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Green Synthesis of ZnO Oxide Nanoparticles using *Acorus Calamus* Leaf Extract: Effect of Solution pH

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Abstract

The green synthesis of zinc oxide nanoparticles (ZnO NPs) is cost-effective, biocompatible, easy and upscale-able. The method gaining attention due to its environmentally friendly applications which use renewable resources. *Acorus Calamus* leaf extract has been used for various purposes, including medicine. This study synthesized ZnO NPs using a greener process using precipitation method under different pH levels. The resulting ZnO NPs were characterized using Fourier Transform Infrared (FTIR), and X-Ray Diffraction (XRD). The results show the existence of spherical, hexagonal wurtzite structures in the synthetic ZnO NPs. Next, Fourier Transform Infrared (FT-IR) analysis was performed to identify the functional groups in ZnO NPs. It shows the difference between pH5, pH 6, pH 7 and pH 8. The more acidic the solution, the sharper the peak of the functional group (C-H) shown. The study found that changing pH affects the activity of the solution, potentially making it useful in medicine.

1. Introduction

Nanotechnology has significantly grown in recent years, with new opportunities in medicine, gene delivery, nanomedicine, and biosensing. Ultra-fine particles with a diameter between 1 and 100 nanometers are known as nanoparticles (NPs), with a high surface-to-volume ratio [1]. Synthesis of nanoparticles is carried out using biological, chemical, or physical methods, such as laser ablation, hydrothermal synthesis, gel synthesis, and lithography. However, these methods require special equipment and skilled labor. Green synthesis techniques, which are more affordable and environmentally benign than chemical and physical procedures, are preferred due to their cost effectiveness, environmental friendliness, and potential for biocompatible nanoparticles [2]. ZnO nanoparticles (NPs) are used in various technologies, such as solar cells, chemical sensors, and antibacterial activities. They have unique properties, including photochemical ability, fungicidal, antibacterial, catalytic, and therapeutic activities [3]. However, these processes require complex machinery, time-consuming procedures, hazardous chemicals as capping agents and demanding experimental setups. Plant-based nanoparticle synthesis involves easily scaled production of NPs and forges a connection between nanotechnology and plants [4].

Acorus Calamus, commonly referred to as "sweet flag," is a marginal aquatic deciduous perennial that spreads widely. It has sword-shaped, iris-like leaf blades and normally grows in basal clusters up to 30" tall. It is an annual, semi-aquatic, sterile triploid plant of the family *Araceae* that has a pleasant scent. It has been found in several regions of North America, Europe, and Asia [5]. The traditional Indian herb *Acorus Calamus* is used to cure a variety of illnesses, including neurological, gastrointestinal, pulmonary, metabolic, renal, and liver problems. *Acorus Calamus's* roots and leaves both have antioxidant, antibacterial, and insecticidal properties. The rhizome contains a diverse spectrum of bioactive substances, including phenolic compounds, alkaloids, flavonoids, and triterpenes that act as natural antioxidants. *Acorus Calamus*, a marginal aquatic deciduous perennial, acts as a capping agent to reduce the size of ZnO NPs. This study describes the environmentally friendly synthesis of ZnO nanoparticles through *Acorus Calamus* and the characterization of the produced particles [5].

During the synthesis of NPs, it has promoted agglomeration of NPs which reduces the surface area of the NPs and further decreases their effectiveness and performance [6]. Agglomeration is the process of creating new particles by the collision of at least two original particles. Agglomerates can form through several different mechanisms, such as electrostatic forces between nano-sized particles, spraying additional liquid between particles to create bridges that can solidify after the liquid in solution or suspension evaporates, or thermal processes such as sintering or glass transition. Incorporation will reduce the surface area, resulting in a small reaction. The physicochemical synthesis process of NPs has produced potentially hazardous by- products that must first undergo extensive treatment before being released into the environment [7]. Therefore, greener methods have been used in synthesizing nanoparticles to reduce environmental issues. In this regard, the synthesis of ZnO NPs from plant extraction as a capping agent was used for this study to clarify the surface morphology and structure of ZnO NPs using the extracted *Acorus Calamus*.

The objective is to prevent agglomeration from happening. The present study explains the environmentally friendly synthesis of ZnO NPs via *Acorus Calamus*, as well as the characterization of the produced particles. ZnO NPs synthesis using plant extracts can enhance antioxidant activity and reduce hazardous chemicals and solvents usage. A greener technique using plant extraction can reduce environmental concerns and protect aquatic life.

2. Materials and Methods

2.1 Materials

Fresh *Acorus Calamus* leaves were obtained from Pagoh, Johor, Malaysian agricultural region. Hydrochloric acid (HCl), sodium hydroxide (NaOH), commercial ZnO (Sigma-Aldrich), oxalic acid and zinc acetate were acquired from R&M Marketing, Essex, UK. The general flow of the overall research methodology is summarized in Fig. 1.

