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ZnO Nanoflowers Synthesized by *Terminalia catappa* Leaf Extract and Its Characterizations

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ZnO nanoparticles have been synthesized using *Terminalia catappa* leaf extract and their characteristics. The properties of ZnO nanoparticles were characterized by UV-Vis spectroscopy, scanning electron microscopy (SEM), and X-ray diffraction (XRD). The highest absorbance peak is in the ultraviolet region, which is in the wavelength range of 300-350 nm. The hexagonal wurtzite crystal structure of ZnO has been confirmed by XRD analysis with the highest reflection peak at a reflection angle of $2\theta = 36.26°$, with a crystal plane (101). SEM characterization showed the presence of nano-flower morphology with a size distribution of 400-494 nm. The results of this study show that ZnO nanoparticles can be applied as photocatalysts for the degradation of methylene blue.

Keywords: Zinc Oxide, Nano-flower, *Terminalia catappa*.

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1. INTRODUCTION

The development of ZnO nanoparticles has attracted researchers because it has an energy gap of 3.37 eV with a binding energy of 60 MeV [1] for application in electronics devices [2], photocatalysts, the cosmetic industry , even as antibacterial [3], antifungal [4]. The uniqueness of the properties and application of ZnO nanoparticles depends on their synthesis technique [5]. There are many methods to synthesize ZnO nanoparticles using chemical and physical ways. However, that method is more expensive, carried out at high temperatures and pressures, and uses toxic chemicals [4]. In this study, green synthesis of ZnO nanoparticles was carried out using *Terminalia catappa* leaf extract because it was considered more environmentally friendly, and the process was simple.

Microwave oven has been used to preparation methods on yield and creat fungsional materials obtained from biomass and environment waste materials. Plant extracts have been reported for the biosynthesis of ZnO nanoparticles, such as Bush Tea (*Athrixia phylicoides* DC.) [1], *Capsicum chinense* Fruit Extract [5], *Cassia fstula* and *Melia* azadarach. [6], *Myristica fragrans fruit extracts* [7], *Scoparia Dulcis* [8], and *Trifolium pratense* flower extract [9]. Terminalia catappa is one of the plants that can be used as a bioreductor. Terminalia catappa leaves contain secondary metabolite compounds, flavonoids, alkaloids, saponins, quinones, and phenolics. Tannin compounds contained in *Terminalia catappa* leaves are phenolic compounds that are polymerizations of simple polyphenols. The flavonoid and phenolic content obtained from *Terminalia catappa* leaf extract have a molecule that can reduce metals in the formation of nanoparticles [10].

2. EXPERIMENTAL

The Zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}4H_2O$) as Zn precursor and sodium hydroxide (NaOH).

The location of the *Terminalia catappa* leaf in this study comes from the botanical garden University of Riau, Indonesia. Distilled water was used as the solvent in all experiments. First, the leaves of Terminalia catappa are dried at 100 °C for 90 minutes. After that, the dried leaves are mashed to form a powder with a smooth size. About 5 g of *Terminalia catappa* powder was dispersed in 200 mL of distilled water to obtain an extract concentration of 50 mL. The solution stirred uses a magnetic stirrer for 10 minutes at 80 °C to increase the polyphenol content. *Terminalia cattapa* leaf extract is further cooled at room temperature, filtered using whatman no 1 filter paper, and then stored in a dark vial at 4 °C for further use in the ZnO nanoparticle biosynthesis process.

$Zn(NO_3)_2{\cdot}4H_2O$ with a concentration of 0.05 M was prepared by dissolving 1.3 grams of $Zn(NO_3)_2{\cdot}4H_2O$ in powder form and then dissolved using 100 mL of distilled water in a 250 mL erlenmeyer then stirred until the solution was thoroughly mixed. The synthesis of nanoparticles was carried out by the biosynthetic method by reacting *Terminalia catappa* leaf extract as a bio-reductant with a previously prepared zinc solution. The synthesis of ZnO nanoparticles was carried out by ratio the sample volume of the *Terminalia catappa* leaf extract solution and the $Zn(NO_3)_2{\cdot}4H_2O$ 0.05 M solution, was 1:5. The NaOH solution, which plays a role in regulating acidity is dripped into the synthesized solution until it reaches pH 12. The synthesized solution is stirred until it is homogeneously mixed using a magnetic stirrer. After that, the solution was heated on a hot plate at 60 °C for one hour to produce a white precipitate. The resulting white precipitate was then separated using a centrifuge with a rotational speed of 4000 rpm for 10 minutes. The resulting ZnO nanoparticle powder was dried on a hot plate at 150 °C until it was dry and ready for characterization.ZnO nanoparticles were characterized using UV-Vis spectroscopy, X-ray diffraction (XRD), and electron microscope scanning characterization (SEM).

