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Green synthesis and Characterization of ZnO Nanoparticles using Sterculia Foetida Leaf extract and its Photocatalytic Activity

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ABSTRACT

Zinc oxide nanoparticles (ZnO NPs) were prepared using zinc nitrate and Sterculia foetida (S. foetida) leaf extract as fuel by solution combustion method at 400 oC. The obtained material was characterized by UV-Vis, FT-IR, powder X-ray diffraction (PXRD), Energy dispersive X-ray analysis (EDAX) spectroscopy and morphological studies were carried out by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The PXRD result shows that, the average size of the synthesized ZnO particles are 20.38 nm. The band gap of the ZnO NPs was found to be 3.29 eV. The SEM image shows the ZnO NPs are spherical in shape and agglomerated. The photocatalytic activity of ZnO NPs was examined by degradation of Methylene blue (MB) under UV light irradiation.

Keywords : ZnO nanoparticles, Methylene blue, Photocatalytic activity.

I. INTRODUCTION

There are many organic compounds hazardous to human and animals such as dyes, pesticides and herbicides released from various industries without any treatment directly in to sea, lake and river water leads to water pollution. Water pollution is one of the most significant problems in the world, since it has an inauspicious effect on the water living organisms and human health [1]. Some standard techniques [2] engaged for the removal of aquatic pollutants suffer from several drawback like more cost, sludge of toxic compounds, environmental pollution and others. The organic wastes, such as dyes, drugs, etc. from aqueous media are degraded using semiconductor materials such as zinc oxide (ZnO) nanoparticles by photocatalytic technique [3]. The band gap energy of ZnO is 3.37eV and has innumerable applications due to its nano and microstructural properties [4-6]. It has found propitious applications in solar cells, gas sensors, UV-absorbers, electronic batteries, etc. [7-10]

There are various techniques employed to synthesize ZnO nanoparticles such as hydrothermal, sol-gel, microwave, wet chemical method [11-13]. The nanoparticles synthesized using plant extracts, i.e. green synthesis methods are simple, eco-friendly and non-toxic. *C. paradise* outer peel extract was used to synthesize ZnO nanoparticles, photocatalytic activity of the above ZnO nanoparticles was carried using methylene blue (MB) dye and it showed 56% degradation efficiency after 6h illumination time against MB dye [14]. The dye sensitized solar cells (DSSCs) use TiO_2 as a photo anode material to achieve high efficiency. Recently, ZnO nanoparticles have been substituted in the place of TiO_2 as a photo anode material, ZnO nanoparticles shows the efficiency up to 6.8% [15]. To achieve high efficiency in DSSCs, ZnO nanoparticles structure and size modification is required.

The present work aims for the synthesis of ZnO nanoparticles using *Sterculia foetida* leaf extract. *S. foetida* belongs to Malvaceae family. It is found in Europe, Africa and Asia [16]. *S. foetida* has anti-inflammatory activity as a CNS depressant [17], anti-obesity [18], anti-fertility [19], anti-oxidant [20]. The synthesized nanoparticles were characterized by various techniques such as UV-Vis, FTIR, PXRD, SEM–EDAX and TEM to check the presence of ZnO and to know about the size of the nanoparticles. The photocatalytic activity was carried out using methylene blue (MB) in presence of UV-light, the degradation efficiency was noticed at various time intervals, decrease in absorbance with time has been recorded.

II. MATERIALS AND METHODS

A. Chemicals Required

Zinc nitrate hexa hydrate and methylene blue were purchased from Himedia Laboratories Pvt.Ltd with 99% purity.

B. Preparation of leaf extract

The *Sterculia foetida* leaves were collected from Bangalore, Karnataka, India. The dust particles were removed by washing it with water and dried in the absence of sun light at room temperature. Then the leaves were powdered mechanically using mixer grinder, sieved and subjected to extraction through Soxhlet apparatus using deionized water for 68 hours. The obtained aqueous solution is concentrated using rotary flash evaporator at 40±5 °C under reduced pressure (Buchi, Flawil, Switzerland), then it is dried in hot air oven at 55-60°C, from the dried crude extract small amount is used for the synthesis nanoparticles.

C. Synthesis of ZnO nanoparticles

The ZnO nanoparticles were prepared by solution combustion method using aqueous leaf extract of *Sterculia foetida* as a fuel. In this process 0.1g of crude dried aqueous leaf extract of *Sterculia foetida* and stoichiometric amount of zinc nitrate hexa hydrate was dissolved in 10ml of distilled water and constantly stirred for 10 minutes to get homogeneous mixture. This reaction mixture was kept in a muffle furnace for 10 minutes maintaining the temperature of the muffle furnace at 400±10°C. The material was removed from muffle furnace, cooled to room temperature and the obtained dirty white colored powder sample was stored in airtight container till further usage [21].