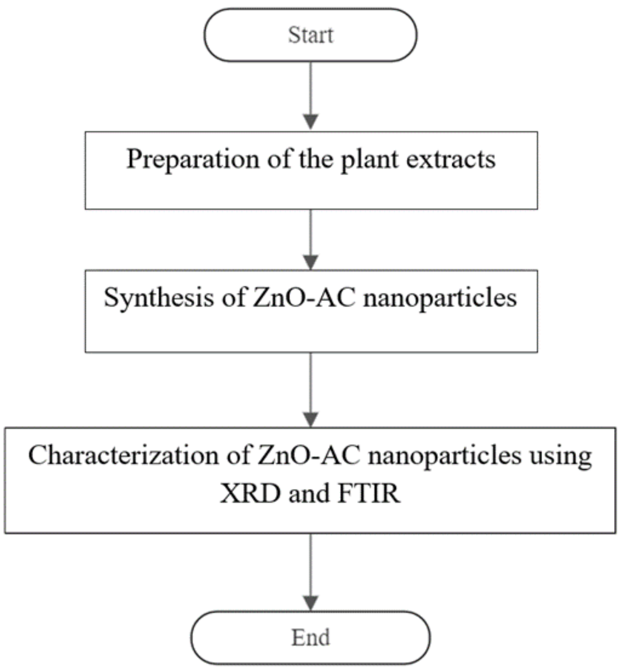

Fig. 1 Flow chart of research activities

2.2 Preparation of the Plant Extracts

The obtained *Acorus Calamus* leaves were rinsed multiple times with tap water to remove any dust, and dried before being ground in a grinder and sieved. 10 g of *Acorus Calamus* leaf powder was taken and boiled in deionized water at 80°C for 60 minutes. The extract was filtered using Whatman filter paper and vacuum pump and stored in an incubator shaker at 4°C with 150 rpm.

2.3 Synthesis of ZnO-AC Nanoparticles

ZnO was synthesized by adding 0.15 M of oxalic acid dehydrating solution to the 0.1 M of zinc acetate dehydrating solution at room temperature. The ZnO mixture was added to 10 mL of *Acorus Calamus* leaf extract (pH 5-8) in the ratio of 3:1, after 5 minutes. Then, the mixture was agitated, and the precipitate was filtered using Whatman filter paper and vacuum pump and the remaining water was removed by drying in an oven for an hour at a temperature of 100°C and calcined in a furnace for 2 hours at 550°C.

2.4 Characterization of ZnO-AC Nanoparticles

The functional groups of ZnO-AC were characterized via Fourier Transform Infrared Spectroscopy (FTIR; Agilent Tech Cary 600 Series) with the wavelength range of 600-2500 cm-1. The crystalline structures and purity of the samples were characterized via X-Ray Diffraction (XRD; Bruker D2 Phaser).

3. Result and Discussion

3.1 FTIR Analysis

Fig. 2 illustrates the results of the FTIR study used to identify the functional group compositions of ZnO-AC at pH 5, pH 6, pH 7, and pH 8. The C-H bending corresponded to the stretch bands at Fig. 2 (a)-(c), at 1439 cm^{-1}, 1439 cm^{-1}, and 1446 cm^{-1}, confirming the existence of the alkane group. Figure 2(a)-(c) reveals the stretching vibration at 1105 cm^{-1}, 1109 cm-1, and 1112 cm^{-1} is assigned to the stretching of the C-O groups of alcohols in Acorus Calamus. Stretch bends at 841 cm^{-1}, 841 cm^{-1}, and 767 cm^{-1} in Fig. 2 (a)-(c) were responsible for the C=C bending, indicating the existence of the alkene group. The C-H peak becomes sharper with increasing solution acidity. Consequently, it displays the maximum strength. When ZnO-AC at pH 8, C-H bending, and C-O stretching does not present. This is because the solution is too alkaline. While the peak is not sharp, it becomes more pronounced as the solution becomes more alkaline. Consequently, the peak displayed is not strong. The observation of these functional groups in the spectrum of zinc oxide nanoparticles is related to the presence of natural compounds in plant extract that cover the surface of nanoparticles. Based on the results it can be observed that the smaller peak is on the left side indicating that it is aromatic and if the peak at the right it indicating as alkyl.

