size and shape distribution were examined by the surface topographic data with atomic resolution. Figure 8, shows space morphology captured by the AFM microscope, whereas figure 9, demonstrates the surface distribution for the granularity of the synthesized ZnO nanoparticles. AFM test results vividly confirm that more than 50% of the synthesized ZnO nanoparticles has an average diameter size of 35.96 nm. Furthermore, AFM results also showed that about 90% of the ZnO nanoparticles has particle size of 58 nm, whereas, 10% of the ZnO nanoparticles has particle diameter of 16 nm. The AFM results are further confirming the purity and homogeneity of the synthesized ZnO nanoparticles size that gave similar results to XRD, FE-SEM and EDS test, all of which are consistent and confirm that the synthesized ZnO nanoparticles have diameter size within the range of (26-58) nm.

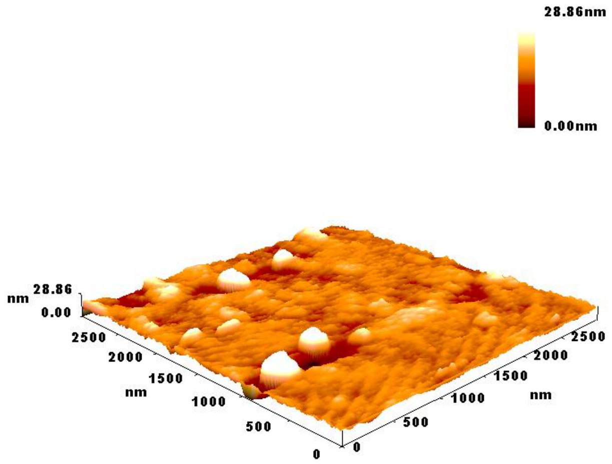


Figure. 8. AFM image showing the synthesized ZnO nanoparticles topography

Figure. 7. EDS graph for elemental composition for the synthesized ZnO Nanoparticles

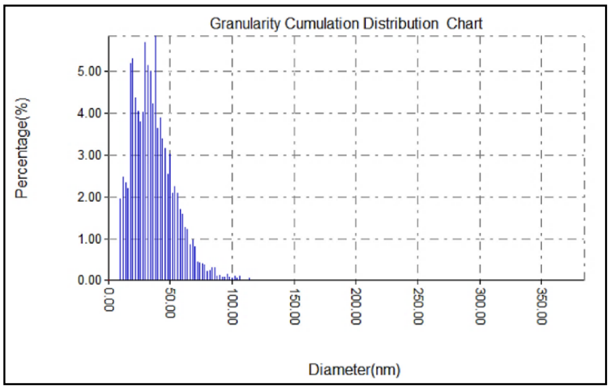


Figure. 9. Shows the granularity distribution for the synthesized ZnO nanoparticles using AFM atomic microscopy

5. Conclusions

In short words, this paper presented experimental work for synthesizing bio-based nano-sized of ZnO particles using zinc chloride salt and celery leaf plant extract with aid from NaOH aqueous solution as precipitate agent. The synthesized process utilized the “bottom-up” approach in producing the nanoparticles exploiting the particular method of “Co-precipitation”. The conditions at which the reaction that led to formation of ZnO nanoparticles occurred at room temperature using mechanical agitation. Calcination of the nanopowder was performed to ensure good crystals alignment and order of atoms periodicity. The characterization tests included XRD, FE-SEM, EDS and AFM. All of which confirmed the formation of good quality and purity of ZnO nanoparticles with an average particle size of about 35 nm. Additionally, the synthesized ZnO nanoparticles were compatible with JCPDS card no. (36-1451) with similar peak profile for pure ZnO nanoparticles as shown through XRD graph analysis. Morphology analysis included FE-SEM and EDS tests, FE-SEM analysis showed that shape for the synthesized ZnO nanoparticles is hexagonal wurtzite nanocrystals structure, whereas, EDS analysis confirmed purity percentage for the synthesized ZnO nanoparticles to be 93.8% accounted from 69.6% purity for Zn-element and 24.2% purity for O-element.

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