

Zn–Catharanthus roseus and Cu–Catharanthus roseus Complexes as Green Organometallic Photosensitizers for DSSC Applications

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ABSTRACT

The global energy crisis and climate change issues have driven the development of environmentally friendly renewable energy sources, one of which is the dye-sensitized solar cell (DSSC) utilizing natural pigment-based photosensitizers. *Catharanthus roseus* (tapak dara) was selected due to its abundance of secondary metabolites and its potential to be modified into Zn and Cu nanoparticles to enhance stability and efficiency. In this study, *tapak dara* leaf extract was obtained using ethanol through ultrasonic extraction, then synthesized into Zn and Cu nanoparticles with Zn:Cu ratios of 0:4, 1:3, 2:2, 3:1, and 4:0 via green synthesis. The resulting nanoparticles were deposited onto TiO₂ electrodes, with Pt used as the counter electrode in the DSSC. The cells were tested under 9 mW halogen light. The results revealed that the Zn:Cu ratio influenced crystallite size, morphology, and solar cell efficiency. XRD characterization confirmed the formation of ZnO and Cu phases with crystallite sizes ranging from 9 to 40 nm, while SEM analysis showed nanometer-sized particles with a tendency to agglomerate. The highest efficiency was achieved at a 3:1 ratio, reaching 120×10^{-4} %, indicating that Zn/Cu–*tapak dara* nanoparticles hold strong potential as effective photosensitizers.

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Introduction

The global energy crisis and climate change issues are rapidly growing problems today. Excessive use of fossil fuels increases air pollution due to CO₂, NO_x, and SO_x emissions from combustion [1]. These gases not only trigger global warming through the greenhouse effect but also negatively impact human respiratory health [2]. Observations in 2021 in Indonesia showed a 75% increase in exhaust gas emissions [3]. To overcome this problem, the use of renewable energy such as solar cells is needed [4]. Solar cells are able to harvest electrical energy directly from light with a relatively constant voltage of around 0.5–0.7 V and an efficiency of up to 32% [5]. However, conventional silicon-based solar cells have limitations in the form of high production costs and decreased efficiency. Therefore, a new

generation of solar cells, namely dye-sensitized solar cells (DSSC), has been developed, which offer lower costs, simple fabrication methods, and high embeddedness in their applications [6], [7].

The main component of a DSSC is the photosensitizer, a dye that functions to absorb light energy and transfer electrons to the semiconductor material. Although synthetic dyes have been widely used, they raise concerns related to toxicity, high costs, and dependence on non-renewable chemicals. Consequently, recent research has focused on the utilization of natural dyes, which are environmentally friendly and readily available. Various natural sources such as red cabbage extract, spinach leaves, strawberries, roselle flowers, dragon fruit, as well as combinations of *Beta vulgaris*, *Curcuma longa*, and *Pandanus amaryllifolius*

have been tested as photosensitizers. However, the energy conversion efficiency of natural dyes remains low, generally below 0.1% [8], [9].

The main drawbacks of directly using natural extracts are their low stability, rapid degradation under light exposure, and limited efficiency. To overcome these challenges, strategies for photosensitizer modification have been developed, such as through complex compounds or the integration of metal nanoparticles. Complex-based modifications, such as Fe–tannic acid, have been reported to improve efficiency up to 1%, while Fe(III)–chlorophyll achieved 1.35% [10]. The use of nanoparticles has also shown promising results; for instance, Ag–polyacrylate enhanced efficiency up to 2.5%, compared to natural extracts, which typically reach only 1.5% [11]. Other studies have reported that biologically synthesized ZnO nanoparticles support electron transfer, while dual doping of Cu–Ag in ZnO can further improve efficiency and enhance optoelectronic properties [12], [7]. These findings highlight that metal nanoparticles can serve as effective mediators to boost photosensitizer performance.

