Mustansiriyah Journal of Pure and Applied Sciences, Print ISSN: 2957-9910. Online ISSN: 2957-9929, Vol. 3, No. 3, 2025, Pages 18-28
DOI: 10.47831/mjpas.v3i3.283

MJPAS

MUSTANSIRIYAH JOURNAL OF PURE AND APPLIED SCIENCES

Journal homepage:
https://mjpas.uomustansiriyah.edu.iq/index.php/mjpas

RESEARCH ARTICLE - PHYSICS

Synthesis and Characterization of Zinc Oxide Nanoparticles Using Green and Chemical Methods for Photocatalytic Degradation Applications

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Article Info.

Article history:

Received
8 Augeust 2024

Accepted
16 October 2024

Publishing
30 June 2025

Abstract

Zinc oxide nanoparticles (ZnO NPs) were successfully synthesized by a green method using Cumin and Curcuma and zinc acetate dihydrate [Zn (CH3COO)2. 2H2O]as salt precursors. The particle size and phase structure of zinc oxide were determined by X-ray diffraction and field emission scanning electron microscopy. The produced zinc oxide nano particles are crystalline with a grain size of 23–30 nm and agglomerated form. Energy Dispersive Spectrum (EDX) results affect the composition of Zinc and Oxygen, giving high energy signals of 76.9.4% and 23.1% for Oxygen and zinc, respectively. (FT-IR) spectroscopic analysis shows the absorption peak of Zinc and oxygen Zn–O bonding between 400 and 600 cm^{-1}. The different characterization methods carried out prove the formation of nano Zinc oxide. The ZnO NPs were used as catalysts for the photodegradation of methylene blue under visible light radiation. The results indicate that the prepared ZnO material excellently removed MB methylene blue after 120 min, this green method of synthesis was found to be eco-friendly.

The official journal published by the College of Education at Mustansiriyah University

***Keywords**:* ZnO, photocatalytic degradation, Green synthesis, methylene blue.

1. Introduction

Progress in nanotechnology have led to fabricated of new nanosized materials with a range of applications, such as in nanomedicine consumer products, and nanoelectronics [1, 2]. For its their distinct physical and chemical properties compared to their bulk counterparts[3], these materials have been acquired great importance in Last few years[4]. Nanostructures of several metal oxides have been synthesized and have various applications in various fields. Zinc oxide has large band gap (3.3 eV) semiconductor. It has applications in gas sensors, dye degradation, solar cells, and more [5, 6]. ZnO nanoparticles have been synthesized using different physical and chemical methods, inclusive arc discharge [7] Sol–Gel [8] pulsed laser ablation [9] hydrothermal l [10]. Due to its advantages over physical and chemical methods used green synthesis method has been employed to synthesize zinc oxide nanoparticles, as it does not involve dangerous chemicals and is cost-effective. and eco-friendly [11, 12]. It uses biological materials which are easily extractable from plants cumin and curcuma were used as they are easily available. Triterpenic acids, tannins, flavonoids, alkaloids are found in phytochemicals present in this Cumin and Curcuma extract [13]. When plant materials are extracted, the content of polyphenols can be improved. The dried seeds were found to have polyphenols in the extract. It is well known that these polyphenols have strong antioxidant properties. Our findings imply that flavonoids were present in the plant seeds' aqueous extract, by chemical reduction these phytoconstituents act as stabilizing and reducing agents and synthesize metal oxide, these phytoconstituents create metal oxide nanoparticles and serve as stabilizing and reducing agents. Intensely colored Methylene Blue (MB) is a frequent water contaminant and is used in textile dyeing and printing. Dye impurities in events from the textile, printing, and industrial sectors contribute significantly to environmental pollution. This trash gets into the aquatic ecosystem, endangering the health and ecology of aquatic life and ultimately having an impact on human life. There are several methods for removing dyes from rivers and other bodies of water, including adsorption, osmotic pressure, coagulation, and others, but each has advantages and disadvantages of its own. Among the several methods used, photocatalytic treatment for dye removal offers a reasonably priced and environmentally beneficial solution to this issue. Due to its unique physical and chemical characteristics, ZnO nanoparticles, are indispensable nanoparticles used in many aspects of daily life. They have been widely used as additives in cosmetics, antibacterial agents, biomedical imaging, rubber, and textile industries [14-17]. Aside from their diverse uses in the field of catalysis, the present investigation involved the production of ZnO NPs through the utilization of a green synthesis approach, green methods are easy, cheap and environmentally safe methods that are manufactured using plant extracts. Aside from their diverse uses in the field of catalysis, the present investigation involved the production of ZnO NPs through the utilization of a green synthesis approach, green methods are easy, cheap and environmentally safe methods that are manufactured using plant extracts. after direct expose sun light to study photocatalytic degradation of methylene blue.