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3. RESULTS AND DISCUSSION

After the synthesis process, a beige-coloured precipitate appeared by heating the solution for 1 hour on a hotplate at various temperatures of 80 °C while stirring using a magnetic stirrer. Fig. 1(d) shows ZnO nanoparticles. The Zn ion reacts with the hydroxide ion from the *Terminalia. The ZnO nanoparticles catappa* leaf extract, resulting in a cloudy light brown solution that, after centrifugation, changes colour to a beige-coloured precipitate. Fig. 1(e) ZnO nanoparticles powder dry with colour and ZnO nanoparticles with a flower-like morphology photo characterized by SEM.

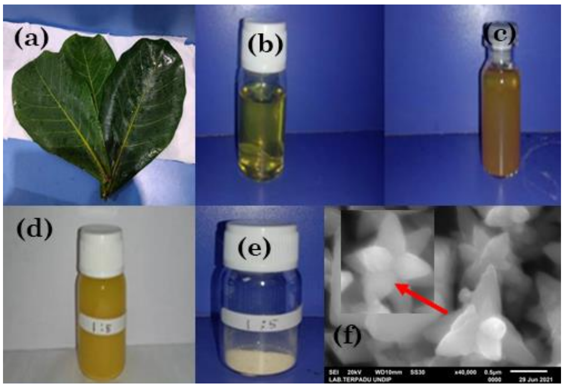

Fig. 1 – (a) *Terminalia catappa* leaf (b) *Terminalia catappa* extract colour before adding NaOH, (c) after adding NaOH, (d) biosynthesis of ZnO nanoparticles after one hour of stirring, and (e) ZnO nanoparticle powder, and (f) ZnO nanoparticles with a flower-like shape

UV-vis spectra of ZnO nanoparticles that had been synthesized using *Terminalia catappa* leaf extract and ZNH precursor in a ratio of 1:5 using a UV-vis spectrometer at a wavelength of 300-600 nm. The absorbance spectrum is shown in Fig. 2. ZnO nanoparticles are transparent in the absorption range in the ultraviolet region. The presence of peaks at 347 nm shows maximum red-shifted absorption, proving the formation of ZnO nanocrystals. That peak confirms the formation of ZnO nanoparticles due to the transition of electrons from the valence band to the conduction band [11]. The ZnO nanoparticles energy band gap was 3.29 eV calculated using the Tauc plot, where results similar to the ZnO energy gap band reported in the literature of 3.10-3.39 eV [12, 13]. These findings confirm that the leaf extract of *Terminalia catappa* is a bioreductor that acts as a reducing agent.

Crystal structure and size of the ZnO sample observe from the X-Ray Diffraction (XRD) characterization. The results of the X-ray diffraction pattern of the ZnO sample are presented in Fig. 3. The peak of diffraction lies at the diffraction angle as well as its crystalline plane of 31.75° (100); 34,43° (002); 36,26° (101); 47,58° (102); 56.63° (110) and 62.88° (103) are phases of the hexagonal Wurtzite ZnO.

The crystal plane indexing is based on the Crystallography Open Database (COD) database No. 96-210-7060 using match 3! software. However, there are several diffraction peaks in addition to the ZnO phase. ZnO sample crystal size can be calculated using

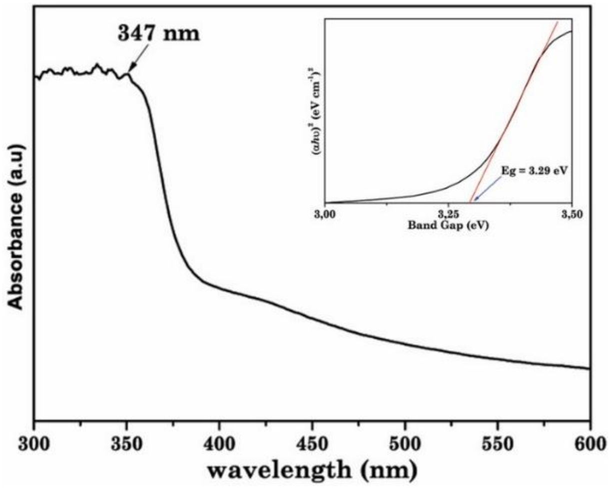

Fig. 2 – UV-Vis spectra of ZnO nanoparticles synthesized using *Terminalia catappa* leaves extract

Debye Scherrer equation, which is known to be full width half maximum (FWHM) magnitude. The FWHM value is taken from the highest diffraction peak in the crystal plane (101) with an FWHM value of 0.63. The crystal size of the ZnO sample was 14.48 nm. ZnO nanoparticles size synthesized from *Terminalia catappa* leaf was lower than *Vitex negundo* leaf at 38.17 nm [14].