One of the promising plants to be developed as a photosensitizer is *Catharanthus roseus* (commonly known as tapak dara). This ornamental plant, native to Madagascar, is widely found in tropical regions, including Indonesia, and is known to be rich in secondary metabolites such as alkaloids, flavonoids, phenolics, tannins, and terpenoids [13]. These compounds function as protectors against photo-oxidation reactions, free radical scavengers, UV radiation blockers, and are capable of absorbing UV-A and UV-C [14], [15], [16]. Moreover, the visible light absorption band of *C. roseus* extract is relatively broad, making it a potential candidate for application as a photosensitizer [12].

Furthermore, *Catharanthus roseus* has also been successfully modified into nanoparticles through green synthesis methods, such as Pd-NPs with a size of 38 nm and Zn-doped NPs ranging from 10–20 nm [17]. These synthesized nanoparticles exhibited bandgap narrowing, which potentially facilitates charge transfer in DSSCs [18]. In addition, the integration of Cu nanoparticles through anthocyanin-based green

synthesis has also been reported to enhance photosensitizer performance [19]. Therefore, the integration of Zn and Cu nanoparticles with *C. roseus* pigments offers an innovative opportunity to develop hybrid photosensitizers that are more stable, environmentally friendly, and capable of achieving higher energy conversion efficiency [20]. Based on the above description, this research focuses on the utilization of Zn–*Catharanthus roseus* (tapak dara) and Cu–*Catharanthus roseus* nanoparticles as photosensitizers in DSSCs. The objectives of the study are to isolate natural pigments for synthesis into Zn–tapak dara and Cu–tapak dara nanoparticles, to evaluate their performance in DSSCs, and to compare the resulting energy conversion efficiencies. The proposed hypothesis is that the integration of Zn and Cu nanoparticles with *C. roseus* pigments can enhance light absorption efficiency, stability, and electron transfer, with the expectation that Cu–tapak dara nanoparticles will demonstrate superior performance compared to Zn–tapak dara nanoparticles.

Materials and Methods

The materials used in this study were *Catharanthus roseus* leaves, 96% ethanol, ZnSO₄, CuSO₄, distilled water, H₂PtCl₆·6H₂O, Pt metal, TiO₂, HCl, NaOH, conductive FTO glass (6 Ω/cm²), and binder clips. The instruments used in this study included a rotary evaporator (Yamato RE301A-W), a water bath (Mettler WTB24), a DC power supply (Cody 3005DT), a digital multimeter (Fluke 115), a lux meter (SNDWAY SW-582), a halogen lamp (Philips), an X-ray diffractometer (Rigaku MiniFlex 600), and a scanning electron microscope (SEM, Hitachi FlexSEM 1000).

Preparation of *Catharanthus roseus* Leaf Extract

Fresh *Catharanthus roseus* leaves, totaling 5 kg, were used as the raw material. The leaves were sorted and dried in an oven at 50°C. The dried material (simplisia) was then ground into a fine powder using a blender to facilitate extraction. Ultrasonic extraction was employed, wherein 250 g of the powdered simplisia was placed in a glass beaker and 750 mL of 96%

ethanol was added. The mixture was subjected to ultrasonic extraction for 4 hours. Subsequently, the extract was concentrated using a rotary evaporator, and any remaining solvent was removed with a water bath to obtain a viscous extract.

Synthesis of Zn and Cu Catharanthus roseus Nanoparticles

Stock solutions of ZnSO_4 (0.02 mol/L) and CuSO_4 (0.02 mol/L) were prepared by dissolving 0.48 g of each salt in 150 mL of distilled water. The Zn-to-Cu ratios for the nanoparticle synthesis were prepared according to **Table 1**.

Table 1. Zn:Cu ratios used in the synthesis of Catharanthus roseus nanoparticles

No	ZnSO ₄ : CuSO ₄	Volume ZnSO ₄ (ml)	Volume CuSO ₄ (ml)
1	0:4	0	50
2	1:3	12,5	37,5
3	2:2	25	25
4	3:1	37,5	12,5
5	4:0	50	0

A total of 20 mL of Catharanthus roseus leaf extract at a concentration of 10% was added to the solutions prepared according to Table 1. The extract solution was prepared by dissolving 4 g of leaf extract in 20 mL of distilled water. The mixture was added dropwise while being stirred at a constant speed. The resulting solution was further stirred using a magnetic stirrer for 1.5 hours at room temperature. Subsequently, 0.2 mol/L sodium hydroxide (NaOH) solution was added slowly to adjust the pH to 8. The mixture was then continuously stirred with a magnetic stirrer for an additional 3.5 hours to ensure the reaction proceeded completely. After completion, the mixture was centrifuged at 4000 rpm for 5 minutes to separate the precipitate from the supernatant. The supernatant was discarded, and the precipitate was washed twice with distilled water. Finally, the precipitate was dried at 70°C until completely dry.