2.Experimental Procedure

Synthesis green methods: cumin and Curcuma, zinc acetate dehydrate [(CH3COO)2Zn.2H2O] and purity (99%) were used as a solvent. cumin first has been Rinse it with distilled water. After that, the curcuma was left to dry then Weigh 10 grams of cumin and Curcuma and mix them with deionized water. The aqueous extracts were obtained after boiling for 15 minutes, then left to cool and placed in different container. After cooling, the product was filtered through Filter paper and keep at 40° Even it was needed Another application.then (0.1M) zinc acetate was dissolution in 50 mL deionized water in a container and then placed on a magnetic stirer for 15 minutes until homogenous .then taken (10ml) cumin, (10 ml) curcuma, Both are merged with 10 mL of Zn (CH3COO) 2 2H2O solution in a beaker using a magnetic stirer for 15 minutes and then permissible to stand in the room temperature for 24 hours before the test.

Chemical method:

(0.1 mol) zinc acetate was dissolved in 50 ml of deionized water in a container and then placed on a magnetic stirer for 15 minutes until homogeneous, 3.1 gm of NaOH were taken and dissolved in 50 ml deionized water and added drop by drop to the zinc acetate solution on a magnetic stirer for 15 minutes to complete the dissolution process, then the solution was left for an hour to obtain a homogeneous solution. Figure 1 shows the steps of preparing zinc oxide. the solution was washed, filtered and dried at 100 C^o a for one hour to become ready for the required tests, to obtain the weight to be dissolved within the previous standard, the following relationship was used equ(1)[18]:

$M = (Wt/MWt).\ (1000/V)$ …………… (1)

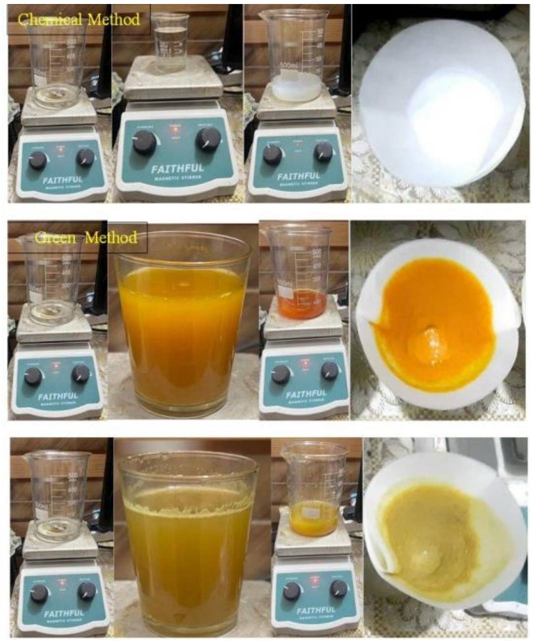

Figure :1 step preparing Zinc Oxide

Photocatalytic behavior of ZnO NPs

Due to their high photocatalytic activity, ZnO nanoparticles can be used as photocatalysts. under natural sunlight Photodegradation experiment was carried out and done in the June month at11:30 am the intensity of solar radiation was 13.654MJ, it was utilized a 50 mg L solution in water of methylene blue dye with a 5 mg/20 mL ZnO NP catalytic dose in this study. UV absorbance was measured at 0 minutes, 30 minutes, 60minutes, 90 minutes, and 120minutes after combining the two solutions (dye and catalyst).

