



Enhancing DSSC Performance through Metal-Doped TiO₂ and Poly-Tannin Dye: A Study on Bandgap Reduction and Photon Absorption

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Abstract: Dye-sensitized solar cells (DSSC) are a promising alternative to conventional solar cells by using dye molecules to absorb sunlight and facilitate energy conversion. In this study, tannins were used as the dye. Tannins can be polymerized into poly-tannins, which increases the amount of double bonds and therefore improves photon absorption. Furthermore, this study will also examine how metal doping affects the TiO₂ semiconductor to further enhance its performance. The selected dopants—Cu, Fe, and Ag—were tested for their capacity to change the bandgap and increase electron mobility. Experiments showed that metal doping lowered the bandgap of TiO₂, resulting in better electron excitation and charge transfer. Under optimum conditions, the combination of poly-tannin dye and TiO₂-Ag semiconductor resulted in the highest DSSC efficiency (9.18%). Based on this, it can be concluded that combining metal-doped TiO₂ with poly-tannin dyes can greatly improve DSSC performance.

Keywords: DSSC, Photovoltaic Efficiency, Poly-Tannin, Semiconductor Bandgap, TiO₂ Modification.

1. Introduction

The global energy problem is becoming more obvious as fossil fuel supplies decrease, and electricity demand rises [1], [2], [3], [4], [5]. As a result, reliable and sustainable alternative energy sources are required [6], [7], [8], [9], [10], [11]. One possibility is the use of solar energy via solar cells [12], [13], [14], [15], [16], [17], [18]. Solar energy is the most abundant form of energy that is limitless, pollution-free, and continuously available [19], [20], [21], [22], [23].

Solar cells are categorised into three main generations. The first generation consists of single and polycrystalline silicon-based solar cells [24], [25], [26], [27]. The second generation includes thin-film solar cells, while the third generation includes dye-sensitised solar cells (DSSC) [28], [29], [30], [31]. DSSCs offer a solar cell solution that is environmentally friendly, non-toxic, and has a lower production cost compared to silicon-based solar cells [32], [33], [34]. However, the efficiency of DSSCs still needs to be improved to be more competitive with other solar cell technologies [35], [36].

DSSCs have a layered structure consisting of TiO₂ nanocrystal-based semiconductors, dyes, working electrodes, carbon or platinum-based opponent electrodes, and electrolyte solutions that maintain the continuity of redox reactions in the cell [37], [38], [39], [40], [41]. TiO₂ is one of the most used inorganic semiconductors in DSSCs due to its photocatalytic, stable, abundant and low-cost properties [42], [43], [44], [45], [46]. However, with a bandgap of just 3.2 eV, TiO₂ only absorbs about 5% of the solar spectrum, limiting it to the ultraviolet (UV) range [44], [47], [48], [49]. As a result, strategies are needed to improve its absorption efficiency in the visible light spectrum.

Several approaches have been proposed for overcoming this, including changing semiconductor composition and doping with metal atoms. Doping has been proven to lower TiO₂'s bandgap, allowing it to absorb light at longer wavelengths [50], [51], [52]. Furthermore, doping can reduce electron-hole recombination, increasing the device's efficiency [53], [54], [55], [56]. Among them, doping with metals including Cu, Fe, and Ag improves TiO₂'s photocatalytic activity [57], [58], [59], [60], [61]. Cu enhances photocatalytic activity by promoting electron transport. Fe has a lower bandgap than TiO₂, allowing for increased absorption of visible light [62], [63], [64], [65], [66]. Meanwhile, Ag is recognized as a transition dopant that can lower particle size and increase the surface area of TiO₂. It also contributes to the plasmonic effect, which can increase light absorption [67], [68], [69], [70], [71].