Preparation of TiO₂-Coated Working Electrode

The TiO₂ coating on the working electrode was applied onto fluorine-doped tin oxide (FTO) glass substrates, which were pre-cleaned with ethanol and an ultrasonic cleaner. TiO₂ paste was made by dissolving 0.5 grams of TiO₂ powder in 4 mL of ethanol. The paste was stirred with a magnetic stirrer for 30 minutes at a rotation speed of 300 rpm. Next, the ready TiO₂ paste was coated on the FTO surface by dripping the TiO₂ paste until the FTO surface was covered with the TiO₂ paste and rotating at a speed of 1000 rpm for 60 seconds for up to 3 coatings. The TiO₂ coated FTO was left to dry in the open air. Then the electrode that had been made was ready for dehydrolysis at a temperature of 400°C for 10 minutes to evaporate the organic material.

Preparation of Pt Counter Electrode

The electrolyte solution was prepared by weighing 0.065 g of $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$, which was dissolved in a small volume of deionized water. Subsequently, 10 mL of 1 M HCl was added, and deionized water was further added to bring the total volume to 100 mL. The solution was stirred until a homogeneous mixture was obtained.

Platinum (Pt) deposition on the conductive FTO (fluorine-doped tin oxide) glass substrate was performed as illustrated in **Figure 1**. The working electrode (FTO substrate) was connected to the negative terminal of the power supply, while the Pt metal served as the counter electrode. The distance between the electrodes was maintained at 2 cm. Pt deposition on the FTO substrate was conducted at 40°C under a constant current of 15 mA for 30 minutes, with a maximum applied voltage of 0.755 V [21].

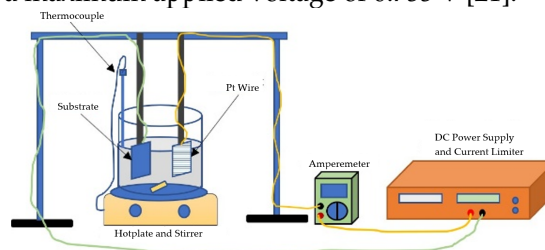


Figure 1. Schematic for depositing platinum (Pt) on a conductive FTO glass substrate, with Pt wire as the counter electrode

DSSC Solar Cell Testing

The DSSC devices were assembled as

shown in **Figure 2**. Each working electrode was loaded with 0.0015 g of Zn- and Cu-based *Catharanthus roseus* nanoparticles. The working electrode was then covered with the counter electrode and secured on both sides using binder clips. The TiO₂ semiconductor layer on the working electrode was carefully infiltrated with electrolyte to fill the inter-electrode gap.

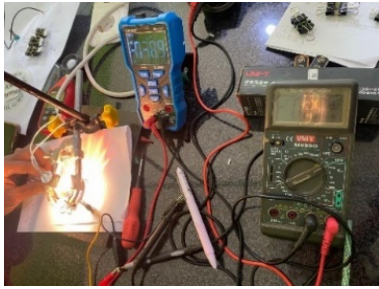


Figure 2. Assembly and testing setup of the DSSC (Dye-Sensitized Solar Cell) device.