2.Result and Discussion

Structural Characteristics

The X-ray diffraction patterns of ZnO nanostructures produced by green synthesis using cumin, curcuma extract and chemical method are offered in Fig. 2. The diffraction peaks at 2θ=31.769 °, 34.42 °, 36.25° and 56.6° correspond to the 100, 002, 101and 102 diffraction planes, respectively, presented in Table .1 and are related to ZnO with a hexagonal wurtzite structure in accordance with the Joint Committee on Powder Diffraction Standards (JSPDS Card number 00-036-1451). The highest intense (002) peak observed at 34.42° in the XRD patterns reveals that the ZnO nanostructures indicate preferential growth along the (100) direction.[12]. The crystallite size of ZnO nanostructures can be evaluated from the highest intensity peak (002) using the Scherrer formula. The calculated crystallite size was found ranging from (33,36 and 40) nm for cumin, curcuma and chemically, respectively using the Debye–Scherer Equation was obtained from the XRD spectrum [19-22]:

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \ldots\ldots. (2)$$

Where D is the ZnO crystallite size (nm), λ = 0.15418 nm, β is the full width at half maximum (FWHM) and θ is the Bragg angle. Films undergo dislocation and strain in their structure, from Equations (3) and (4), respectively these parameters were calculated [11].

$$\varepsilon = \frac{\beta \cos\theta}{4} \ldots\ldots\ldots\ldots\ldots\ldots (3)$$

$$\delta = \frac{1}{D2} \ldots\ldots\ldots\ldots (4)$$

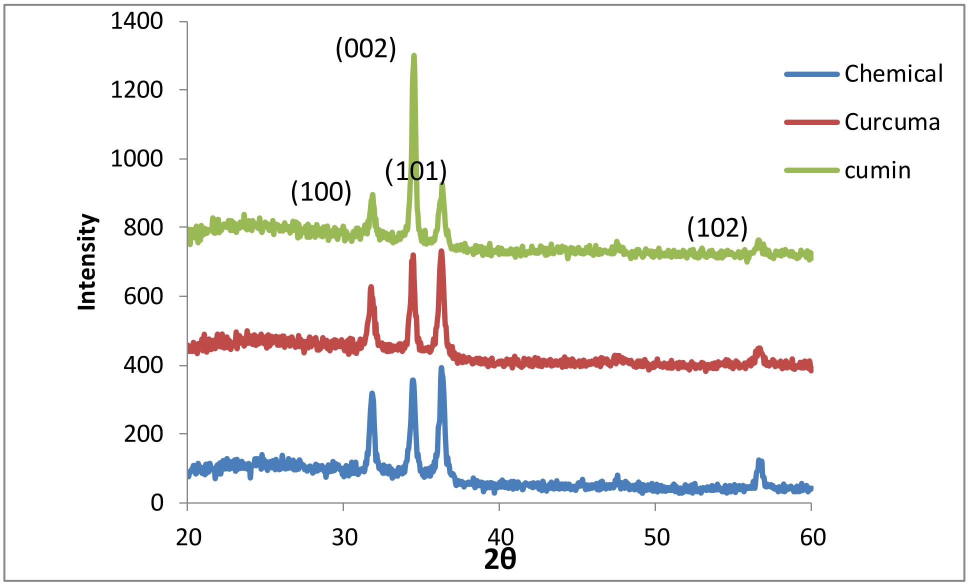

Figure 2: XRD pattern of as-synthesized ZnO using Cumin, Curcuma extract and chemical method

Table 1: Summary of X-Ray characterization for the ZnO

Crystal plane	2θ	FWHM	Crystallite size D(nm)	εx10^{14} lines.m^{-2}	δ *strain* X10^{-3}
Cumin extract					
(100)	31.76	0.643	23.83	1760.7	1.546
(002)	34.3	0.87	28.523	12.28	2.415
(101)	36.3	0.696	25.08	15.89	3.144
(102)	56.5	0.651	23.8	17.66	1.433
Curcuma extract					
(100)	31.68	0.437	23.4	18.2	1.05
(002)	34.325	0.384	24.6	16.5	0.917
(101)	36.24	0.427	23.19	18.5	1.14
(102)	56.58	0.69	25.06	15.9	1.518
Chemical method					
(100)	31.76	0.437	30.83	12.7	0.71
(002)	34.3	0.385	30.60	17.361	0.81
(101)	36.3	0.437	28.13	17.36	1.49
(102)	56.5	0.624	23.06	30.36	1.98

Compositional analysis and Surface Morphology

Scanning electron microscopy images were taken at various magnifications to examine the size and shape of the nanoparticles synthesized, as shown in Fig. 3(a, b, c). The surface morphology accentuates the formation of nanoparticles in their agglomerated shape. Different literatures indicate the influence of surface morphology and its relationship to the characteristic activity of zinc oxide [13]. Procedure (a), using Cumin extract, (b) using Curcuma extract and (c), by chemically method