D. Characterization

The UV-Visible spectrum of the synthesized ZnO NPs was measured by UV-2301 (Techcom) spectrometer. FT-IR spectrum was recorded organic and inorganic constituents in wavelength ranging from 500-4000 cm^{-1} using IS5 (Thermo Fisher). EDAX analysis was carried out in OXFORD XMX N. PXRD was done in a Panalytical X'pert pro MPD Cu-Kα using nickel filter (1.541Å). SEM analysis of synthesized nanoparticles was carried out by (TESCANVEGA3) instrument. The exact size of the nanoparticles was found through TEM (Jeol/JEM2100) instrument. The photocatalytic activity was done under UV light and the absorbance was measured.

E. Photocatalytic activity

The dye methylene blue was taken for the Photocatalytic activity study of the ZnO NPs synthesized using the leaf extract of *Sterculia foetida*. In this degradation process, 15 mg of ZnO nanoparticles was added to 10 mL dye solution of methylene blue. At a wavelength of 660 nm, the absorbance spectrum of the solution was monitored at different time intervals by using UV-Visible spectrometer.

III. RESULTS AND DISCUSSION

The UV peak was observed at 300 nm for ZnO nanoparticles, phytochemical constituents present in the *Sterculia foetida* leaf extract acts as an reducing agent during the formation of zinc oxide nanoparticles. The broad absorption peak at 300 nm indicates the reduction of Zn^{2+} ions that is confirmed the formation of zinc oxide nanoparticles portrayed in Figure-1a**.** The FT-IR spectrum was recorded for *Sterculia foetida* leaf extract mediated green synthesized ZnO NPs given in Figure-1b**.** The broad peak at 3350 cm^{-1} and 2800 cm^{-1} shows the stretching vibrations of N-H groups, which indicates the presence of amino group [22]. The peak at 1650 cm^{-1} assigned for the asymmetric stretching vibration of metal chelated carboxylic groups. The peak observed at 1300 cm^{-1} assigned for the asymmetric and symmetric stretching vibration of $-CH_3$ groups. The peak observed at 800 cm^{-1} confirms the presence of compounds such as poly phenols, carboxylic acid, amino acid and protein [23]. The Powder X-ray diffraction spectroscopy (PXRD) also confirms green synthesized ZnO NPs using *Sterculia foetida* leaf extract shown in Figure-2**.** The perceived reflection lines around at 2θ=31.8°, 34.47°, 36.25°, 47.4°, 56.6° and 68.0° were assigned to (100), (002), (101), (102), (110) and (103) respectively. In addition, the average crystallite size of ZnO NPs was calculated using the Debye-Scherrer formula [24].

$D=k\lambda/\beta\cos\theta$

Where D, k, λ, θ and β are crystalline size, shape factor, wavelength of the X-ray beam, diffraction angle and full width half maximum (FHHM) of the peak respectively. The calculated crystallite size was found to be 20.38 nm.

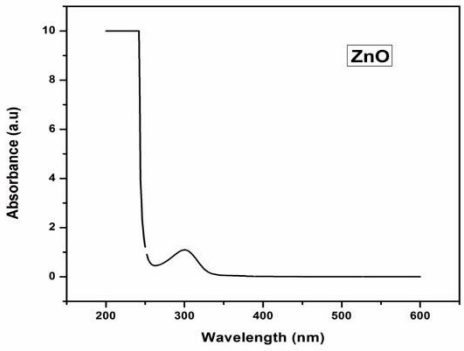

Figure-1a.UV-Vis spectrum of ZnO NPs

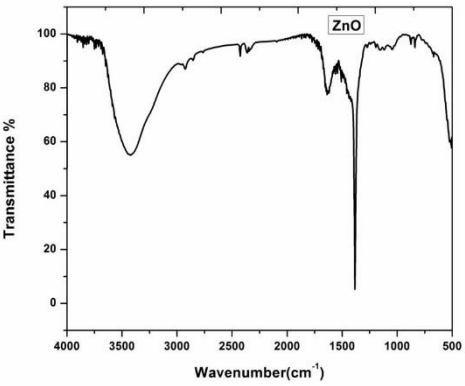

Figure-1b.FT-IR Spectrum of ZnO NPs

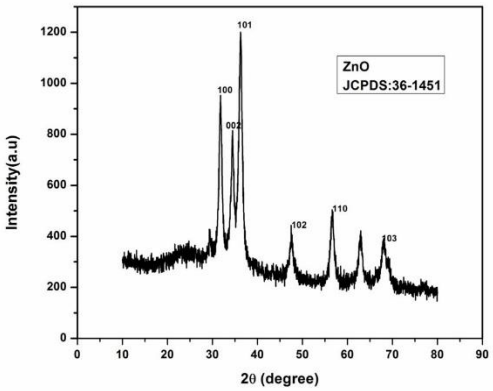

Figure-2.PXRD analysis of ZnO NPs

The EDAX spectra of green synthesized ZnO NPs using *S. foetida* leaf extracts are shown in Figure-3. The zinc and oxygen peaks present in the EDAX spectrum confirms the formation of ZnO NP. The involvement of phytochemical groups was indicated by the carbon peak [25]. The SEM images are shown in Figure-4**,** synthesized ZnO NPs are spherical in shape and they have shown a number of aggregations

of nanoparticles due to binding with plant extracts. TEM images are shown in Figure-5 which also portrayed the shape of the nanoparticles were in spherical with the size in range of 20-51 nm [26].

