Maghrebian Journal of
Pure and Applied Science
e-ISSN : 2458-715X

Maghr. J. Pure & Appl. Sci., 2023, Vol. 9, Issue 01, Page 01-08

https://revues.imist.ma/index.php/mjpas

https://doi.org/10.48383/IMIST.PRSM/mjpas-v9i1.41022

Synthesis of zinc oxide nanoparticles using *Dieffenbachia seguine* aqueous leaf extract for dye degradation

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***Received** 28 May 2023,*
***Revised** 19 June 2023,*
***Accepted** 20 june 2023*

***Citation**: Bankole-Ojo O. S., Oyedeji F. O. (2023) Synthesis of zinc oxide nanoparticles using Dieffenbachia seguine aqueous leaf extract for dye degradation, Maghr. J. Pure & Appl. Sci., 9(1), 01-08.*

Abstract: Dye pollution became a serious environmental and health challenge in many industrialized cities of the world. Natural dye degradation process takes a long time and thus, dye wastewater requires treatment before release into the environment. Zinc oxide (ZnO) nanoparticles were readily synthesized for photocatalytic dye degradation using phytochemicals from the leaves of *Dieffenbachia seguine* Linn. The phytochemicals present in the aqueous leaf extract of dumb cane or *Dieffenbachia seguine* (L) were used as reducing and stabilizing agents for the synthesis of ZnO nanoparticles (ZnONPs). The ZnONPs shows effective capping and stabilization by the phytochemicals as observed by the Fourier-Transform Infrared Spectroscopy. The particle size of the synthesized ZnONPs was studied using transmission electron microscopy. In this study, we used phytosynthesized ZnONPs for the degradation of Methylene Blue under UV Light. The ZnONPs photocatalyst showed excellent activity with over 95% of the dye degraded within 30 minutes. This highlights the strong potential of the ZnONPs as a photocatalyst for the treatment of dye wastewater.

Keywords: ZnO nanoparticles, *Dieffenbachia seguine*, Dye degradation, Phytosynthesis

1. Introduction

Nanottechnology is a relatively new branch of chemistry in which the small shape and size of particles determine its properties (Modi *et al*., 2022; Azzaoui *et al.* 2022). ZnO nanoparticles have particularly attracted significant attention because of their energy band gap of 3.36 eV and binding energy of 62 meV (Aldeen *et al*., 2022). ZnO nanoparticles have high surface to volume ratios (Ifijen *et al*., 2022). These nanoparticles exhibit unique properties that make them suitable for a wide range of applications like drug delivery, semiconductor diode, solar cells, and photocatalytic degradation (Islam *et al*., 2022; Shaba *et al*., 2021; Gómez-López *et al*., 2020). ZnO nanoparticles are already in use in commercial devices (Garcia *et al*., 2021). There are several methods used to synthesize ZnO nanoparticles. They include are biological, physical and chemical methods. Examples include hydrothermal (Asjadi and Yaghoobi, 2022), electro-deposition (Ruba *et al*., 2019), sol-gel (Arya *et al*., 2021), atomized method (Suhel *et al*., 2023), sonochemical (Noman *et al*., 2019), solvothermal (Wang *et al*., 2021), pulse laser spraying (Blažeka *et al*., 2022), physical vapour deposition (Saravanavel *et al*., 2021) chemical vapor deposition (Kulkarni *et al*., 2021), use of plant extracts (Mbenga *et al*., 2022).