It was found rugged surface, majority of particles are horizontal in shape, also agglomerated form of some nanoparticles is seen to be spherical and others are irregular in shape the average size of nanoparticles found to be 24 nm for sumin as shown in Fig. 5a, 25 nm using Curcuma extract Fig .5b and 28 nm with chemical methos Fig .5c, as indicated by the X- ray diffraction (XRD) result [15].

green synthesized ZnO nanoparticles EDX measurement result is shown in Fig. 3 (a, b, c) using instruments EDX the element analysis of green synthesized zinc oxide nanoparticles. Chemical purity and stoichiometry of the specimens were investigated. The EDX spectrum confirms the presence of Zn and O ions in zinc oxide nanoparticles synthesized using Cumim and Curcuma extract and Chemical methods.

The elemental analysis detected 70.7% of zinc and 29.3% of oxygen for sample using Cumin, 76.9% of zinc and 23.1% of oxygen using Curcuma,46%9of Zinc and 28.1% of oxygen, for chemical method respectively all suggesting that the ZnO has perfect purity and very few other elements can be seen. Fig.3(a, b, and c) provides evidence of the existence of minute quantities sodium and Carbon alongside zinc and oxygen as Impurities [23].

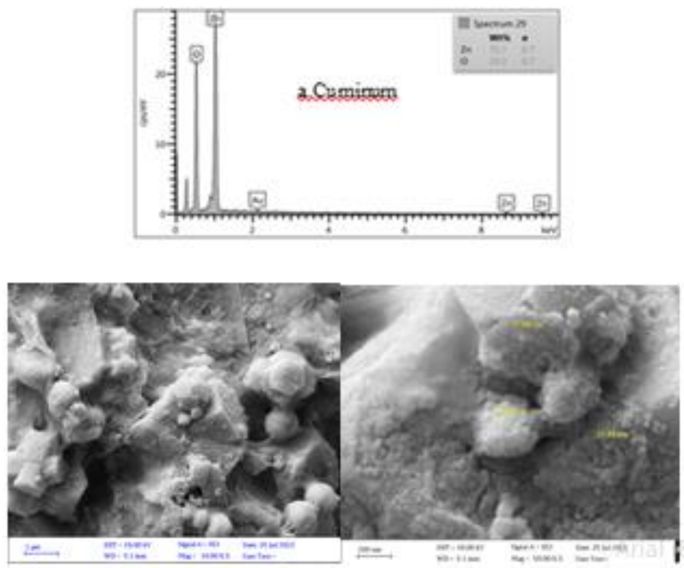

Figure 3a: SEM images and EDX spectra of ZnO prepared by cumin extract technique**.**

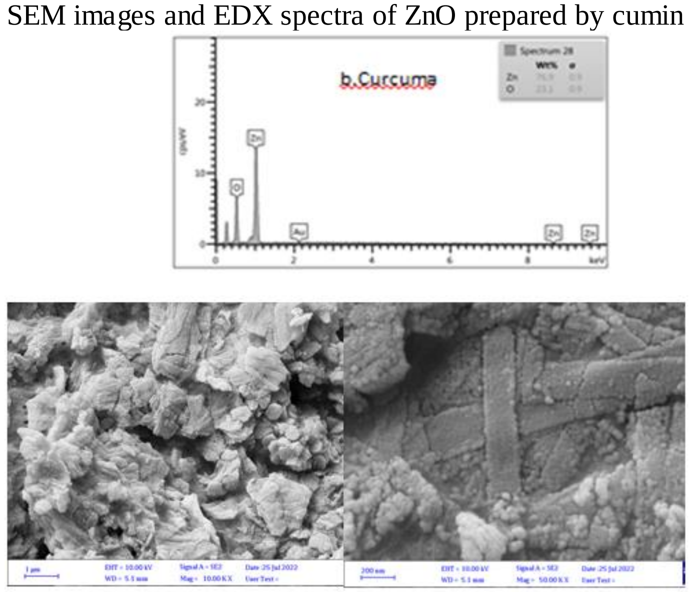

Figure 3b: SEM images and EDX spectra of ZnO prepared by curcuma extract technique.