DSSC Solar Cell Efficiency Measurement

The efficiency of the DSSC devices was measured using a series of static resistances to obtain the corresponding current-voltage (I - V) data for each variation. The applied resistances were 0 Ω (I_{sc}), 2.2, 3.3, 4.7, 10, 22, 47, 100, 220, 470, 800 Ω , and ∞ (V_{oc}). Measurements were conducted under halogen lamp illumination at an intensity of 9 mW, which was verified using a lux meter positioned directly at the location of the solar cell. The resulting current and voltage data were then used to calculate the fill factor (FF) according to Equation 1:

$$FF = \frac{V_{mpp}}{V_{oc}} \times \frac{I_{mpp}}{I_{sc}} \quad (1)$$

Meanwhile, the efficiency of the DSSC was calculated using Equation 2:

$$\eta = \frac{V_{oc} \times I_{sc} \times FF}{P_{chy}} \quad (2)$$

Keterangan

- V_{mpp} : maximum voltage produced by cell
 I_{mpp} : maximum current produced by cell
 V_{oc} : cell potential in an open circuit
 I_{sc} : cell current in a closed circuit
 P_{chy} : light power is exposed to cell

Results and Discussion

Preparation of *Catharanthus roseus* Leaf

Extract

The leaf extract of *Catharanthus roseus* was prepared using the ultrasonic extraction method, aiming to obtain higher quality extract. Ethanol 96% was used as the solvent, yielding a viscous extract of 55.6 g with an extraction yield of 22.42%. Ultrasonic extraction is considered an effective technique as it utilizes high-frequency sound waves (typically >20 kHz) to produce a mechanical effect known as cavitation, which involves the formation and collapse of microbubbles in the liquid. The advantages of this method include a relatively fast process, high efficiency, no requirement for additional chemicals, and the ability to produce a more stable dispersion [22].

Synthesis of Zn and Cu Nanoparticles from *Catharanthus roseus*

The synthesis of Zn and Cu nanoparticles was carried out with Zn:Cu ratios of 0:4, 1:3, 2:2, 3:1, and 4:0, as shown in **Table 1**. The *Catharanthus roseus* leaf extract was added dropwise to each solution variation and stirred with a magnetic stirrer for 1.5 hours. Subsequently, NaOH was gradually added while monitoring the pH with a pH meter until reaching pH 8. This procedure aims to control the particle size, shape, and stability of the nanoparticles. The pH condition influences the nucleation rate and crystal growth; with optimal pH, the resulting particles tend to be smaller, more uniform, and more stable in suspension [23]. The mixture was then further stirred for 3.5 hours and centrifuged. The precipitate was dried in an oven at 70 °C. The synthesis results, shown in **Figure 3**, reveal fine powder characteristics, with a darker coloration observed in samples containing higher Zn content.

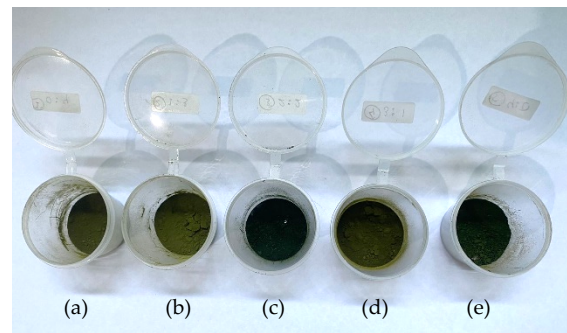


Figure 3. Synthesis results of *Catharanthus roseus* Cu and Zn nanoparticle complexes:

(a) 0:4, (b) 1:3, (c) 2:2, (d) 3:1, (e) 4:0.

Preparation of the Working Electrode and the Counter Electrode

The working electrode was prepared by coating an FTO glass substrate with a TiO_2 layer. TiO_2 serves as the semiconductor in DSSCs, receiving electrons injected from the photosensitizer. Additionally, TiO_2 acts as a support for the dye (photosensitizer), which absorbs light, generates electrons, and transfers them to the conductive FTO glass, thereby producing electrical energy [24]. The resulting TiO_2 coating, as shown in **Figure 4.a**, exhibits a clean white color and adheres well to the active surface of the conductive glass.

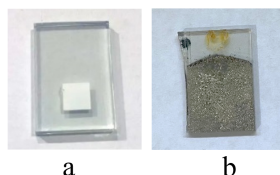


Figure 4. (a) Coating of the FTO working electrode with TiO_2 , (b) Coating of the FTO counter electrode with Pt metal.