Plants have phytochemicals that contain highly active biomolecules as alkaloids, terpenes, isoflavonoids, tannins and steroids that are used to cap and stabilize the nanoparticles to curb aggregation of the nanoparticles. Several extracts of green plants have been used as stabilizers and capping agents for the synthesis of ZnO nanoparticles (Dallatu *et al*., 2020). Several plants have been used for the synthesis of ZnO nanoparticles. They include such as *Azadirachta indica* (Saravanan *et al*., 2020), *Hisbiscus rosa-sinensis* (Chai *et al*., 2019), *Nephelium lappaceum* (Alfina *et al*., 2019), *Psidium guajava* (Zayed *et al*., 2021), *Nyctanthes arbor-tristis* (Rani *et al*., 2023), *Solanum nigrum* (Thomas *et al*., 2022), *Aloe Vera* (Primo *et al*., 2020), *Trigonella foenum-graecum* (Alshehri and Malik, 2019), *Boswellia ovalifoliolata* (Prasad *et al*., 2021), *Terminalia catappa* (Azimpanah *et al*., 2022), *Brassica oleracea* (Osuntokun *et al*., 2019), *Syzygium Cumini* (Sadiq *et al*., 2021). These biosynthesized nanoparticles have been known to exhibit properties such as Photoluminescence (Pramanik *et al*., 2022), good photocatalytic ability (Alprol *et al*., 2023), thus biological synthesized zinc oxide nanoparticles have been widely studied for the aforementioned properties (Kavitha *et al*., 2023; Zafar *et al*., 2023; Shinde *et al*., 2023).

Zinc oxide nanoparticles exhibit bioactivity such as antimicrobial activity against a wide range of bacteria (Abdelghany *et al*., 2023), these also have varied toxicological profile (Anand *et al*., 2023). Phytoassisted ZnO nanoparticles have been shown to exhibit good anticancer properties (Gatou *et al*., 2023). A very important property of ZnO nanoparticles is the degradation of dyes. Dyes such as methyl orange, methylene blue and methyl red have been degraded by ZnO nanoparticles (Dihom *et al*., 2022). The photocatalytic degradation of organic dyes by ZnO nanoparticles present an important opportunity to the elimination of dye wastewater into the environment.

Dieffenbachia seguine also known as dumb cane belonging to Araceae family ''*Dieffenbachia seguine*." linn is very popular as an ornamental plant in various homes across the globe. (Divya *et al*., 2022). It has become the symbol of indoor home plant decoration but its use for the preparation of therapeutics have been limited because it has been shown to be relatively toxic (Ummuhan and Kocabaş, 2020). In this study, we used the aqueous extract of dumb cane leaves for the preparation of ZnO nanoparticles using zinc acetate dihydrate as the precursor. The nanoparticles were used for the photocatalytic degradation of methylene blue (MB) dye.

2. Methodology

2.1 Preparation of aqueous plant extract and Characterization of ZnO nanoparticles

Zinc oxide nanoparticles were synthesized using the aqueous leaf extract of *Dieffenbachia seguine*. In this process, the aqueous plant extract was prepared by adding 20 g of the dried pulverized leaves to 100 mL of double distilled water. The mixture was heated at 100ºC for 30 minutes under reflux with vigorous stirring. The aqueous extract was obtained after filtration. The filtrate was centrifuged at 10,000 rpm at 15ºC for 10 minutes to remove the heavy biomolecules and prevent them from precipitating with nanoparticles during the purification process. The supernatant was obtained after decantation and filtered to separate any remaining particle.

2.2 Preparation of ZnO nanoparticles

25ml of the plant extract was heated and 100ml of 0.1M aqueous solution of 0.1M zinc acetate dihydrate was slowly added in a dropwise manner under intensive stirring for 30 minutes. The solution was then heated to 80ºC for 1 hour under reflux. The precipitate was obtained by centrifugation at 10 000 rpm for 15 min at 4°C. The nanoparticles obtained were washed using ethanol to remove adsorbed impurities. The dry powder obtained after drying at 60°C in a vacuum oven for 6 hours.

2.3 *Characterization of ZnO nanoparticles*

X-ray Diffraction (XRD) analysis was performed using a X-ray diffractometer with Cu Kα radiation (λ = 0.154 nm)) and an operating voltage of 30 mA, 45 kV. PXRD data were obtained with 2θ ranging from 10° to 70°. Absorption spectra were measured with a UV/visible spectrophotometer. Fourier Transform Infra-red (FTIR) spectra were measured on a Thermo Nicolet Nexus 670 spectrophotometer. Field Emission Scanning Electron Microsocope FESEM S4300 HITACHI at 10 kV coupled with Energy Dispersive X-ray Spectroscopy (EDX) equipment was utilized to study the size/morphology and the elemental composition of the ZnO nanoparticles.