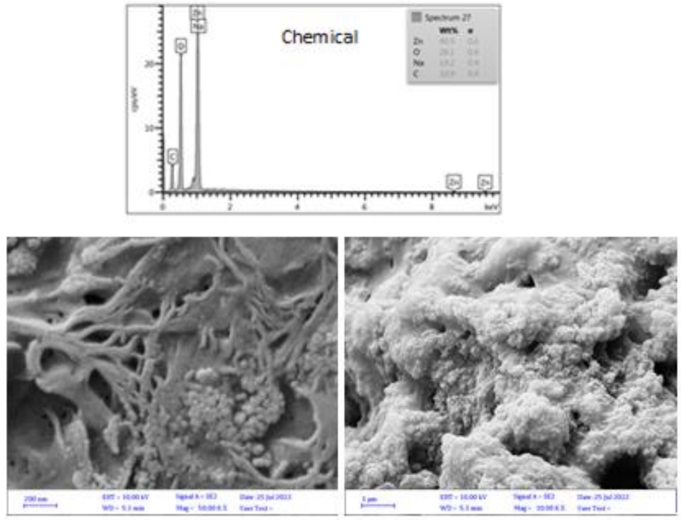

Figure 3c: SEM images and EDX spectra of ZnO prepared by chemical method.

FT-IR analysis

To certain the formation presence of Zn–O bond and to identify the phytoconstituents FT-IR spectroscopy measurements are performed using an FTIR spectrometer .Fig. 4(a, b, c) shown FT-IR spectrum of green synthesized ZnO-NPs, The spectral peaks 3655 cm^{-1} and 3797 cm^{-1} are due to O–H stretching vibration of the hydroxyl group from the water adsorbed , The peak about 1565 cm^{-1} is due to the C=O stretching , The peak around 2359 cm^{-1} is due to C–H stretch ,The peaks at 1725 cm^{-1} corresponds to ZnO bending distortion vibrations. The intense vibrational bands at 663 cm^{-1} are assigned to the stretching mode for the formation of zinc oxide nanoparticles [24, 25].

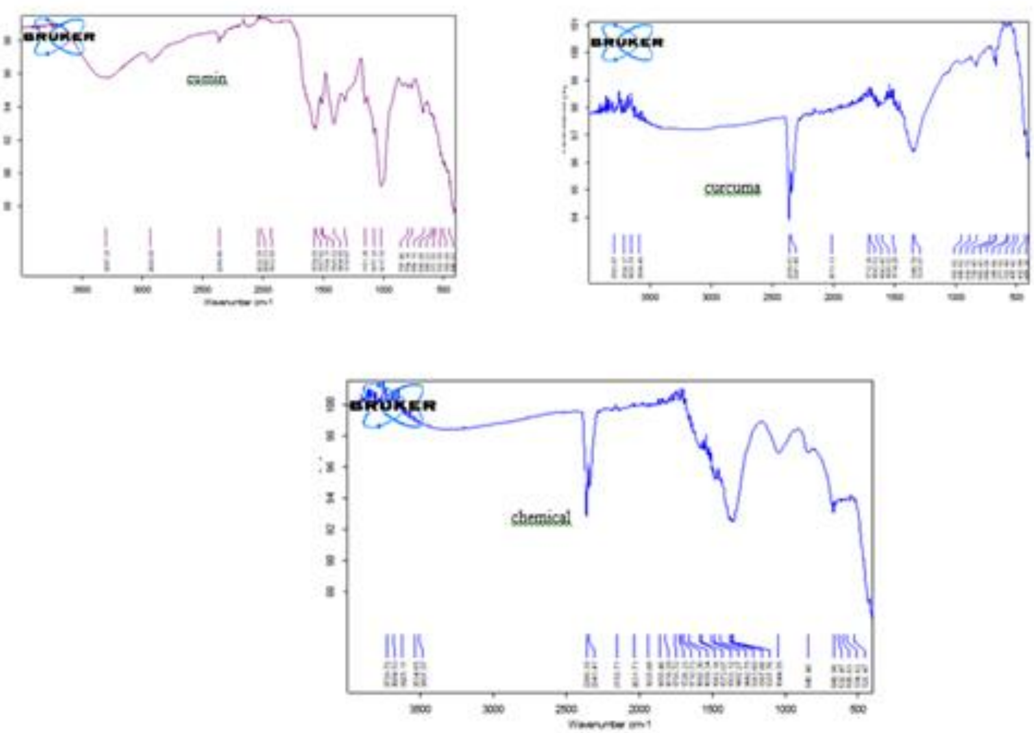

Figure 4**:** FT-IR spectra of as-synthesized ZnO using(a) cumin, (b) curcuma, and (c)chemical method**.**

Optical Properties

Cumin and Curcuma extract is highly rich in numerous phytochemicals especially phenolics and flavonoids (ellagic acid, and luteolin), functionalization and Size properties for nanoparticles carry a private role in changing the properties of materials [14].

