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Green Synthesis of ZnO Nanoparticles Using Potato Extracts (Solanum tuberosum) As an Antibacterial Agent in the Active Packaging For Food Products

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Abstract.
In this work, we have implemented an eco-friendly method for synthesis of zinc oxide nanoparticles (ZnO NPs) using potato extracts (Solanum tuberosum) as the natural bio-reductants and stabilizing agent. Meanwhile the zinc nitrate octahydrate ($Zn[NO_3]$ $2.6H_2O$) and ammonium hydroxide (NH_4OH) were used as the precursor of ZnO NPs. The sediment of $Zn(OH)_2$ was separated from the solvent using centrifuge, and further calcinated at the temperature of 500°C so that formed ZnO nanoparticles. SEM micrograph of ZnO NPs shows the hexagonal shapes with particles size around 50–100 nm. The XRD spectra confirms that ZnO NPs exhibited well crystalline nature, however, there were impurity peaks caused by the potato extracts addition. From the FTIR analysis, we obtained the existence of another chemical compounds, denoting the presence of stabilizing and reducing agent from potato extract.

Keywords: ZnO nanoparticles, green synthesis, antibacterial and active packaging

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1. INTRODUCTION

Packaging is the first thing consumers see in product offerings. Even if consumers do not purchase the packaging, it is essential to change consumers' assessment of the food product itself [7]. Smart packaging is one of the new technologies in the field of packaging. In contrast to conventional packaging providing only information about the product itself (such as manufacturer, expiration date, and composition), smart packaging can inform the changes occurring in the product or its environments such as temperature, pH, and microbial growth. An example of the smart packaging application is packaging that can detect changes in food products in the form of sensors, such as Time-Temperature Indicator (TTI), gas indicator, or food freshness indicator. Another application is packaging that can actively extend shelf life of the consuming product.

Some examples are packaging that can absorb CO_2, water, ethylene gas, or other compounds which can cause a food product to spoil quickly. Then, packaging that actively releases certain gases or substances to increase shelf life; and antibacterial packaging, hereafter known as active packaging [8]. Bacterial/microbial growth is the main factor causing food spoilage; hence, an antibacterial agent is needed on the packaging [2,8].

Active packaging is come under smart packaging in which the packaging systems can actively absorb or release substances into or from packaging or the environment arround the food product. Active packaging propose an innovative approach to keep or extend shelf life and sustaining the quality and security of consuming products [1]. Microbial growth is the leading factor that causes the food to be spoiled [2]. One of the solutions to overcome these problems is the use of antimicrobial packaging. Nanoparticles in which particle diameter less than 100 nm, including titanium dioxide (TiO2) and zinc oxide (ZnO), have been widely research because they have the significant antimicrobial potential [3]. Research on ZnO nanoparticles is impressive for scientists because it is cheap to produce, safe, and easily made. Even the FDA of the United States had registered ZnO as a safe metal oxide [4].

Antibacterial agents can be derived from plant extracts (pomegranate peel, papaya, jack fruit, rosemary, durian husk, etc.), essential oils (oregano oil, rosemary, cinnamon, soybeans), organic acids, polymers (chitosan), nano-materials usage, and so on. [9]. Both plant extracts and essential oils are proven to have antibacterial properties. However, there will be problems applying them on the packaging requiring a high temperature in its manufacture, e.g., the injection molding process, lamination, or extrusion. High temperatures will lead to the loss of bioactive compounds in the plant extracts [9]. As with nanoparticles, high-temperature treatment (annealing) on the nanoparticles is necessary to increase the degree of crystallinity of the nanoparticles formed. The use of nanoparticles in active packaging is the proper solution for broader application in plastic packaging, metal packaging, paper packaging, and biodegradable packaging.

Several types of metal nanoparticles such as silver nanoparticles (Ag-NP) [10], zinc oxide nanoparticles (ZnO-NP) [11], and titanium dioxide (TiO2) [12] have been broadly investigated as antibacterial agents. ZnO nanoparticles are multifunctional materials that demonstrate antimicrobial properties and emit near UV emissions. They are piezoelectric, transparent, and have high electrical conductivity. The antibacterial characteristic of ZnO NPs is affected by the phase, the size of crystal lattice, lattice constant, crystal orientation, surface defects, and particle size. ZnO-NPs is widely used in cosmetics packaging, food packaging, biosensors, and drug delivery due to its excellent biocompatibility. In addition, ZnO has been considered a generally safe ingredient (GRAS) by the United States Food and Drug Administration (21 CFR 182.8991). Therefore, ZnO-NP is often used as an antimicrobial agent in the active packaging [11].