In addition to semiconductor optimisation, the use of dyes in DSSCs also plays an important role in improving energy conversion efficiency. Organic dyes are widely used in DSSCs because they are low cost, easy to synthesise, and environmentally friendly [35], [38], [72], [73], [74]. However, organic dyes generally have a smaller light absorption area than synthetic dyes [75], [76]. In this study, tannins were chosen as colourants because they are natural macromolecules containing polyhydroxyphenyl groups, which have the potential to increase light absorption.

To improve their absorption efficiency, tannins can be polymerised using cross-linking agents such as glutaraldehyde, which contains two aldehyde groups and is highly effective in forming poly-tannins. Polymerisation of tannins aims to increase the number of double bonds in their molecules, which is expected to maximise photon uptake and improve the efficiency of DSSCs. Therefore, this research focuses on the use of poly-tanine dyes in DSSCs as well as the effect of semiconductor doping on device efficiency, to obtain more efficient and sustainable solar cells.

2. Research Methodology

This research used various laboratory tools such as beakers, measuring cups, watch glass, round bottom flask, stirring rod, volume pipette, dark bottle, spatula, ultrasonic cleaner, volumetric flask, oven, hot plate, analytical balance, magnetic stirrer, vacuum, pycnometer, Oswald viscometer, funnel, and UV lamp. Analytical instruments used include FTIR spectrophotometer, UV-DRS, and digital multimeter. Materials used include tannin, glutaraldehyde (GA, Merck, ≥25% in water), filter paper (Whatman No. 1), ITO glass (Sigma-Aldrich, surface resistance ~15 Ω/sq), distilled water, aquabidest (Ikapharmindo, p.a.), formaldehyde 37% (Merck, p.a.), KOH (Merck, ≥85%, pellets), HCl 36% (Merck, p.a.), indicator paper (Merck universal pH indicator), alcohol 70% (Indo Alcohol), aluminum foil (Reynolds), KI/I₂ (potassium iodide and iodine, Merck, p.a.), polyethylene glycol (PEG 400, Merck), acetonitrile p.a. (Merck, ≥99.8%), methanol p.a. (Merck, ≥99.9%), TiO₂ Degussa P-25 (Evonik, anatase/rutile ~70:30), FeSO₄·7H₂O (Merck, p.a.), and carbon from wax (locally synthesized from paraffin candle wax via pyrolysis).

Tannin polymerisation was carried out by mixing 36% HCl and 37% formaldehyde solution with tannin, then stirring at 200°C for 2 hours using a magnetic stirrer. The tannin-formaldehyde resin product was filtered, washed with water, and dried in an oven at 80°C. The resin was then dissolved in 2% NaOH solution as initiator and heated at 60-70°C with continuous stirring before adding glutaraldehyde as crosslinker. The polytanin glutaraldehyde resin (PTGR) was then heated at 120°C and cooled to room temperature.

Analysis was carried out before and after polymerisation to see the structural changes between tannins and poly-tannins. For semiconductor preparation, TiO₂ and FeSO₄·7H₂O were dissolved in methanol and stirred until homogeneous, then sonicated and heated in an oven at 96°C. For Cu-doped TiO₂, TiO₂ was mixed with CuO in methanol, stirred, sonicated, and heated before calcination at 400°C. For Ag-doped TiO₂, PVA was dissolved in distilled water and heated at 80°C while stirring until a suspension was formed, then TiO₂ and AgNO₃ were added and stirred until homogeneous.

In preparation of the DSSC, the ITO glass was cut and cleaned using 70% alcohol in an ultrasonic cleaner. After drying, the resistance of the glass was measured with a multimeter. A layer of scotch tape was attached to the glass, then TiO₂-doped paste was applied using the doctor blade method and heated at 100°C. The electrolyte solution was made by mixing KI and acetonitrile, then adding I₂ and PEG to form a gel. The counter electrode was made by coating ITO glass with carbon obtained from burning candles.

