

Biosynthesis of zinc oxide tri-doping selenium (Se), sulfur (S) and silver (Ag) using matoa leaf extract for methyl orange photocatalyst

Alia Hanafia Fadli*, Ari Sulistyo Rini, Rahmi Dewi

Department of Physics, Universitas Riau, Pekanbaru 28293, Indonesia

*Corresponding author: alia.hanafia7022@grad.unri.ac.id

ABSTRACT

Water pollution caused by synthetic dye waste from the textile industry, particularly methyl orange, is a serious environmental problem due to its carcinogenic properties and difficulty in degradation. This study aims to synthesize tri-doped ZnO nanoparticles with Se, S, and Ag using a biosynthetic method based on matoa leaf extract (*Pometia pinnata*) with the aid of microwave irradiation and to test their photocatalytic activity. The samples were synthesized with a fixed Se concentration of 4%, S varied from 1% – 2%, and Ag varied from 1-3% at pH 8 using 540 Watt microwave irradiation for 3×1 minute. UV-Vis characterization showed a band gap energy of 3.34 – 3.60 eV with absorbance peaks in the range of 344.92 – 372.18 nm. XRD analysis confirmed a hexagonal wurtzite crystal structure with a crystal size of 24.97 – 37.34 nm. FESEM displayed a quasi-spherical morphology with a particle size of 107.69 – 1562.02 nm. EDX confirmed the presence of Zn, O, Se, S, and Ag elements in the sample. BET characterization showed the highest surface area in the (0,1,4)Ag,S,Se/ZnO sample at 16,298 m²/g with a type IV mesoporous structure. Photocatalytic testing against methyl orange under UV-C irradiation showed consistent degradation for 120 minutes. This study proves that the biosynthesis of tri-doped ZnO using matoa leaf extract produces an effective and environmentally friendly photocatalyst material for industrial waste treatment.

Keywords: Biosynthesis; matoa leaf; methyl orange; photocatalyst; ZnO

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INTRODUCTION

Water pollution from synthetic dye waste from the textile industry is a serious environmental and health problem. Methyl orange is an azo dye that is carcinogenic and difficult to degrade naturally due to its benzene group [1]. Conventional processing methods such as chlorination, adsorption, and ozonation have limited effectiveness and high operational costs.

Semiconductor photocatalysis is emerging as an environmentally friendly, efficient, and low-cost alternative [2]. ZnO is a semiconductor material with a bandgap energy of 3.37 eV and an exciton binding energy of 60 meV, making it promising for photocatalytic applications [3]. However, pure ZnO has limitations because it only absorbs 5% of the solar spectrum in the UV range.

Doping strategies with Se, S, and Ag have been developed to improve photocatalytic efficiency. Selenium can narrow the band gap and enhance photocatalytic activity under visible light [4]. Sulfur can modify the structure and increase the degradation rate by up to 99.66% [5]. Silver, as an electron acceptor, can reduce the recombination of electron-hole pairs [6].

The biosynthesis method using plant extracts is an environmentally friendly approach that produces smaller nanoparticles [7]. Matoa leaves (*Pometia pinnata*) contain high levels of phenolic compounds that can act as reducing and stabilizing agents [8]. This study aims to synthesize tri-doped ZnO nanoparticles with Se, S, and Ag using matoa leaf extract-based biosynthesis with microwave irradiation and to test their photocatalytic activity against methyl orange.

LITERATURE REVIEW

ZnO Nanoparticles

Nanoparticles are particles with dimensions under 100 nm that exhibit unique physicochemical properties different from macroparticles [9]. Zinc oxide (ZnO) is a group II – VI semiconductor with a band gap energy of 3.37 eV and an exciton binding energy of 60 meV. ZnO has high chemical and mechanical stability and is abundant in nature [10].

ZnO Crystal Structure

The ZnO crystal structure is divided into three phases: cubic rocksalt, cubic zinc blende, and hexagonal wurtzite. The wurtzite structure is the most thermodynamically stable at room temperature, where each Zn atom is tetrahedrally coordinated with four O atoms [11]. This structure has lattice parameters $a = b \neq c$ with angles $\alpha = \beta = 90^\circ$ and $\gamma = 120^\circ$.

Electronic and Electrical Properties

Pure ZnO is an n-type semiconductor with a direct band gap of about 3.3 eV at room temperature. The electronic configuration of Zn is [Ar] $3d^{10} 4s^2$ and that of O is [He] $2s^2 2p^4$. In ZnO, Zn is in the +2 oxidation state and O is in the -2 state. The electrical properties of ZnO can be modified through doping, structural changes, or synthesis conditions [4].