ZnO NPs generally show UV absorption bands in the λmax ranges from300 to 355 nm Figure 5. Shows the absorption intensity for zinc oxide, measured in the wavelength range from 300 to 900 nm. Zinc oxide nanoparticles prepared using the chemical method of cumin and Curcuma extract showed a maximum at 308 nm, which is confirming by Literature [15, 16] The band gap energy of ZnO NPs was found to be (2.4, 3.15, 3.35) eV for cumin, curcuma extract and chemical method respectively as calculated from plot in Figure 6, which is similar to the band gap energy reported for ZnO (wide band gap 3.10 – 3.39 eV) [19, 20]. These results primarily certain the formation of ZnO NPs following our approaches and it was observed that the energy gap depends on the size and increases with decreasing crystal size of the ZnO nanostructure and is also affected by surface defects.

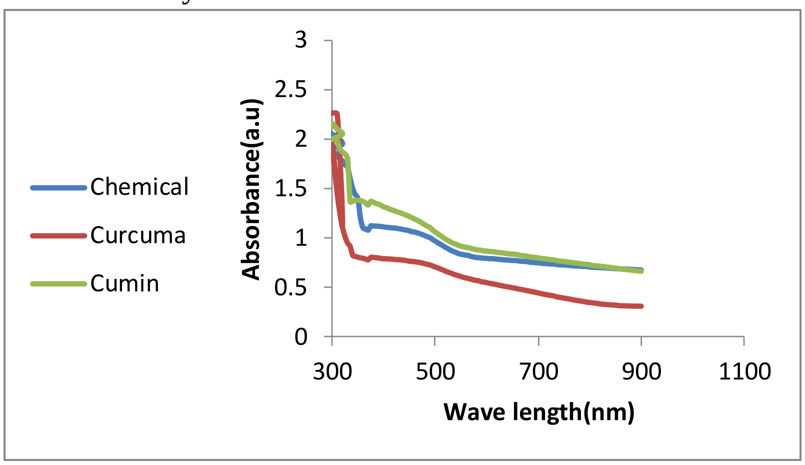

Figure 5: Absorbance of as-synthesized ZnO using Cumin, curcuma and chemical method

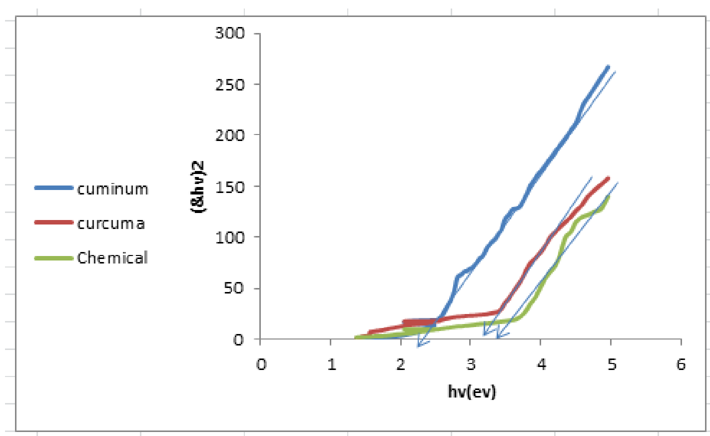

Figure 6: $(\alpha h\upsilon)^2$ for ZnO NPs with photon energy

Photocatalytic activity of ZnO

The effectiveness of as-synthesized ZnO NPs on the degradation of methylene blue (MB), a typical model water contaminant. The photocatalytic degradation of a methylene blue solution under sunlight for samples using cumin, curcuma extract and [Zn (CH3COO)2. 2H2O]. Nanoparticle photocatalytic activity is affected by a different of parameters including Radiation intensity, size and surface area. The solution put on the magnetic stirrer in the dark for 60 min to equilibrate the adsorption. Subsequently, the samples were illuminated by sunlight analyzed by UV-vis Fig. 7.