The counter electrode was prepared by coating Pt onto FTO glass, indicated by the characteristic metallic Pt color on the conductive glass surface, as shown in **Figure 4b**. This coating process involves a redox reaction, where Pt ions from the H_2PtCl_6 electrolyte solution are reduced and subsequently deposited onto the conductive FTO surface through electroplating. The electrolyte solution serves both as a medium for electrical conduction and as a source of Pt ions that are deposited onto the electrode surface (cathode) [25].

Testing of Dye-Sensitized Solar Cells

The dye-sensitized solar cells were tested by placing Zn and Cu tapak dara nanoparticle powders directly onto the TiO_2 layer of the working electrode. The two electrodes were then assembled face-to-face, leaving a small portion of the FTO glass exposed for connection to the instruments measuring the cell's potential and current. A few drops of electrolyte were added into the gap between the glass substrates, allowing the Zn-tapak dara or Cu-tapak dara

dye to be spontaneously wetted by the electrolyte solvent, as shown in **Figure 5**.

The working principle of a dye-sensitized solar cell (DSSC) is based on the process of photosensitization, in which dye molecules adsorbed onto the porous surface of TiO_2 semiconductor absorb sunlight. The absorbed light excites electrons within the Zn-tapak dara or Cu-tapak dara molecules, which are then injected into the conduction band of TiO_2 . Subsequently, the electrons move through the conductive substrate to the external circuit, generating an electric current. The oxidized dye molecules are regenerated by the electrolyte, while the oxidized electrolyte is reduced at the counter electrode. Thus, this reaction cycle repeats continuously, allowing the DSSC to produce electrical energy sustainably from sunlight [26]. After assembly, the DSSC was illuminated using a halogen lamp with a light intensity of 9 mW over an effective area of 0.25 cm^2 .

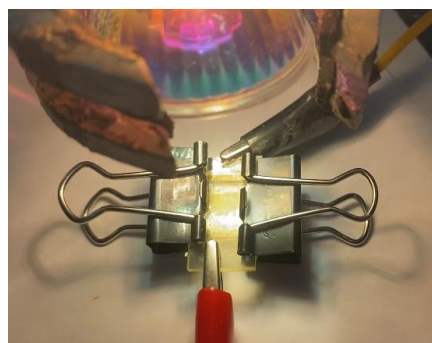


Figure 5. Measurement of the DSSC under halogen lamp illumination.

DSSC Efficiency Measurement

The efficiency measurement results for each variation of the DSSC are presented in the bar chart shown in **Figure 6**. The graph indicates that Zn and Cu tapak dara nanoparticles in the DSSC are capable of generating electric current due to the continuous electron transfer occurring within the system. This process takes place when light irradiates the surface of the semiconductor coated with Zn and Cu tapak dara nanoparticles, causing their molecules to be excited from the ground state to higher energy levels. The excited electrons possess sufficient energy to be injected into the conduction band of the semiconductor. This electron injection mechanism is facilitated by

the interaction between the Zn and Cu tapak dara nanoparticle molecules and TiO_2 through the bonds formed [27].

The graph in **Figure 6** demonstrates that the Zn:Cu ratio significantly affects the efficiency (η) of the dye-sensitized solar cell. At a 0:4 ratio, where Cu-tapak dara is used as the sensitizer, the resulting efficiency is very low. Slight increases in efficiency are observed at the 1:3 and 2:2 ratios, although the values remain relatively low. The peak efficiency is achieved at a 3:1 ratio, where Zn is dominant, reaching the highest value of approximately $120 \times 10^{-4} \%$. This indicates that this composition is the most optimal for achieving superior solar cell performance. However, at a 4:0 ratio (Zn only), efficiency decreases again, although it is still higher compared to the lower Zn ratios. Therefore, it can be concluded that the Zn:Cu ratio of 3:1 provides the best performance, whereas the use of Cu or Zn alone is less effective in enhancing solar cell efficiency. Overall solar cell performance was not satisfactory, as the efficiency was still below 1%. This low efficiency was influenced by several factors, including uneven TiO_2 coating, which resulted in poor dye adhesion, and too little Zn and Cu nanoparticle powder in the efficiency test.