The dried TiO₂-doped layer was soaked in poly-tanine dye to allow complete absorption, then dried and arranged with the carbon-based counter electrode in a sandwich structure. The two electrodes were clamped together, and electrolyte solution was dripped between the electrode gaps. The assembled DSSC was tested using a UV lamp with an intensity of 24 W/m², and its efficiency was calculated based on the voltage and current measured using a digital multimeter

3. Results and Discussion

3.1. Dye Preparation

In this study, tannins were used as colourants. Tannins are polyphenolic compounds that have a hydroxyl group (-OH) attached to a benzene ring. These compounds have conjugated double bonds that allow them to absorb photons from the ultraviolet (UV) to visible light spectrum. In this study, tannins were polymerised to increase the number of double bonds.

Firstly, tannins were refluxed with formaldehyde and hydrochloric acid (HCl) at 200°C for 2 hours. A 37% formaldehyde solution is used to dissolve the tannins and facilitate the condensation reaction, as the phenol groups in tannins are highly reactive. Hydrochloric acid acts as an acid catalyst as well as a solvent in this reaction. Formaldehyde acts as a link between tannin monomers by forming methylene bridges (-CH₂), so that the reaction continues with other monomers to form polymers. The reflux process produces a purple-coloured tannin-formaldehyde product.

After refluxing, tannin-formaldehyde was dissolved in NaOH solution and stirred at 70°C for 1 hour. NaOH acts as an initiator that generates free radicals for the polymerisation process and helps to break the double bonds in the crosslinking agent (glutaraldehyde), which will connect the tannin monomers during the polymerisation process. After the addition of NaOH, the initially purple coloured product turned black due to the strong alkaline nature of NaOH. This initiator also affects the chain length of the polymer and increases its molecular weight.

Next, the tannin-formaldehyde mixture was mixed with glutaraldehyde and stirred at 120°C for 90 minutes, resulting in a brown-black poly-tannin precipitate. Glutaraldehyde was chosen as the crosslinking agent because it has two epoxy aldehyde groups, which open during the reaction with NaOH and bind to the tannin-formaldehyde molecules. Increasing the temperature during the addition of glutaraldehyde aims to accelerate the polymerisation reaction rate. The addition of this crosslinking agent increases the number of tannin monomers and double bonds, thereby improving the light absorption ability. The resulting poly-tanine glutaraldehyde resin (PTGR) is then used as a colourant in DSSCs.

3.2. DSSC preparation

The prepared DSSC consists of several components, namely ITO glass substrate, dye, semiconductor, counter electrode, and electrolyte solution. These components are assembled in a sandwich structure. The electrolyte I⁻/I₃⁻ was applied to both ends of the electrodes, and the voltage generated by the DSSC was measured using a digital multimeter.

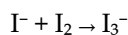
Firstly, the ITO glass used as the substrate was cleaned with 70% alcohol to remove impurities and improve its cleanliness. A sonication process was performed for a more optimised cleaning, then the glass was dried and the resistance was measured, with a resistance of 100Ω.

The semiconductor used as a photocatalyst is titanium dioxide (TiO₂ Degussa P-25), which has two phases, 80% anatase and 20% rutile, with an energy gap (bandgap) of 3.27 eV. Doping is done to reduce the bandgap of TiO₂ to increase the efficiency of DSSC, because the smaller the bandgap of a semiconductor, the easier it is for electrons to be excited and move. The metals used in doping are Fe, Ag, and Cu. FeSO₄·7H₂O is used as a precursor for Fe doping, while AgNO₃ and CuO are used for Ag and Cu doping. The sol-gel method was used in the synthesis of TiO₂-doping, in which a phase change occurs from particles dispersed in a liquid (sol) to a macroscopic phase containing liquid (gel). Methanol, as a polar solvent, was used to aid homogenisation and control of metal concentration in the semiconductor synthesis.

In DSSC preparation, TiO₂-doped semiconductor paste is applied to ITO glass using the doctor blade method and heated at 100°C for 30 minutes to ensure the coating adheres well. After drying, the coating was immersed in poly-tanine dye so that it could be fully absorbed. The layer that has absorbed the dye is dried again and used as a working electrode in the DSSC.