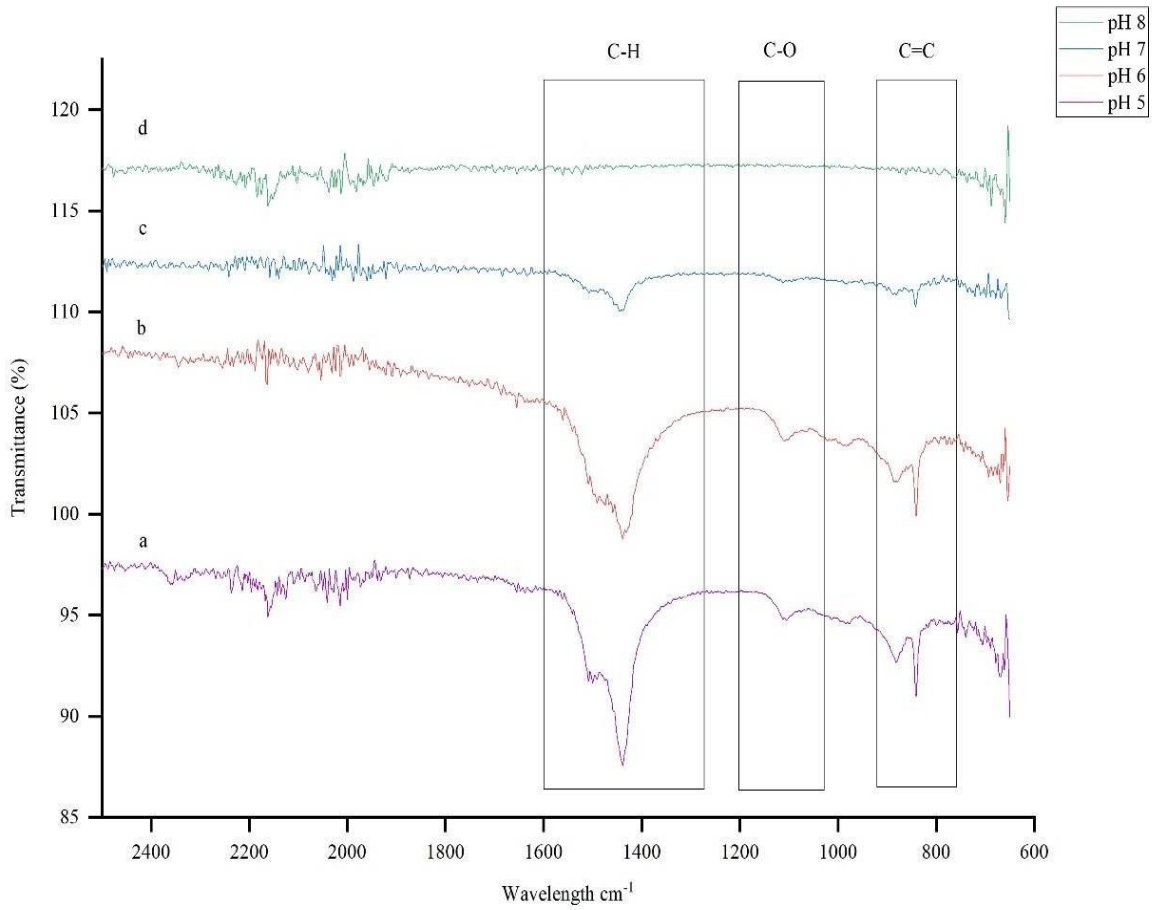

Fig. 2 *FTIR spectra of (a) ZnO-AC at pH 5; (b) ZnO-AC at pH 6; (c) ZnO-AC at pH 7; (d) ZnO- AC at pH 8*

3.2 XRD Analysis

Fig. 3 shows the results of the XRD analysis used to identify the crystallinity structure and purity of ZnO-AC at pH 5 and zinc oxide commercial [8]. Fig. 3 depicts the crystalline phases of the greenly synthesized ZnO-AC NPs, which were characterized by XRD (20°< 2θ<70°). All diffraction peaks (space group P63mc, JCPDS card number 36-1451) were attributed to the development of the hexagonal phase of ZnO. The ZnO-AC nanoparticles appeared to have high crystallinity based on their sharp and narrow peaks. Zincite and ZnO were identified by their unique diffraction peaks at 2θ = 32°, 35°, 36°, 48°, 57°, 63°, 67°, 68°, and 69°. According to [9] and [10], all the strong peaks mostly belonged to the (100), (002), (101), (102), (110), (103), and (112) planes, which fit the ZnO structure well. There were no peaks that corresponded to contaminants, and the peak intensity profiles showed signs of the nanoparticles' hexagonal shape. Commercial ZnO NPs had sharper reflection peaks than ZnO-AC nanoparticles, which might be explained by the former's higher crystallinity. The ZnO-AC samples' diffractograms matched those of commercial ZnO exactly. This was probably because the nanoparticles made by mixing ZnO and A. calamus leaf extract dispersed so well. ZnO nanoparticles were synthesized using Nephelium lappaceum L. extract, and [11] discovered identical diffraction peaks at 2θ values of 32°, 35°, 36°, 48°, 57°, 63°, 67°, 68°, and 69°.

