An environment friendly synthesis and characterization of Zinc Oxide nanoparticles using Mentha viridis leaf extract

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Abstract. Zinc Oxide Nanoparticles (ZnO Nps) are one of the most predominant inorganic nanoparticles, which have diverse applications in the field of cosmetic, coating, medicine and electronic sensors. Morphology and particle size of nanoparticles mainly control its advantages and drawbacks in applications. In this study, an environment friendly and economic synthesis of ZnO Nps was carried out using Mentha viridis leaf extract as reducing and stabilizing agent to investigate the morphology, particle size and other properties of the synthesized ZnO nanoparticles. A series of characterization methods: ultraviolet-visible (UV-Vis) spectroscopy, x-ray diffraction analysis, field emission scanning electron microscopy (FESEM) and energy dispersive x-ray analysis (EDX) were used to confirm the characteristics of the synthesized nanoparticles. It was evident from the XRD analysis that ZnO Nps possess a polycrystalline hexagonal structure (as per the International Centre for Diffraction Data (ICDD) number #98-002-9272) with an average crystallite size of 32.3 nm. The spectra showing peaks in the EDX data indicated the presence of Zn (50.35 %) and O (49.65 %) in higher percentage and FE-SEM analysis revealed the morphology and Average Length and diameter of the nanorods are 156 nm and 48 nm respectively of the synthesized ZnO Nps. The analysis of the UV visible absorbance spectrum revealed the absorbance band peak at 372nm and confirmed the presence of ZnO Nps. From all these analyses, it is evident that Mentha viridis leaf extract can act as a potential reducing agent in the green synthesize ZnO Nps.
1. Introduction

Nanotechnology has framed as a vibrant research field in the recent past. Various science and engineering disciplines pave their way to incorporate nanomaterial with their research topics due to the outstanding properties of nanoparticles. Nanoparticles gain significant attention for their unique surface to volume ratio due to their diminutive size and concerning the bulk form, they exhibit most extensive variations in applicable properties which lead to a diverse application in the field of research such as photonics, semiconductor, medical and packaging [1, 2, 3]. In recent years, the ecofriendly and nontoxic processes have gained great attention in the research arena for producing various metal and metal oxide Nanoparticles. Green synthesis has adopted various bio-friendly materials such as plant extracts, fungi, algae to use as reducing and capping agent to synthesize nanoparticles [4,5,6]. The use of plant extracts offers a green route to control the variance of the shape, size and morphology of the particles [7]. Here plant biodiversity has been a great source for phytochemicals. Flavonoids and so many diverse types of aliphatic or aromatic hydrocarbons are such kind of phytochemicals [8]. Among the various metal and metal oxide nanoparticles, Zinc Oxide Nanoparticle is one of the most intriguing materials which can be produced by Green synthesis process. There are two distinct reasons for this interest: one is due to previous physical and chemical processes were environmentally hazardous and the other reason is, it has extended wide application in Agriculture, Biotechnology, Electronic sensor, Packaging Industry [9,10,11].

In this study, leaves of *Mentha viridis* used as a plant extract to synthesize ZnO Nanoparticles. *Mentha viridis* belongs to Mentha genus and widely available as an essential plant in the Indian subcontinent area. This plant is widely recognized as a medicinal plant because of its use in digestive disorders, headache and tiredness, anti-inflammatory and antimicrobial [12,13,14]. Abdelhakim Bouyahya et el. have shown the contribution of different phytochemicals present in *Mentha viridis* as antidiabetic, dermatoprotective, antioxidant and antibacterial properties [15]. So, the main purposes of choosing leaves of *Mentha viridis* as plant extract are: cost-effective, widely available, non-hazardous and most importantly rich in effective phytochemicals who will contribute to the synthesis as reducing agent. For the first time, this study synthesizes the ZnO Nps in which *Mentha viridis* propagate the system.

2. Materials and Methods

2.1. Materials

Locally produced *Mentha viridis* were collected from Keranigonj which situated at Dhaka, Bangladesh. The leaves were in fresh condition. Precursor of the synthesis was Zinc acetate powder, producer 'Sigma-Aldrich'. HPLC grade Methanol was used which is produced by Loba Chemi, India (purity-99.8%). Water which was used for the solution and cleaning was also HPLC grade. (Producer PubChem)

2.2. Preparation of *Mentha viridis* leaves extract

The leaves of *Mentha viridis* were cut to 8-10 mm and washed with running tap water at first, then washed again with deionized water thoroughly. 25 gm of leaves was added to 100 ml 70% HPLC gradient grade Methanol solution. The solution heated to 1 hour with continuous stirring on the hotplate at 65°C. Subsequently, the solution was kept to cool. Whatman No.1 filter paper was to attain the clear extract of Mentha viridis leaves. It was kept at 4°C for the later use.

2.3. Synthesis of Zinc Oxide Nano particles using *Mentha viridis* leaves

For the synthesis process, 10 ml *Mentha viridis* leaves extract solution was added to 90 ml of 1M zinc acetate aqueous solution. The solution heated to 60 °C and stirred continuously for 1 hour. During this process pH of the mixture was maintained in between 8 to 8.5 by instantly prepared 1M NaOH
solution to achieve smaller size nanoparticles. Subsequently, the solution dried at 90°C for 1 day. Afterwards dried powder transferred to a porcelain crucible for calcination and heated to 500°C for 2 hours. Finally synthesized dried powder of ZnO Nps was preserved for further studies.