The synthesis of nanoparticles can be conducted with either physics or chemistry methods. The physics methods require costly equipment, high temperature and pressure, and an ample space for engine settings. Chemical methods sometimes involve the use of toxic chemicals that are harmful to the environment and the people who handle it. Thus needs an environmentally safe and economize method for nanoparticle synthesis [5]. This study associated with the green method of synthesis ZnO nanoparticles using natural bio-reductants and stabilizers of potato extracts (Solanum tuberosum). Potatoes have a lot of starch which consists of 80% amylopectin and 20% amylose. Both macromolecules have abundant hydroxyl groups to be used as bio-reductants and stabilizers to form nanoparticles [6].

Research methods begin with manufacturing the potato tubers extracts obtained from a local grocery/convenience store based on methods developed by Buazar [6]. Synthesis of ZnO nanoparticles using a precursor of zinc nitrate (Zn (NO3)2.6H2O) and ammonium hydroxide (NH4OH) for analysis from (Merck). This study will be conducted in 3 variations: 1) zinc nitrate + potato tuber extract; 2) zinc nitrate+potato tuber extract + NH4OH with a pH of 8; and 3) zinc nitrate+NH4OH with a pH of 8. The tree samples are used to prove that the extracts of the potato tubers have bio-reductant abilities and are part of the green synthesis. Nanoparticles produced are further characterized by UV-Vis to determine the optical properties. SEM-EDX is used to determine the morphology and size. XRD is used to determine crystal structure. Then, FT-IR is applied to determine the functional groups. Besides, the antibacterial activity test is also carried out. The sustainability of this research is the application of ZnO nanoparticles as an additive ingredient in the packaging. Antibacterial initial testing in the packaging is performed on cardboard paper made of recycled paper.

2. METHODOLOGY/ MATERIALS

The methodology started the production of the potato extracts. The potato was cut and dried under the sun to remove the water content. Further, the 10 grams of dry potato are boiled in 500 mL aquadest with a temperature of 85°C for 30 minutes until it becomes muddy. Next, the extract is separated from its sediment by using a centrifuge. The obtained extract is saved within the fridge for more usage. [6]

The synthesis of nanoparticles ZnO used zinc nitrate precursor (Zn $(NO_3)_2.6H_2O$) and ammonium hydroxide (NH_4OH) from (Merck) analysis. The 3 grams of zinc nitrate dissolved inside 50 mL potato extracted at the temperature 85 °C and stirred for 30 minutes using a hot-plate magnetic stirrer. These samples were used to verify that potato tubers extract had bio-reduction ability and were included in green synthesis. The sediment of $Zn(OH)_2$ is separated from the solvent that is used as a centrifuge, further calcinated at the temperature of 500 °C so that formed nanoparticles ZnO. The generated nanoparticles are further characterized by UV-V is to know the optical traits, SEM-EDX to know its morphology and size, XRD to know the crystal structure, FT-IR to know the cluster function, and the test of antibacterial activity.

3. RESULTS AND DISCUSSIONS

3.1. Morphological Analysis

SEM-EDX measurement results show the shape of the ZnO particles and the composition of the compounds contained in the material. SEM micrographs show the morphological topography of ZnO NPs, indicates hexagonal shapes. ZnO nanoparticles looks agglomerated (not homogeneous yet) and looks like cotton which can be seen at 30.000 times magnification in the SEM results. The occurrence of agglomeration in ZnO nanoparticles is possible due to the excessive amount of potato extract. The EDX results show that the components composed are the element Zn with a mass percentage of the element of 55% and the element O of 19.4%, and other element comes from potato extract.