The carbon counter electrode was made by coating the ITO glass with soot from burning candles. This process is done by passing the glass on the candle flame so that an even layer of black carbon is formed. This carbon layer serves as a catalyst in the electron transfer process.

The electrolytes used are potassium iodide (KI) and iodine (I₂), which act as electron donors for oxidised dye molecules. The electrochemical reaction that occurs is:



I⁻ ions function as electron donors to return the dye to its initial state and prevent electron recapture. The electrolyte is formulated in semi-solid form with the addition of polyethylene glycol (PEG), which serves to prevent evaporation of the electrolyte solution so that it can be used for a longer period of time. The resulting electrolyte solution

was light yellow in colour and dripped into the gap between the working electrode and the counter electrode. Both electrodes were clamped with clips to ensure the DSSC structure remained stable and did not shift.

DSSC testing was conducted in a box with a 24-watt UV light source. The efficiency of the DSSC was calculated based on the voltage and current measured using a digital multimeter. The working principle of DSSC is that when the dye is exposed to UV light, the electrons in the dye molecule are excited from the HOMO to LUMO energy level. These excited electrons then move to the TiO₂ conduction band, causing oxidation of the dye and producing free electrons. These electrons flow through the ITO glass conductive substrate towards the opposing electrode, generating an electric current in the external circuit. The electrons returning to the cell react with the electrolyte, allowing dye regeneration so that the cycle can repeat.

3.3. Doping Characterization

Characterisation of Ag-doped TiO₂ was performed using UV-Vis DRS spectroscopy to determine the bandgap value of the resulting semiconductor. The addition of Ag metal in the synthesis is expected to reduce the bandgap value of TiO₂, thereby increasing light absorption in the longer wavelength region. Bandgap is the energy difference between the valence band and the conduction band in a semiconductor material.

The bandgap value is determined using the Tauc Plot method, which estimates the energy gap by plotting the relationship between the absorbed photon energy ($h\nu$) on the x-axis and the product of the absorption coefficient with photon energy $(\alpha h\nu)^n$ on the y-axis. The equation used is:

$$(\alpha h\nu)^n = K(h\nu - E_g) \quad (1)$$

where E_g is the bandgap energy, K is the proportionality constant, and n depends on the type of electronic transition (for anatase TiO₂, $n = 2$ because the transition is a direct allowed transition).

The absorbance spectrum of TiO₂/Ag obtained through UV-Vis DRS shows a wavelength range between 186-1100 nm. At a wavelength of 304 nm, there is a significant increase in absorbance, which indicates that the $(\alpha h\nu)^2$ value also increases drastically by 1262.46 (eV cm⁻¹)². This is related to the decrease in transmittance value, where the lower the transmittance, the higher the $(\alpha h\nu)^2$ value.

Based on the analysis, the bandgap value of pure TiO₂ (anatase phase) is 3.2 eV. After doping with Ag, the bandgap decreased to 2.922 eV, which indicates that Ag doping successfully reduced the semiconductor energy gap. TiO₂/Ag Absorbance and Bandgap of TiO₂/Ag graph using UV-Vis DRS can be seen in Figure 1. This decrease occurs because Ag⁺ ions play a role in the charge transfer process from dopant cations to Ti⁴⁺ ions, which changes the electronic structure and forms lower energy levels.

In addition, the decrease in bandgap can also be attributed to the plasmonic effect of silver (Ag) nanoparticles. This effect increases photon absorption in the visible light range, which is very useful for DSSC applications as it broadens the absorption spectrum of sunlight. However, since the radius of Ag⁺ ions is larger (1.15 Å) than that of Ti⁴⁺ (0.60 Å), only a small part of Ag⁺ ions can replace the position of Ti⁴⁺ in the TiO₂ lattice, so the doping effect may be limited.