2.4 *Photocatalytic degradation of the dye under solar irradiation*

The photocatalytic property of the phytosynthesized ZnO nanoparticles was evaluated under UV light irradiation via the photocatalytic degradation of methylene blue (MB) dye. The UV light source with a wavelength of 365nm was used. The concentration of MB in the solution was evaluated using Ultraviolet-visible light (UV-vis) spectrophotometer to measure the absorbance at a wavelength of 663nm. The efficiency of dye degradation was estimated by the equation below:

$$MB\ Degradation\ (\%) = \frac{(Co - Ct)}{Ct} x\ 100 \qquad (1)$$

Where Co (mgL^{-1}) and Ct (mgL^{-1}) are the concentrations of dye initially and at any time t respectively.

ZnO nanoparticles of masses 5mg and10mg were separately dissolved in 10ml deionized water to obtain a 0.5mg/ml and 1mg/ml concentrations respectively. The solution was agitated using a vortex mixer before the addition of 50μM MB. The photocatalytic degradation of MB was monitored by obtaining absorbance values at 0, 5, 10, 15, 20, 25, and 30 minutes. The reaction rate constant (Kt) was obtained in from Co and Ct using the equation below:

$$\ln \frac{Co}{Ct} = Kt \qquad (2)$$

3. Results and Discussion

3.1 *Characterization of ZnO nanoparticles*

The phytosynthesized ZnO nanoparticles (**Figure 1**) exhibited a broad frequency band at about 3572 cm^{-1} due to O-H stretching. The band at 2959cm^{-1} and 2420cm^{-1} are due to C-H stretching. The intense band at 1601cm^{-1} can be ascribed to C=C stretching vibration. The two absorptions peaks at 1400 cm^{-1} and 1122 cm^{-1} are attributed to the presence of CH_2 and CH_3 groups. Strong band observed at 700cm^{-1} correspond to Zn-O stretching modes similar to studies by (Manojkumar *et al*., 2023). Powder X-ray diffraction (PXRD) was performed to assess the crystalline nature, purity and crystallite size of the calcined ZnO nanoparticles (**Figure 2**). Prominent peaks corresponding to the planes (100), (002), (101), (102), (110), and (103) were observed. The sharp peaks showed that the ZnO nanoparticles were of a crystalline nature. All diffraction lines match closely with that of the standard data for the Joint Committee Powder Diffraction Standard (JCPDS) Data card number 82-1042 for hexagonal wurtzite structure of ZnO nanoparticles.

FESEM analysis was performed to gain further insight into the morphology and size of the ZnO nanoparticles (**Figure 3a**). Rod-like ZnO nanoparticles having an average width of particle size of 10 nm with sparse lumps of agglomerates. The EDX spectrum (**Figure 3b**) of calcined ZnO nanoparticles confirmed the presence of elemental Zinc, oxygen and carbon at 64.0 %, 23.2 % and 12.8 % by weight respectively. The carbon signal in the spectrum can be attributed to the use of phytochemicals during synthesis.