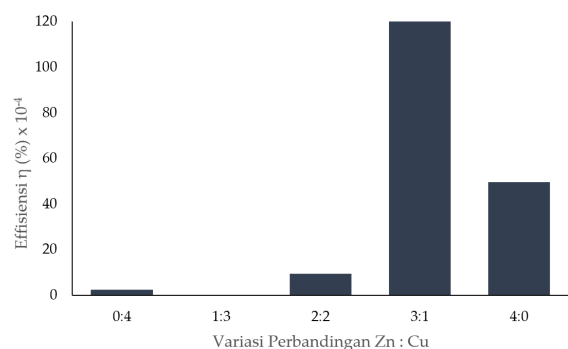


Figure 6. Bar graph of DSSC efficiency measurements for various Zn:Cu ratios.

The 3:1 ratio in the synthesis of Zn:Cu-Catharanthus roseus material appears to be optimal due to the synergy between the two metals. Zn contributes by maintaining stability and enhancing electron transfer efficiency to the TiO_2 conduction band, while Cu assists in improving light absorption. The dominance of Zn with a small addition of Cu likely creates a more balanced system, ensuring effective light

harvesting and efficient electron transfer.

XRD Characterization Results

The crystal structure of Zn and Cu nanoparticles synthesized using Catharanthus roseus leaf extract was characterized by X-Ray Diffraction (XRD). The diffractograms in **Figure 7** show the diffraction patterns for five samples with Zn:Cu ratios of 0:4, 1:3, 2:2, 3:1, and 4:0. Phase identification was performed by comparing the diffraction peak positions with reference data from the Joint Committee on Powder Diffraction Standards (JCPDS), specifically JCPDS No. 01-070-1255 for Zn and JCPDS No. 01-077-1900 for Cu. In the sample with a Zn:Cu ratio of 4:0, a diffraction peak at approximately $2\theta = 21.2^\circ$ with low intensity was observed, indicating the presence of a cubic crystal structure.

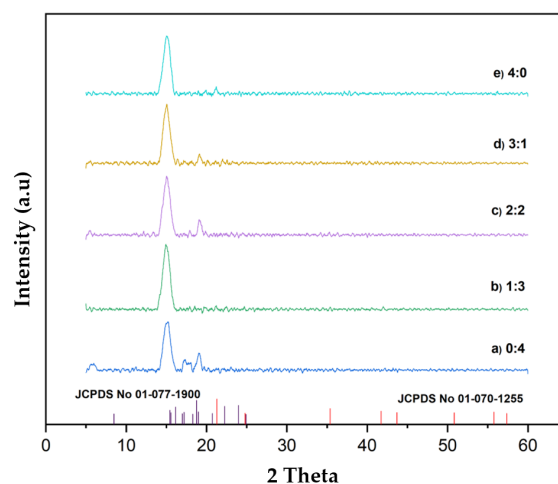


Figure 7. XRD Diffractogram Patterns of Zn and Cu – Catharanthus roseus

In the sample with a Zn:Cu ratio of 0:4, the main diffraction peaks were observed at $2\theta \approx 15.2^\circ$ and 19.1° with moderate intensity, indicating an anorthic crystal structure. For mixed-ratio samples such as 3:1, 2:2, and 1:3, diffraction peaks from both phases (ZnO and Cu) appeared, although with lower intensity compared to the pure samples. This suggests a reduction in crystallinity and the possible formation of mixed phases or structural distortions caused by the interaction of Zn^{2+} and Cu^{2+} ions within the crystal lattice, which is consistent with reports by Ali et al., 2022 [28]. Peak shifts and broadening in the diffractograms indicate that the resulting

particles are smaller, approaching the nanoscale. Additionally, all sample variations exhibited very high-intensity peaks around 15° , corresponding to the crystalline features of *Catharanthus roseus* leaves containing Zn and Cu atoms. The sharp and intense peaks reflect a high degree of crystallinity in the synthesized materials.