The decrease in bandgap obtained in this study indicates a potential increase in DSSC efficiency because it allows electron excitation to occur at lower energies. With a narrower bandgap, TiO₂/Ag can absorb more light in the visible spectrum, thus increasing the number of electrons that can be excited and reducing the recombination rate, which ultimately results in an increase in the energy conversion efficiency of the solar cell.

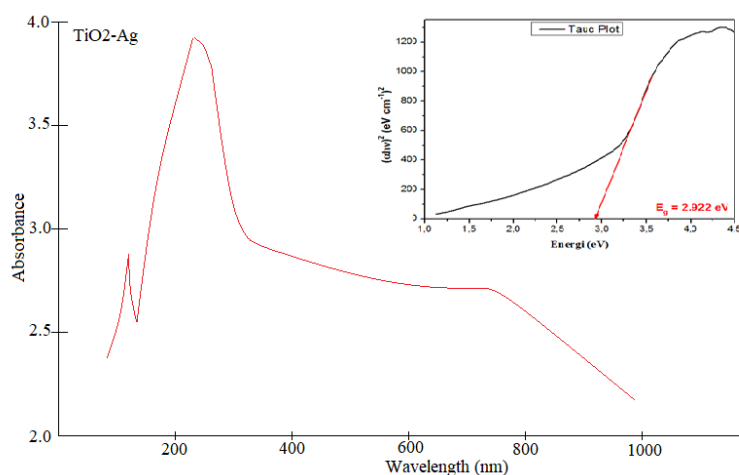


Figure 1. TiO₂/Ag Absorbance and Bandgap of TiO₂/Ag Graph using UV-Vis DRS

Characterisation of CuO-doped TiO₂ was carried out to analyse the change in bandgap energy resulting from the addition of CuO nanoparticles into the TiO₂ matrix. TiO₂ in the anatase phase has a bandgap of 3.2 eV, which causes this material to absorb only about 5% of sunlight, especially in the ultraviolet (UV) spectrum range with wavelengths below 400 nm. Therefore, to increase the photocatalytic activity of TiO₂, doping with CuO was carried out to expand the light absorption spectrum.

TiO₂/Cu Absorbance Graph using UV-Vis DRS and Bandgap TiO₂/Cu Graph using UV-Vis DRS can be seen in Figure 2. The characterization results showed that after doping with CuO, the bandgap value of TiO₂ dropped to 3.01 eV. The reduction was calculated using the Tauc Plot method using the absorbance value acquired from UV-Vis DRS spectroscopy. CuO doping causes a decrease in bandgap due to the interaction of Cu²⁺ ions with the TiO₂ structure, altering the electrical arrangement in the semiconductor lattice. Cu²⁺ ions, bigger than Ti⁴⁺, can partially replace or occupy interstitial sites in the TiO₂ crystal lattice. This results in the development of new energy levels in the bandgap, which narrows the distance between the valence and conduction bands, thereby improving the material's ability to absorb visible light.

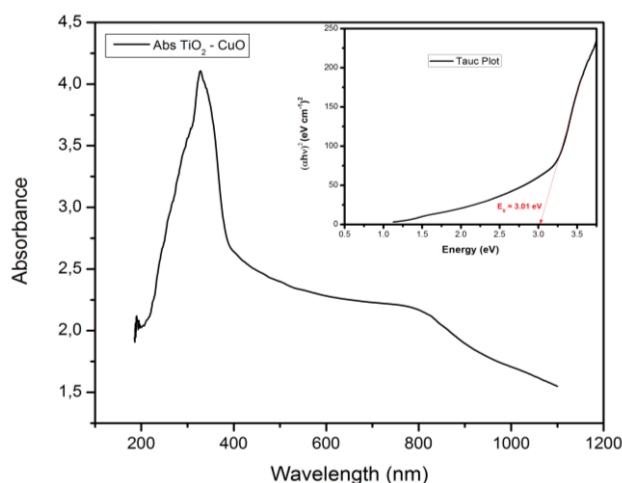


Figure 2. TiO₂/Cu Absorbance Graph using UV-Vis DRS and Bandgap TiO₂/Cu Graph using UV-Vis DRS