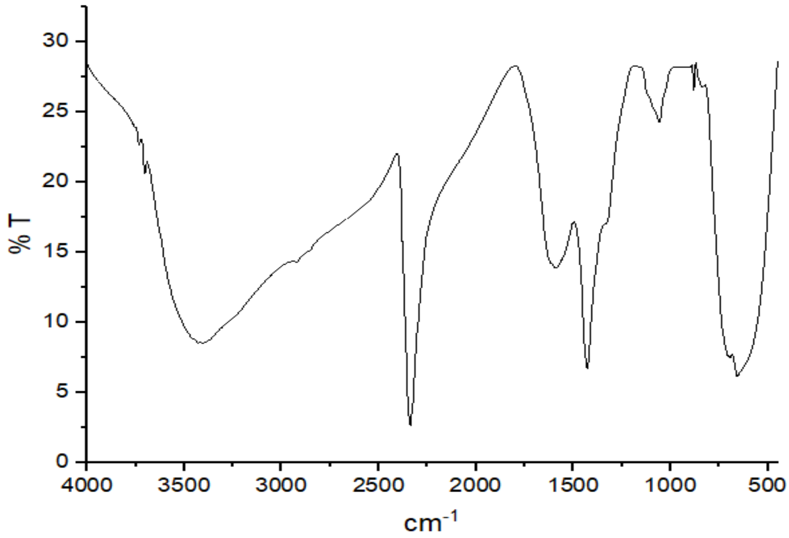

Figure 1. FTIR image of ZnO nanoparticles

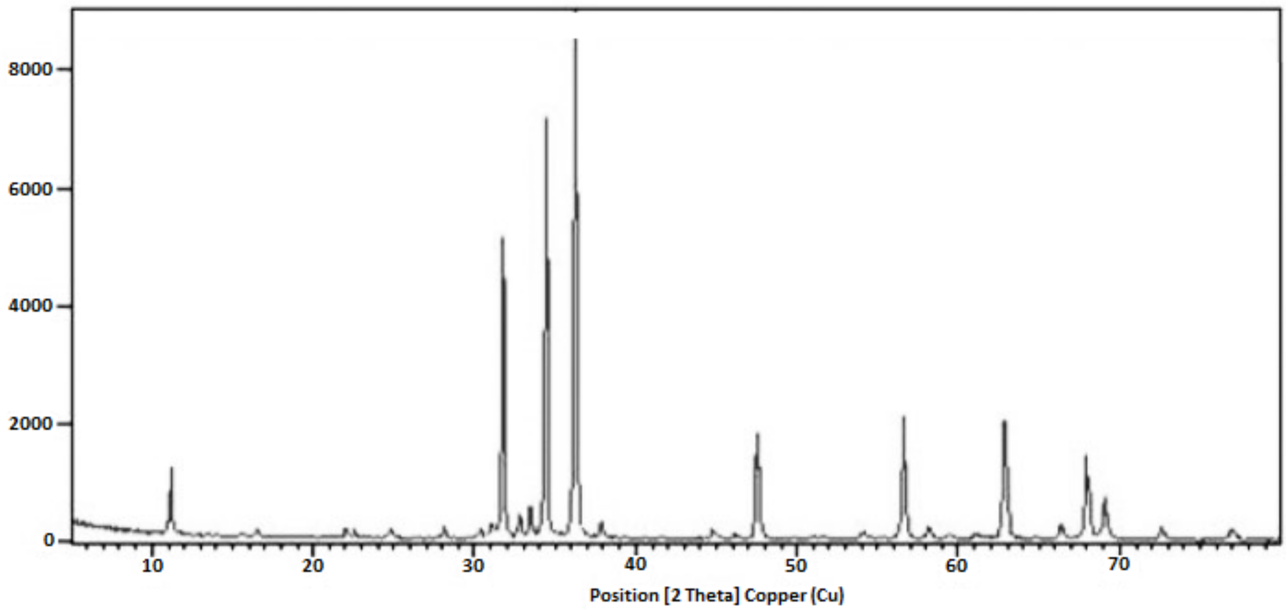

Figure 2. XRD pattern of ZnO nanoparticles

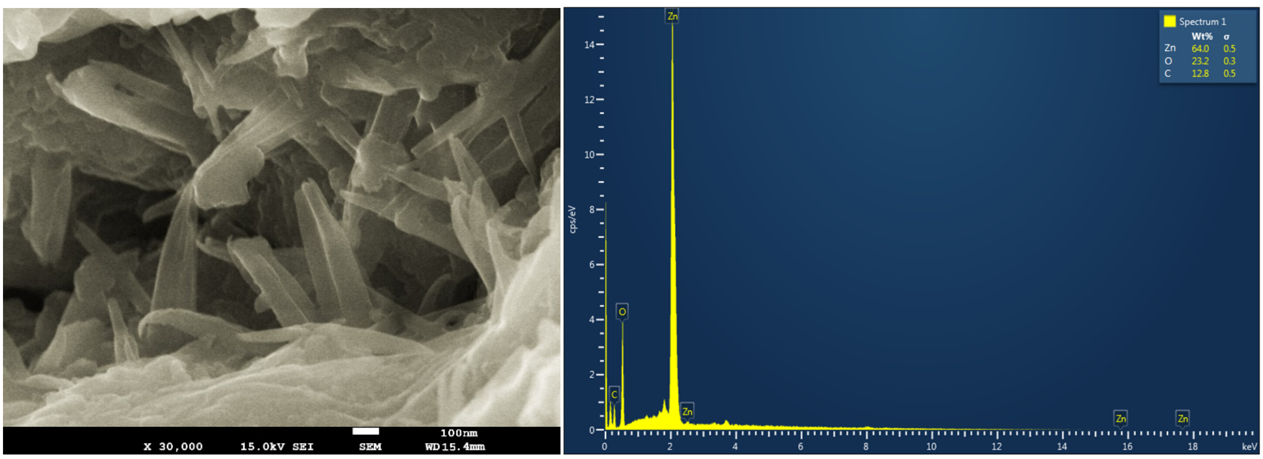

Figure 3. (a) FESEM image (b) EDX spectrum of ZnO nanoparticles

3.1 Dye degradation

The degradation of MB was monitored under UV light irradiation in the absence and presence of ZnO nanoparticles. The degradation of MB solution without the photocatalyst was evaluated as a control under the same reaction conditions as the degradation of MB in presence of the ZnO nanorods at concentrations of 0.5mg/ml and 1mg/ml. The image of the MB solution at the aforementioned time intervals in the presence of ZnO nanoparticles with concentration 0.5mg/mL is shown in **Figure 4**. It was observed that considerable transparency was achieved at 30 minutes for the 0.5mg/ml concentration. The % degradation as shown in **Figure 5a** indicates that slight degradation of MB under UV light occurred in the absence of a photocatalyst. Significant degradation of MB was observed in the presence of the ZnO nanoparticles. More than 50% degradation was achieved within 15 minutes while more than 90% of MB degradation was observed at 30 minutes for both the 0.5mg/ml and 1mg/ml concentrations. The reaction rate constant was determined from **Figure 5b**. A first order reaction with rate constant of 0.1199 min^{-1} was obtained for the phytosynthesized ZnO nanoparticles.