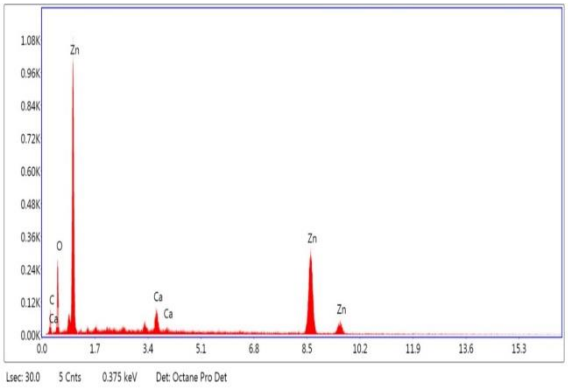

Figure-3. EDAX spectrum of ZnO NPs

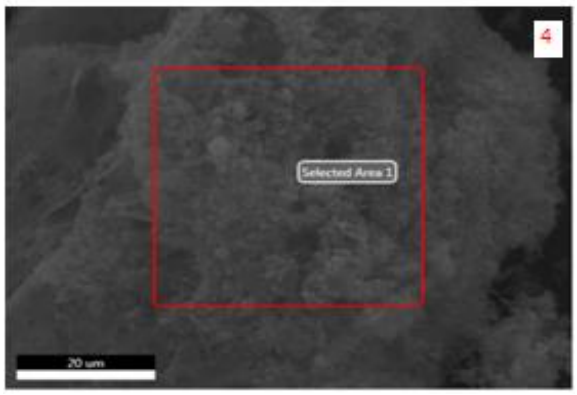

Figure-4 SEM image of ZnO NPs

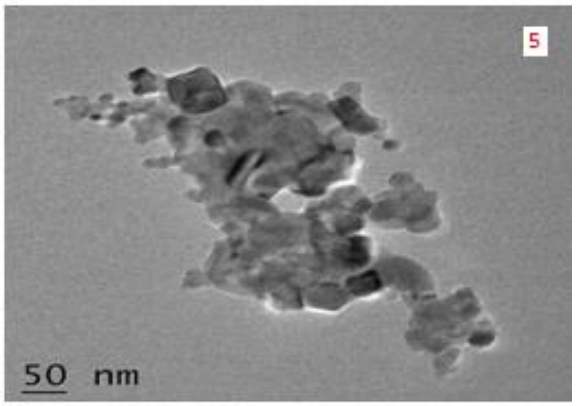

Figure-5 TEM morphological images of ZnO NPs

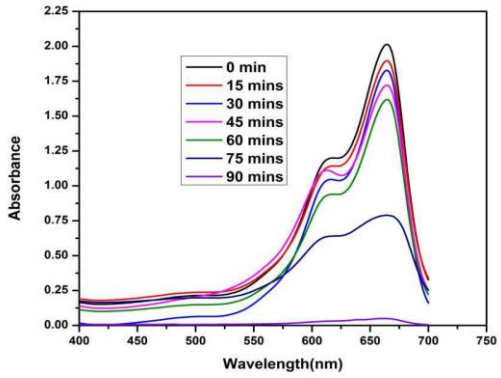

Figure-6.Photocatalytic activity of ZnO NPs for the degradation of methylene blue dye

Methylene blue (MB) dye was taken to evaluate the photocatalytic activity of green synthesized ZnO NPs using *S. foetida* leaf extracts. The absorption spectra of MB solutions in the visible light was recorded and shown in Figure-6. The absorption peak of MB was appeared at 660 nm. The peak becomes weaker as the irradiation time increases this indicates the degradation of the dye molecules in the aqueous solution. The degradation efficiency of the synthesized ZnO NPs against MB is calculated by using the following formula [27].

Degradation Efficiency $\%=C_o-C/C_o x100=A_o-A/A_o x100$ C_o is the initial concentration of the dye in the aqueous solution at the time t=0; C is the residual concentration at time t. Relative absorbance=A/A_o, the initial absorbance of the dye solution was represented as A_o and the absorbance after UV irradiation was represented as A.

IV. CONCLUSION

The solution combustion method has been successfully employed for the synthesis of ZnO NPs using *Sterculia foetida* leaf extract. The UV-peak observed at 300 nm confirmed the formation of ZnO NPs. The crystallite size of ZnO NPs was found to be 20.38 nm through PXRD. The agglomeration of ZnO NPs was found in SEM images. TEM images confirm the size of ZnO NPs are in the range of 20-51 nm. The degradation efficiency of green synthesized ZnO

NPs against methylene blue exposed their remarkable photocatalytic property.

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