CuO doping lowers the bandgap and increases the electrical conductivity of TiO₂. A smaller bandgap allows for electron activation with less photon energy, allowing more electrons to move to the conduction band when exposed to light. As the number of excited electrons increases, the rate of electron-hole recombination decreases, enhancing the photocatalytic efficiency. CuO-doped TiO₂ has a lower bandgap than pure TiO₂, making it more useful for DSSC applications by absorbing more visible light. A reduced bandgap allows more electrons to be excited, improving charge separation and speeding up electron transport in solar cells. This improves DSSCs' energy conversion efficiency, making them more effective in capturing solar energy and converting it into electricity.

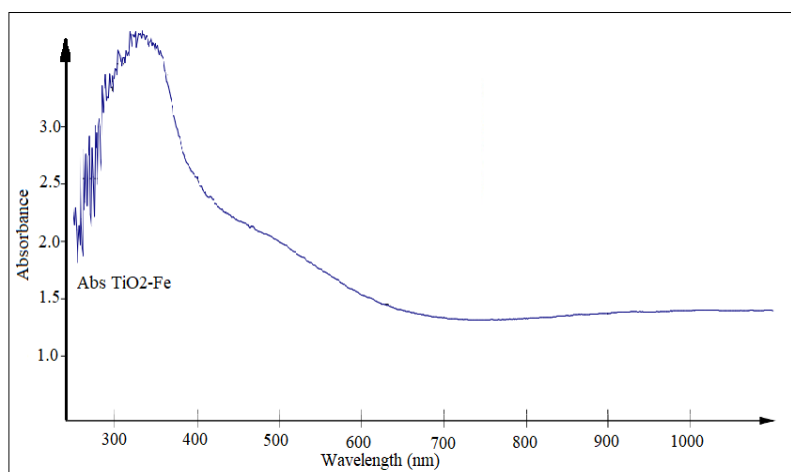


Figure 3. Graph of TiO₂-Fe bandgap values

The graph of TiO₂-Fe bandgap values can be seen in Figure 3. based on the analysis results of the characterization carried out, Fe doping on TiO₂ reduces the band gap from 3.27 eV to 3.05 eV. This decrease is due to the presence of Fe in the TiO₂ structure which forms a metal contact layer. This layer acts as a conductor that accelerates the movement of electrons towards the ITO electrode, thereby increasing the efficiency of charge transfer. Thus, this doping has the potential to increase the efficiency of DSSCs by accelerating electron transport and expanding light absorption to a wider spectrum.

3.4. DSSC Efficiency

Tests were conducted to determine the efficiency of the DSSC based on variations in polymerisation time as well as the effect of tannin monomer concentration in the dye. The efficiency of the DSSC was measured when the cell converted photons into electrical energy with the help of a 24 Watt UV lamp. The efficiency value is obtained by measuring the resistance on the conductive side of the ITO glass and the voltage generated by the cell using a digital multimeter. From the measurement of the resistance and voltage of the DSSC, the electric current produced can be calculated.

DSSC efficiency using pure TiO₂ with tannin and poly-tannin dyes can be seen in Figure 4, while DSSC efficiency using poly-tannin dyes with TiO₂ and doped TiO₂ semiconductors can be seen in Figure 5. The efficiency of the DSSC is calculated using the formula:

$$\eta = \frac{P_{out}}{P_{in}} \times 100\% \quad (2)$$

Where P_{out} is the generated electrical power (V.I), P_{in} is the power from the light source absorbed by the DSSC, and η is the energy conversion efficiency. Based on the experimental results, the use of modified dye in DSSC shows an increase in efficiency. Polymerisation of tannins produces molecules with more double bonds, which increases their ability to absorb photons from sunlight. In addition, the prepared dyes were tested on semiconductors that had been modified through doping.