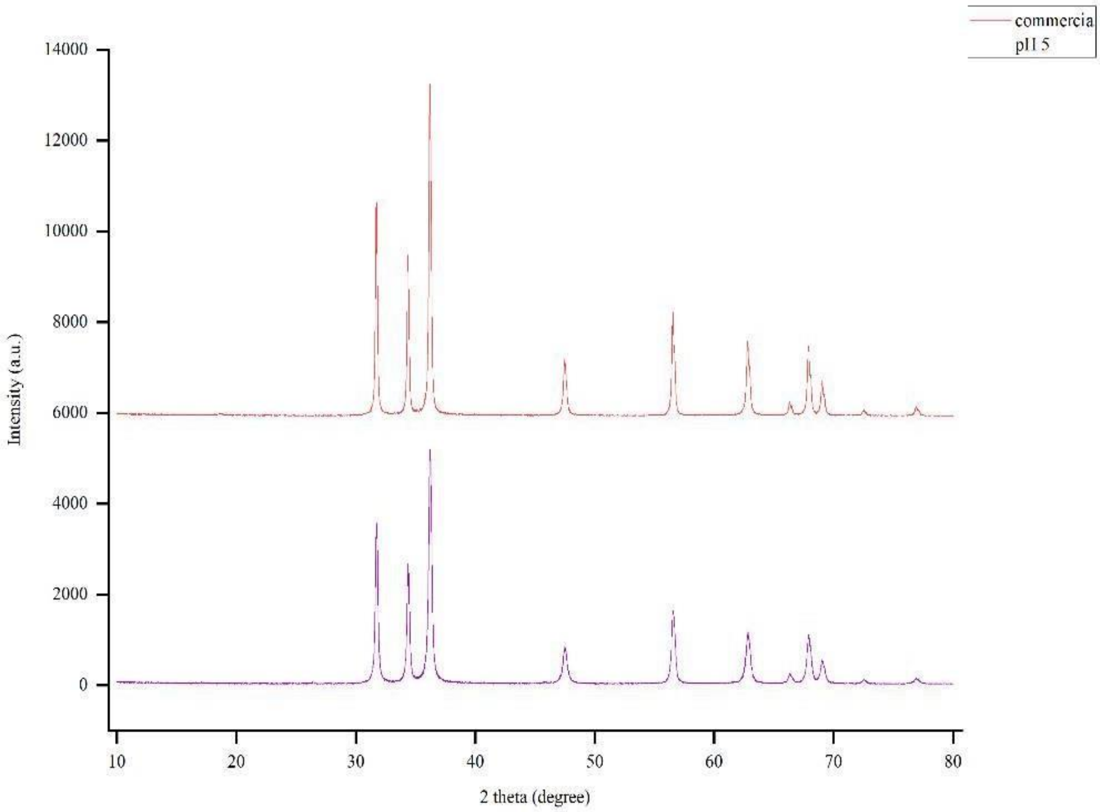

Fig. 2 *XRD spectra of ZnO-AC at pH 5 and zinc oxide commercial*

4. Conclusion

In conclusion, the green synthesis of ZnO NPs was discovered. The green synthesis Acorus Calamus in the presence of ZnO-AC nanoparticles is projected to have great potential. The synthesis of ZnO from Acorus Calamus extract was successfully obtained. Studies on the *Acorus Calamus* plant demonstrate the viability of green synthesis at the nanoscale. Recently, green synthesis at the nanoscale has become popular. However, there are several obstacles that need to be resolved with green synthesis, including challenging extraction processes, irregular particle size, and others. By employing inexpensive and readily accessible raw materials, it is possible to enhance the production of nanoscale zinc oxide particles. Today, there is a problem in effectively synthesizing ZnO NPs from leaves. Therefore, it is probable that the green production of zinc oxide offers developers a vast and excellent possibility. This new discovery will not only provide the elucidation of ZnO synthesis from Acorus Calamus, but also provide a fundamental knowledge to pursue the green synthesis technology in nanoparticles industries.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Aina Nabila Rosli, Nurul Aisyatul Alissa Mohd Agos, Dilaeleyana Abu Bakar Sidik, Nur Hanis Hayati Hairom;* ***data collection:*** *Aina Nabila Rosli, Nurul Aisyatul Alissa Mohd Agos, Dilaeleyana Abu Bakar Sidik, Nur Hanis Hayati Hairom;* ***analysis and interpretation of results:*** *Aina Nabila Rosli, Nurul Aisyatul Alissa Mohd Agos, Dilaeleyana Abu Bakar Sidik, Nur Hanis Hayati Hairom;* ***draft manuscript preparation:*** *Aina Nabila Rosli, Nurul Aisyatul Alissa Mohd Agos, Dilaeleyana Abu Bakar Sidik, Nur Hanis Hayati Hairom. All authors reviewed the results and approved the final version of the manuscript.*

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