Fig. 8 (a, b, c) illustrates the absorbance spectrum of ZnO nanoparticles prepared using sumin, curcuma and chemical method and shows the photocatalytic dye degradation activity of NPs by using MB. Following 120 min, the absorbance decreased, suggesting that ZnO NPs degraded the dye for three samples.

Spectroscopy result showed with maximum adsorption wavelength of MB of 660 nm respectively [15, 16, 22]. The best photocatalytic dye degradation using Curcuma extract at wavelength 660 nm.

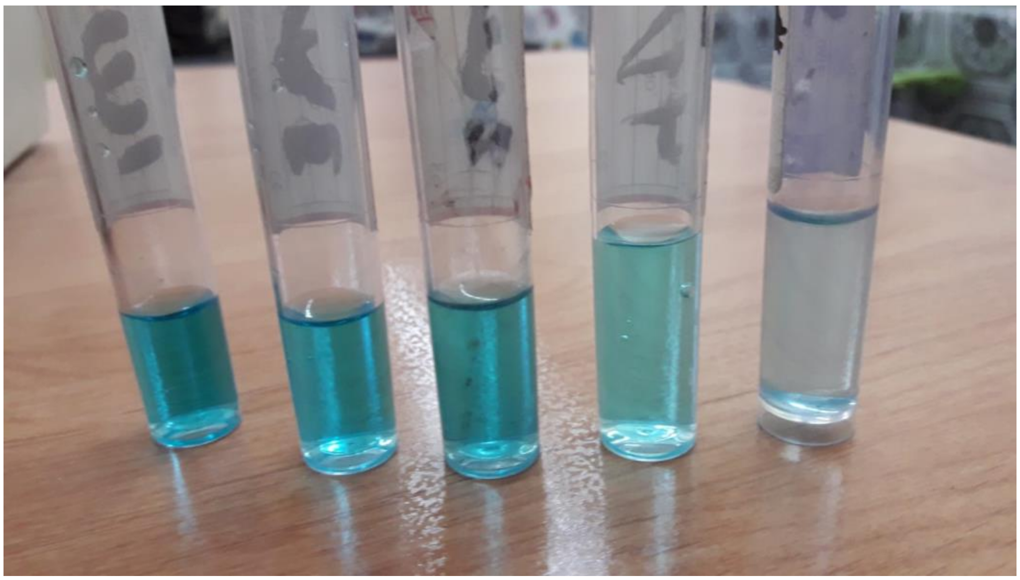

Figure 7: stages Decomposition of methylene blue dye

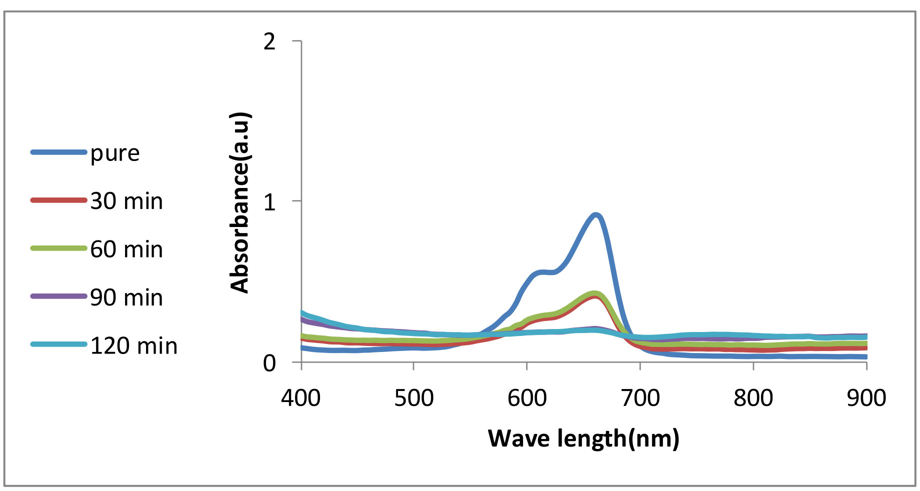

Figure 8a: Absorption as a function of wavelength for degradation of MB blue using Cumin extract