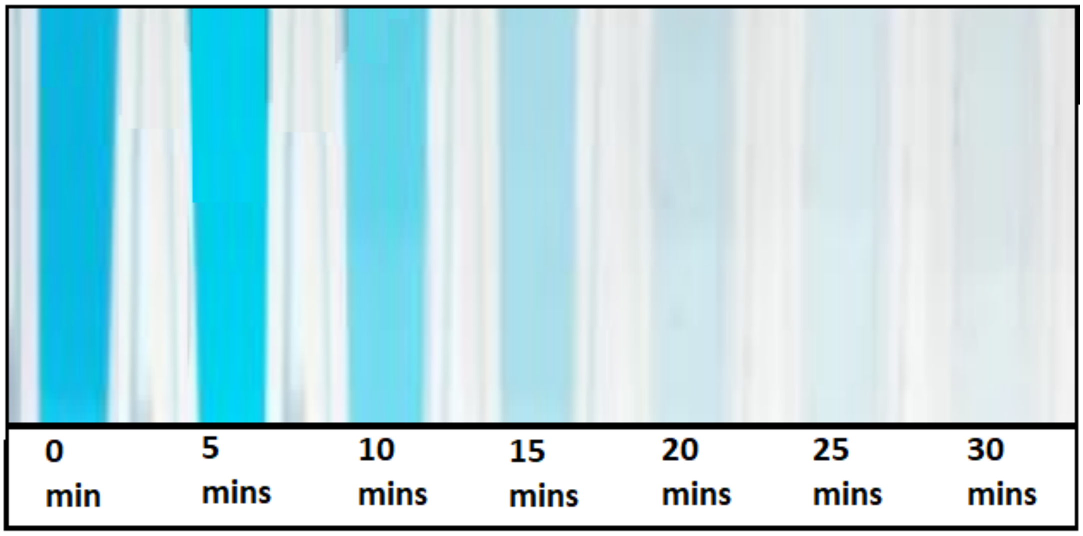

Figure 4. Visual illustration of methylene blue degradation in the presence of 0.5mg/ml ZnO nanoparticles.

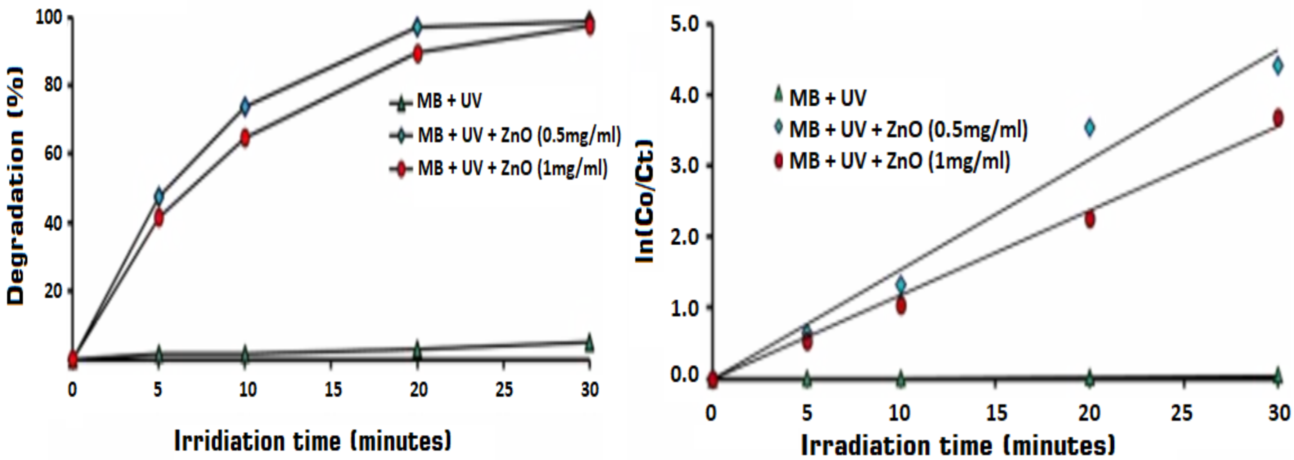

Figure 5. (a) Percentage dye degradation (b) Kinetic relationship of ln(Co/Ct) and time.

Conclusion

ZnO nanoparticles were synthesized using Dieffenbachia seguine aqueous leaf extract. The nanoparticles were characterized using UV-Vis, FT-IR, FE-SEM, XRD and EDX and the results showed that the ZnO nanoparticles possess rod-like morphology with crystalline nature and significant presence of adsorbed phytochemicals on the nanoparticle surface. Excellent photocatalytic degradation of MB was observed for the ZnO nanoparticles with over 95% of the MB dye degraded within 30 minutes at both concentration under the UV light irradiation. Therefore, the ZnO nanoparticles synthesized using phytochemicals from *Dieffenbachia seguine* aqueous leaf extract have a good potential for use in the photocatalytic degradation industrial dye wastewater.

Acknowledgement: The present research work was funded by CSIR-TWAS Postgraduate fellowship. The authors would like to thank the Director, CSIR-IICT and extend their appreciation to the entire staff and students of Polymers and Functional Materials division, CSIR-IICT, India for their support. Appreciation goes to the authorities of the University of Ibadan, Nigeria for permission granted to execute the research.

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.
Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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