Table 2. Crystallite Size Calculation Results Using the Scherrer Equation

Zn:Cu Tapak Dara Variations	2θ ($^\circ$)	β ($^\circ$)	D (Scherrer, aprox.) (nm)
0:4	15,2	0,25	32 nm
1:3	16,0	0,6	13 nm
2:2	16,5	0,9	9 nm
3:1	18,0	0,4	20 nm
4:0	21,2	0,2	40 nm

The calculation of crystallite size presented in **Table 2** was obtained by inserting the diffraction values into the Scherrer equation for various Zn:Cu ratios. The results indicate significant differences in crystallinity size. For the sample synthesized with a 0:4 ratio (Cu only), the crystallite size was approximately 32 nm. The crystallite size decreased sharply for the mixed ratios of 1:3 and 2:2, reaching 13 nm and 9 nm, respectively, indicating that the addition of Zn into the system caused particle size contraction. For the 3:1 ratio, the crystallite size increased again to 20 nm, whereas for the 4:0 ratio (Zn only), the crystallite size reached the highest value of approximately 40 nm.

These variations suggest that the formation of Zn–Cu mixed phases can induce lattice distortion, contributing to particle size reduction to the nanometer scale. In contrast, single-element samples (only Zn or only Cu) exhibit more stable crystal structures, resulting in relatively larger crystallite sizes. Such distortions are believed to benefit the solar cell by enhancing the conversion of light energy into electrical potential for injection into TiO_2 .

SEM Characterization Results

Morphological characterization using Scanning Electron Microscopy (SEM) revealed that the Zn–*Catharanthus roseus* and Cu–

Catharanthus roseus nanoparticles with a 3:1 ratio—showing the highest efficiency—exhibit relatively irregular particle shapes with a tendency toward sphericity and amorphous aggregation (**Figure 8**). At $1000\times$ magnification, the particles appear to cluster into larger agglomerates, indicating van der Waals interactions between particles. At higher magnification, the nanoparticle surfaces become more distinct, with particle sizes increasingly well-defined, although some overlap remains. At $5000\times$ magnification, the particle morphology shows a more detailed size distribution, with particle sizes ranging from the nanometer to submicrometer scale.

The observed aggregation may result from the synthesis process based on *Catharanthus roseus* extract, which is rich in secondary metabolites such as alkaloids, flavonoids, and terpenoids that act as both reducing and stabilizing agents. However, these compounds are not entirely able to prevent particle clustering [29]. Overall, the SEM results indicate that Zn–*Catharanthus roseus* and Cu–*Catharanthus roseus* nanoparticles were successfully formed, exhibiting characteristic nanomaterial morphologies that have the potential to enhance their performance as photosensitizers in DSSC solar cells.

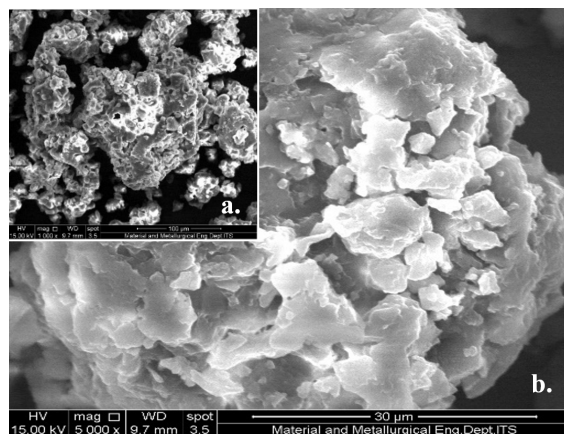


Figure 8. SEM images of the 3:1 Zn:Cu variation, a. micrograph at $1000\times$ magnification and b. micrograph at $5000\times$ magnification.

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Conclusion

Based on the results of this study, it can be concluded that Zn:Cu–*Catharanthus roseus* leaf extract is capable of producing natural pigments with potential as environmentally friendly photosensitizers. The variation in the Zn:Cu ratio affects the crystallite size and the performance of DSSCs, with the 3:1 ratio yielding the best results and the highest efficiency of $120 \times 10^{-4}\%$. The synthesized particles exhibit crystallinity at the nanoscale, as confirmed by XRD analysis.

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