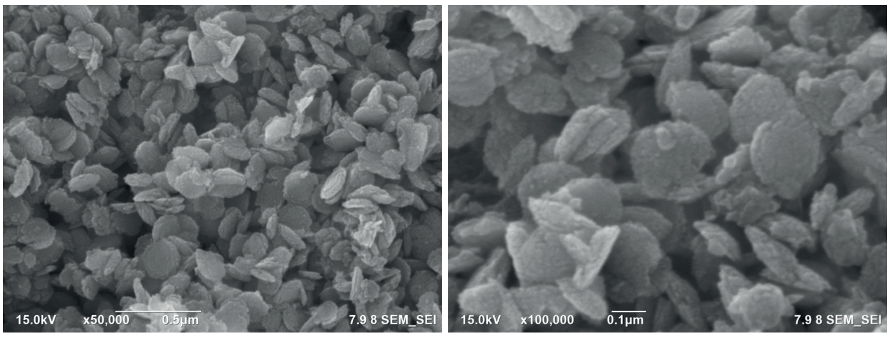

Figure 1: SEM Micograph of the synthesized ZnO NPs using potato extract as stabilizing and reducing agent.

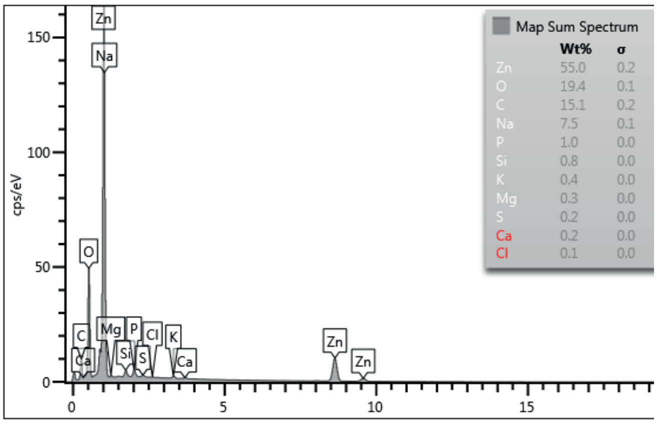

Figure 2: The EDX Result of the synthesized ZnO NPs using potato extract as stabilizing and reducing agent.

3.2. XRD Analysis

The X-ray diffractometer (XRD) was used to identify the NPs crystalline structure and clarity phase of ZnO NPs. This figure below represents the diffractograms of synthesized ZnO NPs.

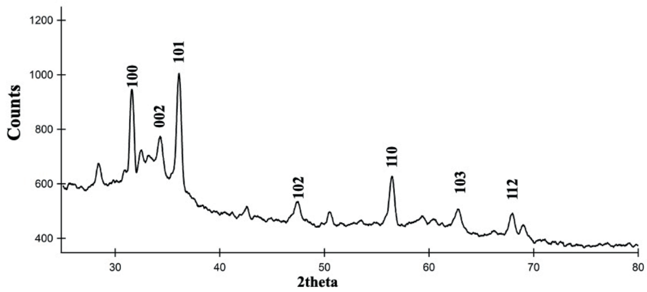

Figure 3: The XRD Spectra of the synthesized ZnO NPs using potato extract as stabilizing and reducing agent.

The diffracted peaks of ZnO NPs matched with the JCPDS standard value number 3-888. The XRD spectra shows that ZnO NPs exhibited well crystalline, but there are impurity peaks caused by the adding of potato extracts. The degree of crystallinity of ZnO nanoparticles appears at 2theta: 31.58^o, 34.3^o, 36.1^o, 47.48^o, 56,48^o, 62.86^o, and 67.98^o. The values (h,k,l) of ZnO nanoparticle crystals at 2 tetha are (100), (002), (101), (102), (110), (103), (112), and (112). The XRD spectra exhibit a hexagonal crystal phase with the hkl plane of (101) as the strongest peak

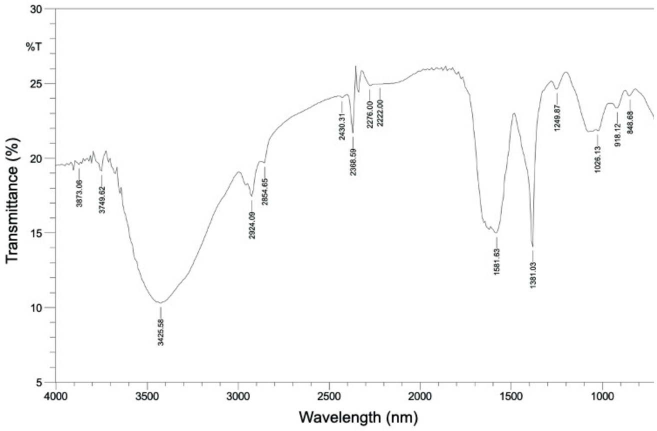

Figure 4: The FT-IR Analysis of the synthesized ZnO NPs using potato extract as stabilizing and reducing agent.