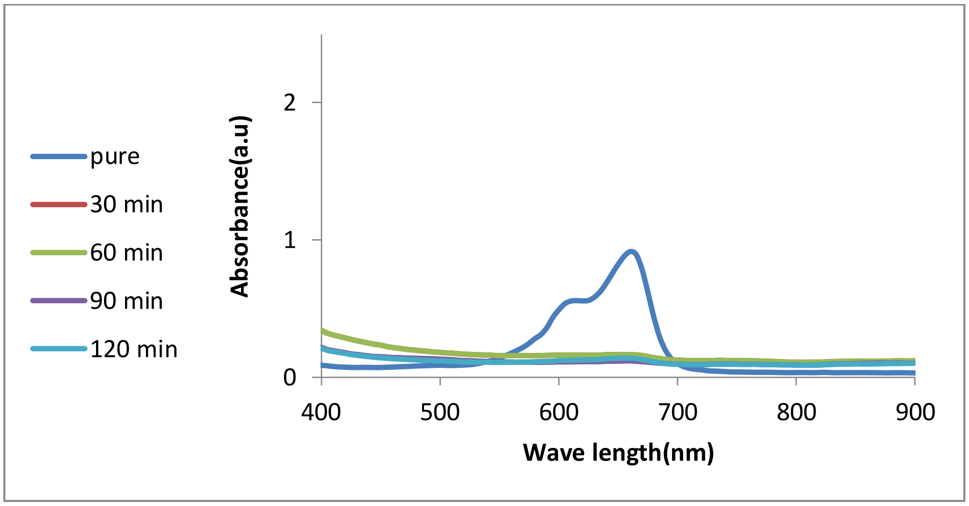

Figure 8b: Absorption as a function of wavelength for degradation of MB blue using Curcuma extract

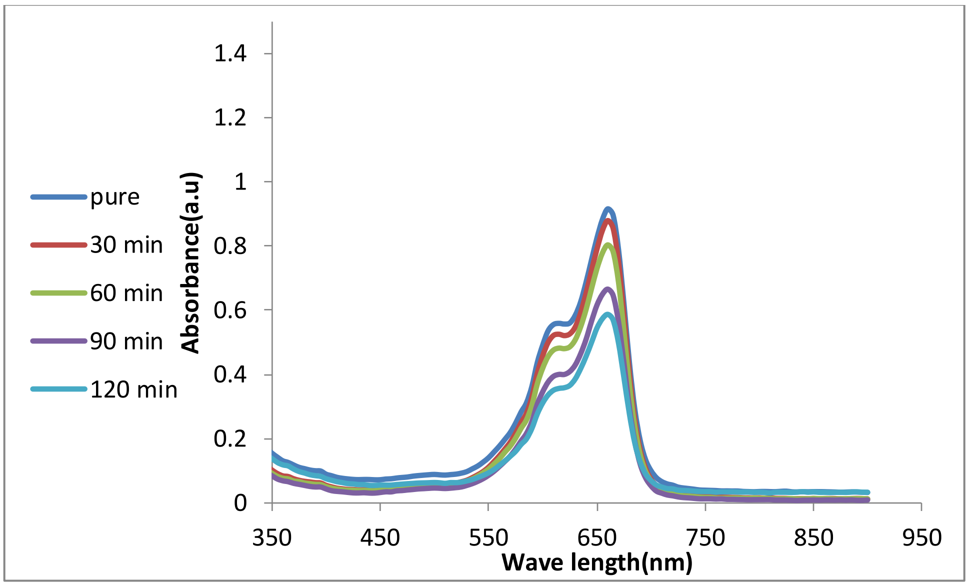

Figure 8c: Absorption as a function of wavelength for degradation of MB blue using chemical method

4. Conclusion

ZnO nanoparticles were synthesized by a green method using Cumin and Curcuma extracted and chemically. ZnO has the hexagonal wurtzite structure form and ZnO NPs size in the range of 23-30 nm. This was revealed by XRD diagram. energy gap of synthesized ZnO was (2.4, 3.15, 3.35) eV for Cumin, Curcuma extract and chemically respectively, Zinc Oxide NPs confirm degraded a methylene-blue solution of 50 mg/L for 120 min under sun light and showed activity in dye degradation (MB) greater by using Curcuma extract at wavelength 660 nm than that of cumin and chemical method. Thus, this study signifies a simple cheap and eco-friendly way to synthesize ZnO-NPs from Cumin and Curcuma extracted, and how to use them as a green photocatalyst for practical uses.

Acknowledgment:

The author thanks the Department of Physics, College of Science - Mustansyriah University for providing its laboratory facilities. We would like to thank everyone for their participation in any manner in this initiative.

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