Doping of ZnO

Doping is the process of adding impurities to an intrinsic semiconductor to modify the number of charge carriers. Commonly used doping agents for ZnO come from group III (B, Al, Ga, In), group IV (Sn, Pb), group VI (S, Se), and group IB (Cu, Ag, Au) [12].

Selenium (Se)

Selenium (Se) is a group VI element with atomic number 34 and exhibits cationic (Se^{4+} , Se^{6+}) and anionic (Se^{2-}) characteristics. Se

doping in ZnO can create structural defects that enhance photodegradation efficiency by: (1) narrowing the band gap to the visible region, (2) increasing electron-hole separation, and (3) facilitating reactive oxygen species formation due to its high reduction potential [4, 13].

Sulfur (S)

Sulfur is one of the best dopants for modifying the structure and optical properties of ZnO [5]. S doping has good photoabsorption properties and can accelerate photodegradation activity. Studies have shown that ZnO:S can degrade methylene blue up to 99.66% in 150 minutes [14, 15]. The molar ratio of S to Zn affects the structure, morphology, luminescence, and photocatalytic properties of ZnO.

Silver (Ag)

Silver is a Group 11 transition metal with the highest electrical and thermal conductivity. Ag is used as a dopant due to its high reduction potential, allowing it to act as an electron acceptor and reduce electron-hole recombination [6]. The optimal concentration of Ag added to ZnO is 3 – 5 mol%. Studies have shown that Ag-ZnO can degrade Rhodamine B by 85% – 90% in 240 – 300 minutes [16, 17].

Matoa Leaves

Matoa leaves (*Pometia pinnata*) are endemic to Southeast Asia, particularly Papua, Indonesia. Matoa leaves contain high levels of antioxidant compounds such as flavonoids, tannins, saponins, and glycosides [8]. The high phenolic content makes matoa leaves a potential bioreductant and biostabilizer in nanoparticle synthesis because they can produce smaller and more stable particles [18].

Synthesis Method

Biosynthesis

Biosynthesis is an environmentally friendly method for synthesizing nanoparticles that

utilizes plant extracts or microorganisms as reducing and stabilizing agents. This method produces smaller particles, is low-cost, fast, and less polluting than conventional chemical or physical methods [7, 19].

Microwave Method

The Microwave-Assisted Method (MAM) utilizes electromagnetic wave energy (300 MHz – 300 GHz) to cause molecular movement. This method is superior to conventional heating because heat is distributed evenly throughout the sample through two mechanisms: (1) dipolar polarization—molecular dipoles align with the direction of an oscillating electric field, and (2) ionic conduction—ions move back and forth, creating friction [20, 21]. MAM produces nanoparticles with good crystallinity, a narrow size distribution, and controlled morphology [5].

ZnO Photocatalysis

Photocatalysis is a combined process of photoreaction and catalysis to accelerate reactions using light. When ZnO is irradiated with an energy greater than the bandgap energy, electrons are excited from the valence band to the conduction band, producing electron-hole pairs (e^-/h^+). The holes react with H_2O or OH^- to produce hydroxyl radicals ($\bullet OH$), while electrons react with O_2 to produce superoxide radicals ($O_2\bullet^-$). These radicals degrade organic pollutants into CO_2 and H_2O [22, 23]. A limitation of ZnO is its wide bandgap (3.37 eV), which absorbs only 5% of the solar spectrum (UV). Therefore, modification through doping is necessary to increase photocatalytic efficiency below visible light.

Methyl Orange

Methyl orange is an azo dye widely used in the textile industry with a bright orange structure. This dye is highly dangerous due to its carcinogenic and mutagenic properties due to the benzene group in its structure [1]. Methyl

orange is difficult to degrade naturally and can inhibit light penetration in aquatic ecosystems even at low concentrations [24]. pH affects the adsorption process - high pH favors the adsorption of methyl orange because the surface becomes negatively charged [25].

Characterization Techniques

UV-Vis Spectroscopy

UV-Vis spectroscopy measures the absorbance or transmittance of light in the range of 200 – 1100 nm to determine the optical properties of a material. The band gap energy is calculated using the Tauc plot method with the equation:

$$(\alpha h\nu)^2 = c(h\nu - E_g) \quad (1)$$

where h is Planck's constant, ν is the frequency, and E_g is the band gap energy [26, 27].

X-Ray Diffraction (XRD)

XRD is used to analyze crystal structure, phase, and crystal size based on X-ray diffraction according to Bragg's law:

$$2d \sin \theta = n\lambda \quad (2)$$

Crystalline size is calculated using the Debye-Scherrer equation:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (3)$$

where D is the crystal diameter, K is a constant (0.9 – 1), λ is the X-ray wavelength, β is the FWHM, and θ is the diffraction angle [28, 29].