2.4. Characterization methods

2.4.1. X-ray diffraction

Synthesized Zinc oxide Nanoparticles was investigated by X-ray diffractometer. Thus we got the crystal orientation and phase. Rietveld refinement method was used to determine the size of the crystal, parameter of lattice and analyze the phase of the particles.

| Operating parameters   | Values          |
|------------------------|-----------------|
| Target                 | Cu              |
| λ                      | 1.5406 Å        |
| Scanning Speed         | 0.1°/min        |
| Step Differences       | 0.02°           |
| Voltage Source         | 40 kV           |
| Tube current           | 40 mA           |
| Range of 2θ            | 20°-80°         |
| Mirror                 | Gobel mirror    |

2.4.2. FESEM and EDX

A field emission scanning Microscope was used to analyze the morphology of producing Zinc oxide nanoparticles. Size, shape, distribution system, thus identified by the image of SEM. Linear intercept method was used to determine the average grain size. To attain the elemental composition EDX analysis was procured by the same machine. Operating parameter: Voltage 15 kV, Probe Current 1.00 nA and acquisition range of time 50-100s.

2.4.3. UV-Vis

To obtain the absorption spectrum UV-Visible absorption spectrophotometer (Shimadzu UV-1800, Japan) was used. The characteristic absorption spectrum confirms the stable production of the ZnO. Scanning parameter was the 600-200 nm with the speed of 200nm/min.

3. Result and Analysis

3.1. X-ray diffraction analysis

Figure 1 exhibits the XRD spectrum of ZnO nanoparticles synthesized from the leaf extracts of *Mentha viridis*. The sharp diffraction peaks were identified at the 2θ values of 31.73°, 34.42°, 36.25°, 47.53°, 56.56°, 62.78°, 66.33°, 67.82° and 69.1°. These peaks were corresponded to the lattice planes of (100), (002), (101), (102), (110), (103), (200), (112) and (201) respectively. These distinct peaks conformed to the International Centre for Diffraction Data (ICDD) file no: 36-1451.

The formation of narrow and sharp characteristic diffraction peaks ascertains the crystallinity of biosynthesized ZnO. Furthermore, no characteristic diffraction peak other than ZnO was identified. That indicates the formation of pure ZnO. The average crystallite size of the synthesized ZnO NPs was 32.3 nm.
Figure 1: X-ray Diffraction pattern of Zinc oxide nanoparticles prepared by using *Mentha viridis*

Table 2: The rietveld refinement parameters of crystallographic data of Zinc oxide and goodness of fit parameters

| Parameters                        | Value       |
|-----------------------------------|-------------|
| Crystal orientation              | Hexagonal   |
| Space group                       | P63mc (186) |
| Quantitative analysis             | 100 %       |
| Lattice Parameter, a (Å)          | 3.25145     |
| c (Å)                             | 5.20834     |
| Cell volume, V (Å³)               | 47.685      |
| Crystallite size (Lvol-IB) K:1    | 32.3 nm     |
| (LVol-FWHM) K:0.89                | 38.4        |

| Goodness of fit parameters (R indices) |
|----------------------------------------|
| Rexp                                   | 9.31        |
| Rwp                                    | 16.84       |
| Rp                                     | 13.79       |
| Goodness of fit                        | 1.809       |

3.2. *FE-SEM and EDX analysis*

In the Figure 2 FESEM image of Synthesized ZnO nanoparticles, morphology of the nanoparticles clearly depicts the nanorod like structure. Some of the nanorods are clustered and some of them are free. Average Length and diameter of the nanorods are 156 nm and 48 nm respectively. Nanorod of ZnO represents 1D array and used vastly in the field of electronics [16]. Formation and growth of the ZnO nanorod mainly supported by the lowest surface energy of the facet of the nanorod [17]. Energy-dispersive x-ray spectroscopy (EDX) analysis one of the effective techniques to determine the elemental composition of particles. Actually the surface plasma resonance causes an absorption peak.
EDX confirms the presence of 80.12% Zn and 19.88% O in mass percentage scale in Figure 3, represents the purity of the Zinc oxide.

Figure 2: FESEM micrographs of Zinc Oxide prepared by Mentha viridis leaf extracts

Figure 3: EDX of ZnO nanoparticles by Mentha viridis

3.3. UV-vis spectrum analysis
In this study, optical property of green synthesized ZnO Nps investigated by absorbance spectrum from UV-vis characterization. Previous studies indicated that an absorption spectrum around 370nm confirms the successful synthesis of ZnO Nps. From Fig. 4, absorption peak obtained at 372nm confirmed the presence of ZnO Nps. Absorbance peak by ZnO [18,19,20] Nps mainly depends on particle size and shape which depends on several factors: calcination temperature, pH value, concentration of the precursor etc. [21,22,23]
4. Conclusion
This paper attempted an environment friendly synthesis of ZnO Nps mediated by the extract of leaves of Mentha Viridis. The characterization results show the shape of the Zinc Oxide particle like Rod, which is varies in the length within range of 80-135 nm and within diameter of 20-35 nm. XRD results show the stable phase is hexagonal wurtzite. Elemental Zinc and Oxygen are found in composition of particles which has been confirmed by the EDX result. The study can be further extended to the antimicrobial impact of ZnO Nps.

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