The decrease in the bandgap of the semiconductor contributes to the increase in DSSC efficiency because electron excitation becomes easier with a smaller energy gap. The experimental results show that the highest efficiency of the DSSC obtained is 9.18%, which results from the DSSC with a combination of poly-tanine dye and TiO₂-Ag semiconductor. This shows that the combination of the modified dye and a semiconductor with a lower bandgap provides optimal performance in increasing the efficiency of converting light energy into electricity.

Other parameters that influence DSSC efficiency include the durability of the poly-tanine dye, the effect of Ag doping on electron conductivity, and semiconductor shape. Poly-tanine dyes degrade when exposed to UV light and interact with the electrolyte, which can diminish efficiency over time. Ag doping not only decreases the bandgap, but it also boosts electron mobility, which reduces electron-hole recombination and speeds up charge transfer. Furthermore, the more porous TiO₂-Ag structure can improve the surface area and dye adsorption, resulting in higher efficiency.

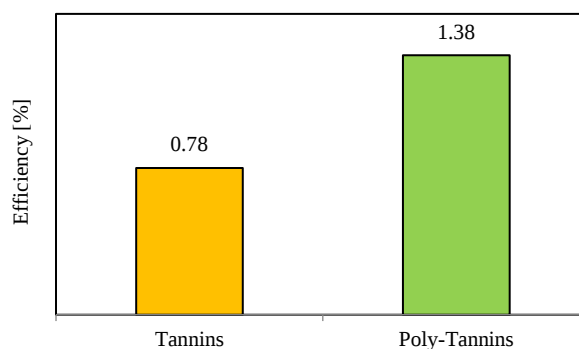


Figure 4. DSSC efficiency using pure TiO₂ with tannin and poly-tannin dyes

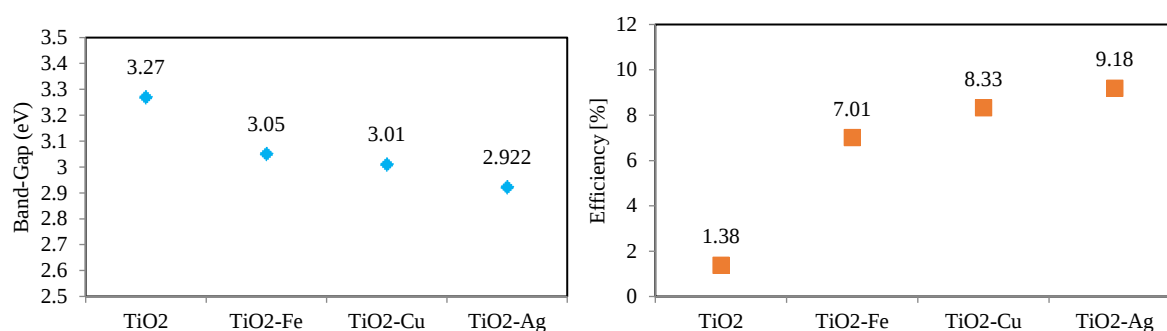


Figure 5. DSSC efficiency using poly-tannin dyes with TiO₂ and doped TiO₂ semiconductors

4. Conclusion

Due to the presence of more double bonds in the poly-tanine structure, which improves light absorption and energy conversion efficiency, Poly-tanine dyes perform better in DSSCs than pure tannins. Furthermore, it is also known that semiconductors with shorter bandgaps offer higher efficiency. This is because they facilitate electron excitation, which boosts electron mobility in the system. This work reached a maximum efficiency of 9.18% using a DSSC with Ag-doped TiO₂ semiconductor and poly-tanine-based dye. These findings imply that combining metal doping with dye modification can be an effective strategy for improving DSSC performance.

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