FESEM-EDX

Field Emission Scanning Electron Microscopy (FESEM) provides high resolution (magnification up to 300,000×) to analyze the surface morphology of nanoparticles. Energy Dispersive X-ray (EDX) is integrated into

FESEM to analyze the qualitative and quantitative elemental composition [30, 31].

Brunauer-Emmett-Teller (BET)

BET measures the specific surface area and porosity of a material based on the adsorption-desorption of N₂ gas. Type IV isotherms indicate a mesoporous structure. Pore size distribution analysis using the Barrett-Joyner-Halenda (BJH) method [32, 33].

RESEARCH METHODS

Materials and Synthesis

The materials used include matoa leaves, zinc nitrate hexahydrate (Zn(NO₃)₂·6H₂O), selenium (Se), sodium sulfide nonahydrate (Na₂S·9H₂O), silver nitrate (AgNO₃), sodium borohydride (NaBH₄), sodium hydroxide (NaOH), and methyl orange. Matoa leaf extract was prepared by boiling 2 grams of leaf powder in 100 mL of DM aqua at 100°C for 10 minutes, then filtered using Whatman No. 1 paper.

The precursor solution was prepared by dissolving 11.899 grams of Zn(NO₃)₂·6H₂O in 80 mL of DM aqua (0.5 M). Se (4%), S (1-2%), and Ag (1% – 3%) solutions were prepared with a concentration of 0.5 M. All solutions were mixed with 20 mL of matoa leaf extract and the pH was adjusted to 8 using 5 M NaOH. The mixture was irradiated using a microwave at 540 Watt for 1 minute three times. The precipitate was sonicated, centrifuged (2000 rpm, 10 minutes), washed, and dried at 110°C.

Characterization and Photocatalyst Test

The samples were characterized using UV-Vis (optical properties), XRD (crystal structure), FESEM-EDX (morphology and composition), and BET (surface area). The photocatalyst test was carried out by adding 0.010 grams of sample into 50 mL of 10 ppm methyl orange solution, stirred for 1 hour, then irradiated with four 8 Watt UV-C lamps.

Sampling was carried out every 20 minutes for 120 minutes and absorbance was measured using UV-Vis.

RESULTS AND DISCUSSION

Optical Properties

The UV-Vis spectrum shows a strong absorbance peak at 300 – 390 nm (UV region) and a weak one at 390 – 600 nm (visible light). The highest absorbance peak is found in the (1,1,4) Ag,S,Se/ZnO sample and the lowest in the (2,2,4) Ag,S,Se/ZnO sample, confirming the formation of ZnO nanoparticles [34].

The band gap energy ranges from 3.34 – 3.60 eV, with some samples showing lower values than commercial ZnO (3.37 eV). The decrease in the band gap energy is due to the influence of doping and quantum confinement effects [35]. Se doping plays a key role in narrowing the band gap and enhancing photocatalytic activity under visible light [36]. The addition of Ag can shift the absorption, but remains optimal under UV light [37].

Crystal Structure

The XRD pattern shows eight sharp peaks corresponding to COD no. 9004179, confirming the hexagonal wurtzite crystal structure [38]. The addition of Ag and S doping does not change the crystal structure, but causes peak broadening and a decrease in intensity, indicating a reduction in crystal size and reduced crystallinity [39].

The crystallite size on the (111) plane ranges from 24.97 to 37.34 nm. The (3,1,4) Ag,S,Se/ZnO sample has the largest crystallite size (37.34 nm), and the (2,1,4)Ag,S,Se/ZnO sample has the smallest crystallite size (24.97 nm). The shift in the diffraction peak is caused by the larger ionic radii of Se and S than O, as well as their intercalation into the crystal lattice [38]. Too high a doping concentration of Ag can cause not all atoms to integrate into the ZnO crystal structure [40].

Morphology and Elemental Composition

FESEM shows a quasispherical morphology with a particle size of 107.69 – 1562.02 nm. The shape is close to spherical but not perfect, with irregularities such as flattening, ovality, or protrusions due to the high dopant concentration. Matoa leaf extract acts as a capping agent that controls nanoparticle growth [38].