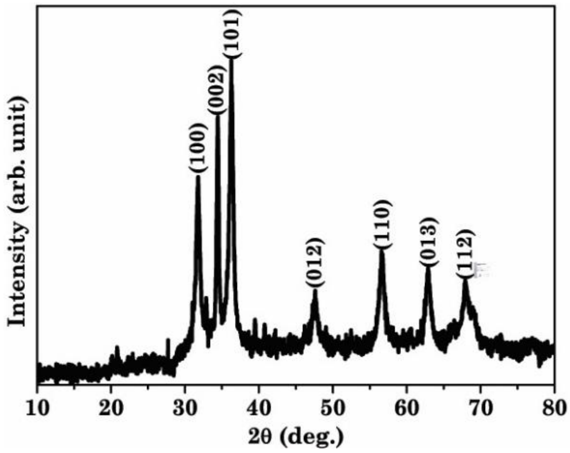

Fig. 3 – XRD pattern of the ZnO nanoparticles synthesized using *Terminalia catappa* leaves extract

The morphology of the surface of the ZnO sample synthesized using a mixture of zinc solution and Terminalia catappa leaf extract in a ratio of 1:5 is shown in Fig. 4. The characterization using SEM with magnifications of 4.000 and 40.000× shows the surface morphology of the flower-shaped ZnO sample with six petals. Fig. 1(a) the morphological form of homogeneous ZnO nanoparticles (4000× magnification). Fig. 4 (b) shows a photo of the surface of the ZnO nanoparticle uniquely shaped micro-flower visibly composed of 6 petals similar to the porcelain flower ornamental plant.

The morphology produced by synthesis using *Terminalia catappa* leaves is the same as that of the results of synthesis using pineapple peel extract (Ananas comosus) [15]. The particle size of ZnO was

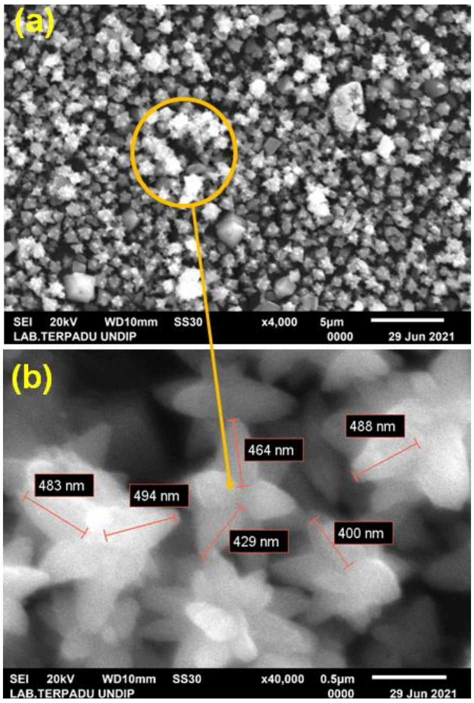

Fig. 4 – SEM images ZnO nanoparticles synthesized by using *Terminalia catappa* leaves extract (4.000×) and (b) grain size of biosynthesis ZnO with porcelain flower-like shape (40.000×)

measured using IC Measure software which displayed the ZnO nanoparticle size of 400-494 nm obtained by adjusting the scale indicated by SEM characterization, as shown in Fig 4. The increasing agglomeration of crystal size indicates that the *Terminalia catappa* extract is important in particle size.

4. CONCLUSSIONS

The biosynthesis of ZnO nanoparticles performed using *Terminalia catappa* leaf extract gave the result of SEM characterization with flower-shaped morphology. Uv-vis spectroscopic absorbance spectrum with a high absorption rate in the wavelength range of 300-380 nm. The crystalline phase of ZnO obtained from XRD characterization is the wurtzite hexagonal phase. Based on the analysis of physical properties above, the biosynthesis of ZnO nanoparticles uses *Terminalia catappa* leaf extract potential for photocatalysts.

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Наночастинки ZnO, синтезовані за допомогою екстракту з листя *Terminalia catappa* та його характеристики

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Наночастинки ZnO були синтезовані з використанням екстракту листя *Terminalia catappa*. Властивості наночастинок ZnO були охарактеризовані методом УФ-видимої спектроскопії, скануючої електронної мікроскопії (SEM) та рентгенівської дифракції (XRD). Найвищий пік поглинання припадає на ультрафіолетову область, яка знаходиться в діапазоні довжин хвиль 300-350 нм. Гексагональна кристалічна структура вюрциту ZnO була підтверджена аналізом XRD з найвищим піком відбиття при куті відбиття $2\theta = 36.26°$ з площиною кристала (101). Характеристика SEM показала наявність морфології нанoквітки з розподілом розмірів 400-494 нм. Результати цього дослідження показують, що наночастинки ZnO можна застосовувати як фотокаталізатори для розкладання метиленового синього.

Ключові слова: Оксид цинку, ZnO, Наночастинка, *Terminalia catappa*.