3.3. FT-IR Analysis

The FT-IR characterization used to determine the functional compounds involved in the ZnO NPs synthesis and confirm the Zn-O bonds in the sample. The functional groups of the synthesized NPs has been analysed in the range of 4000–400 cm^{-1}. The bands at 462 cm^{-1} shows the vibrational modes and stretching frequency of Zn and O. The peak at 1381 occurred due to stretching vibrations of C–O bond, while band at 1581 cm^{-1} showed the C=C bond. The stretching mode of water was shown by O–H broadband at 3425 cm–1 shows. The obtained results confirm the presence of another phenolic compounds, indicating the existence of reducing and stabilizing agent from potato extract.

3.4. Diffuse Reflectance Spectroscopic Analysis

Diffusion Reflectance Spectrum was measured by UV–Vis spectrophotometer in the range of 200–800 nm. The ZnO sample shows absorption spectra at a wavelength of less than 400 nm as shown in figure below. The UV-Vis absorbances spectroscopy shows the the characteristic peak of ZnO at wavelength of 356 nm.

3.5. Antibacterial Activity

The antibacterial activity in ZnO had research in the form of ZnO composite and chitosan as an antimicrobial packaging material [15, 16]. The result shows that the account of dielectric constant and its conductivity increased when ZnO NPs is added. The potential

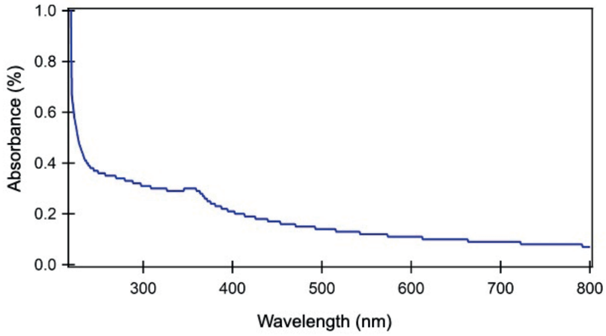

Figure 5: The FT-IR Analysis of the synthesized ZnO NPs using potato extract as stabilizing and reducing agent.

of composite development shows the enhancement of antimicrobials comparing to the purely chitosan.

The antibacterial activity of nanoparticles ZnO that is combined in the polymer of LDPE (Low-Density Polyethylene) and LLDPE-MA (Linear Low-Density Film Anhydrida Polyethylene-co-maleic) had been researched [17]. This research innovatively produces the antibacterial movie of polymer active by combining nanoparticles zinc oxide (ZnO-NP) to be LLDPE grafted with LLDPE-MA and LDPE used polymer melting and ZnO-NP coating. The evaluation results of physical traits and antibacterial effect towards foodborne pathogens through fluid and disc diffusion in agar test. The result showed ZnO-NP content increased antibacterial activity [17].

The green synthesis method is required in the production of nanoparticles ZnO so that it is more eco-friendly. Meanwhile, the further implementation of nanoparticles ZnO as antibacterial activity in the food packaging is extremely promising, proven by ZnO could be made in the form of composite, or dispersed in polymer for plastic packaging, also still possible to be applied to others kind of packaging, for instance antibacterial metal packaging and biodegradable packaging.

4. CONCLUSION AND RECOMMENDATION

We have successfully synthesis the ZnO Nanoparticles with Green Method using Potato Extracts (Solanum tuberosum) as an Antibacterial Agent in the Active Packaging for Food Products. The SEM graphs of ZnO NPs exhibits the hexagonal shapes, while the EDX report legalized the elemental composition of Zn and O in synthesized elements and other material from potato extract as stabilizing agent. The diffracted peaks of ZnO NPs obtained peaks matched with the JCPDS no. 3-888. From XRD pattern shows that ZnO NPs exhibited well crystalline nature and this shows the synthesized ZnO NPs had been crystallized, but there are impurity peaks caused by the adding of potato extracts. From the FT-IR analysis, we obtained the presence of phenolic compounds, indicating the existence of secondary metabolites from potato extract.

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