The largest particle size was found in the (1,1,4)Ag,S,Se/ZnO sample (1562.02 ± 47.44 nm) and the smallest in the (0,0,4)Ag,S,Se/ZnO sample (107.69 ± 1.66 nm). The addition of Ag doping reduced the particle size, consistent with the research of Kumar et al. (2021), which showed that Ag controls the growth of ZnO particles [4]. The EDX spectrum confirmed the presence of Zn, O, Se, S, and Ag in the samples. The composition of Zn ranged from 44.96% – 50.63% atoms, O 48.08% – 53.51% atoms, Se 0.06% – 1.29% atoms, and Ag 0.74% – 2.16% atoms. The presence of Se and S reduces charge recombination, increasing the photocatalytic efficiency, as observed in methyl orange degradation tests.

Surface Area and Porosity

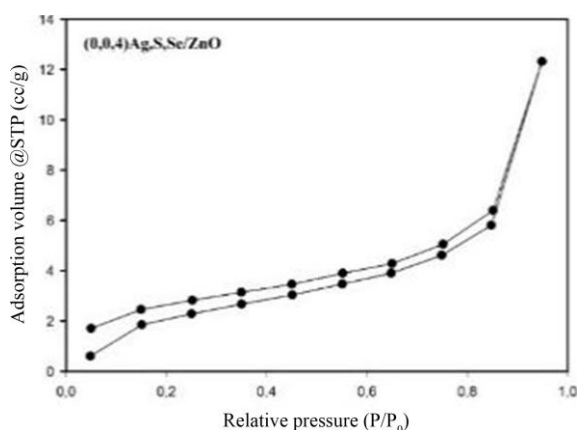


Figure 1. Ag,S,Se/ZnO isotherm.

Type IV BET isotherms indicate a mesoporous structure [33]. The (0,1,4)Ag,S,Se/ZnO sample had the highest surface area ($16.298 \text{ m}^2/\text{g}$) and the largest pore volume (0.036 cc/g). The addition of 1% S doping resulted in a larger surface area

compared to other tri-doping variations, but the addition of up to 3% Ag resulted in a drastic decrease ($0.863 \text{ m}^2/\text{g}$).

These results align with FESEM, where particle size is inversely proportional to surface area [33]. A large surface area is important for pollutant degradation because it increases the number of active sites [41], making the Ag,S,Se/ZnO biosynthetic sample excellent for application as a photocatalyst.

Photocatalytic Activity Test

The absorbance curve showed a consistent decrease every 20 minutes for 120 minutes, indicating successful methyl orange degradation. The (1,2,4)Ag,S,Se/ZnO sample showed a significant decrease in absorbance. UV-C light with energy higher than the band gap directly hit the sample, accelerating the degradation process [42]. Figure 2 shows the absorbance curve of methyl orange degradation.

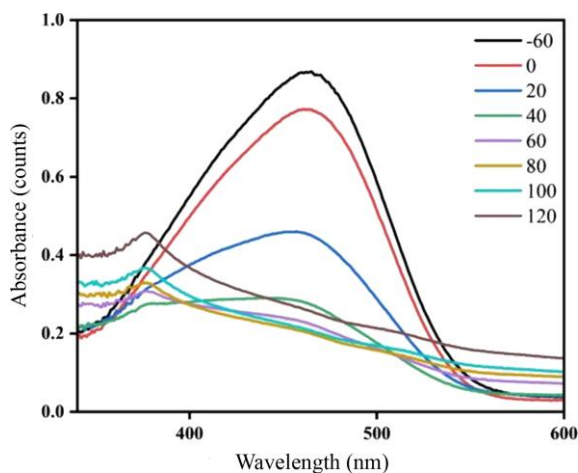


Figure 2. Absorbance curve in the degradation process of methyl orange Ag,S,Se/ZnO.

The color change of the solution from yellow to clear indicates successful degradation. The decrease in the absorbance peak at 470 nm indicates a decrease in the concentration of methyl orange. The presence of Se and S in the ZnO structure reduces charge recombination, increasing photocatalytic activity [43]. At 120 minutes, there was a slight increase in absorbance due to the formation of intermediate products.

CONCLUSION

Tri-doped ZnO nanoparticles of Se, S, and Ag have been successfully synthesized using matoa leaf extract-based biosynthesis with microwave irradiation. Characterization confirmed the hexagonal wurtzite crystal structure with a band gap energy of 3.34 – 3.60 eV, a crystal size of 24.97 – 37.34 nm, and a quasispherical morphology. The presence of Se, S, and Ag elements was confirmed by EDX. The sample with 1% S doping showed the highest surface area (16.298 m²/g). The photocatalytic test demonstrated the material's ability to degrade methyl orange for 120 minutes under UV-C irradiation. This study proves that biosynthesis using matoa leaf extract is an environmentally friendly and effective method to produce tri-doped ZnO photocatalytic materials.

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