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Green Synthesis of Barium Doped Titanium Dioxide Using Palm Leaf Extract (*Elaeis guineensis Jacq.*)

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ABSTRACT

Conventional synthesis of titanium dioxide (TiO_2) nanoparticles often involves the use of toxic chemicals, high costs, and extreme operating conditions. This research aims to develop a cost-effective and environmentally friendly green synthesis method for TiO_2 by utilizing oil palm leaf (*Elaeis guineensis Jacq.*) extract as a reducing and stabilizing agent. Subsequently, doping with barium (Ba) was carried out to enhance the photocatalytic, optical, and electrical properties of the resulting TiO_2. Phytochemical analysis revealed that oil palm leaf extract contains alkaloids, flavonoids, terpenoids, saponins, and tannins, which play a role in the reduction and stabilization processes of TiO_2 nanoparticles. Characterization using *Fourier Transform Infrared Spectroscopy* (FTIR) identified Ti-O bonds in pure TiO_2 at a wavenumber of 588 cm^{-1}, and a shift in absorption peaks in the range of 548-595 cm^{-1} in Ba-doped TiO_2 indicated the formation of the barium titanate phase. *X-ray Diffraction* (XRD) analysis showed that Ba doping increased the crystallite size of TiO_2 from 1.17 nm to 1.65 nm, which may have implications for photocatalytic applications.

Keywords: Barium Doping, Environmentally Friendly, Green Synthesis, Titanium Dioxide.

ABSTRAK

Sintesis nanopartikel titanium dioksida (TiO_2) secara konvensional sering kali melibatkan penggunaan bahan kimia beracun, biaya tinggi, dan kondisi operasi ekstrem. Penelitian ini bertujuan untuk mengembangkan metode sintesis hijau TiO_2 yang hemat biaya dan ramah lingkungan dengan memanfaatkan ekstrak daun sawit *(Elaeis guineensis Jacq.*) sebagai agen pereduksi dan penstabil. Selanjutnya, doping dengan barium (Ba) dilakukan untuk meningkatkan sifat fotokatalitik, optik, dan listrik dari TiO_2 yang dihasilkan. Analisis fitokimia menunjukkan bahwa ekstrak daun sawit mengandung alkaloid, flavonoid, terpenoid, saponin, dan tanin, yang berperan dalam proses reduksi dan stabilisasi nanopartikel TiO_2. Karakterisasi menggunakan *Fourier Transform Infrared Spectroscopy* (FTIR) mengidentifikasi ikatan Ti–O pada TiO_2 murni pada bilangan gelombang 588 cm^{-1} dan pergeseran puncak serapan pada rentang 548–595 cm^{-1} pada TiO_2 terdoping Ba menunjukkan pembentukan fase barium titanat. Analisis *X-ray Diffraction* (XRD) menunjukkan bahwa doping Ba meningkatkan ukuran kristal TiO_2 dari 1.17 nm menjadi 1.65 nm, yang dapat berimplikasi pada aplikasi fotokatalitik.

Kata Kunci: Doping Barium, Ramah Lingkungan, Sintesis Hijau, Titanium Dioksida.

1. Introduction

Nanotechnology has undergone rapid development in recent decades, offering innovative solutions to a wide range of environmental and technological challenges [1]. One area that has received significant attention is the synthesis of nanoparticles (NPs), which have great potential in the detection, diagnosis, and remediation of environmental contaminants [2]. Among various nanotechnology materials, metal nanoparticles, particularly titanium dioxide (TiO_2), stand out for their unique properties and wide applications.

Conventional nanoparticle synthesis methods, such as sputtering, solvothermal, chemical reduction, sol-gel, chemical vapor deposition, and electrochemistry [3], often involves high costs, the use of toxic chemicals, extreme operating conditions, and potential environmental hazards [4,5]. Therefore, the development of more sustainable and environmentally friendly synthesis methods is very important.

Green synthesis has emerged as a promising alternative, utilizing natural materials such as plant extracts to reduce metal ions into nanoparticles [6]. This approach offers advantages like simplicity, biocompatibility, and cost-effectiveness [7]. Plant extracts contain various phytochemicals with potent antioxidant and reducing properties, which not only facilitate nanoparticle formation but also minimize the risk of contamination and environmental pollution [8]. These compounds, along with proteins that function as natural stabilizing agents, enable the synthesis of stable and uniform nanoparticles [9,10]. This aligns with research [11].

Titanium dioxide (TiO_2) is a highly attractive NP for green synthesis due to its outstanding optical, dielectric, biocompatibility, and thermal stability properties [5]. The applications of TiO_2 are highly diverse, ranging from pigments in paints and cosmetics to photocatalyst in the degradation of organic pollutants and electrodes in lithium batteries. In the context of TiO_2 nanoparticle synthesis, the utilization of oil palm leaf extract (*Elaeis guineensis Jacq.*) as a reducing and stabilizing agent presents a promising sustainable and economical approach [12] .In line with this, research by [13] indicates that the concentration of palm leaf extract has a significant influence on the morphology and size of the TiO_2 nanoparticles formed. Specifically, a lower extract concentration (10% v/v) tends to yield the formation of more defined rod-like structures, while increasing the concentration to 20% and 30% v/v results in shorter rod structures with a tendency for increased particle size. Furthermore, the concentration of palm leaf extract also affects the optical properties of TiO_2, yielding band gap energies of 2.98 eV, 3.08 eV, and 3.15 eV, respectively. To produce TiO_2, TTIP is used as a precursor in green synthesis because it is soluble in water based palm leaf extract. According to [14], the advantage of TTIP lies in its compatibility with such natural solvents, thus enabling the efficient and environmentally friendly formation of TiO_2.

In this research, Ba doping process was carried out on TiO_2 to further enhance the, optical, and electrical properties of the TiO_2 NPs. According to research by [15] Ba doping is expected to modify the crystal structure, expand the light absorption range, reduce the recombination of electron-hole pairs, and improve the thermal stability of TiO_2. Furthermore, Ba doping can influence the particle size and surface area of the nanoparticles, which ultimately enhances the material's performance in various applications. Therefore, this research aims to explore the synthesis of Ba doped TiO_2 NPs using palm leaf extract.

2. Experimentals

2.1 Equipment and Materials

Palm leaf powder, Titanium Tetraisopropoxide (TTIP), deionized water, Barium Nitrate ($Ba(NO_3)_2$), Whatman filter paper no. 42, aluminum foil, pyrex glassware, drip pipette hotplate, magnetic stirrer, porcelain cup, stirring rod, analytical balance, watch glass, funnel, centrifuge, oven, furnace.

2.2. Palm Leaf Extraction

Extraction of palm leaves was done with a modified procedure from [16], which is detailed in Figure 1. The palm leaves used are dark green leaves from Tanjung Morawa, which have gone through a process of washing, drying, and pulverizing. Palm leaf aqueous extract was prepared in a ratio of 1:10 (w/v) using deionized water heated at 70°C for 1 hour with 150 rpm stirring. The filtrate was collected after filtration using Whatman No. 42 filter paper and stored at 4°C. In addition, a phytochemical screening was carried out to determine the content of bioactive compounds in the extract of the palm leaf.

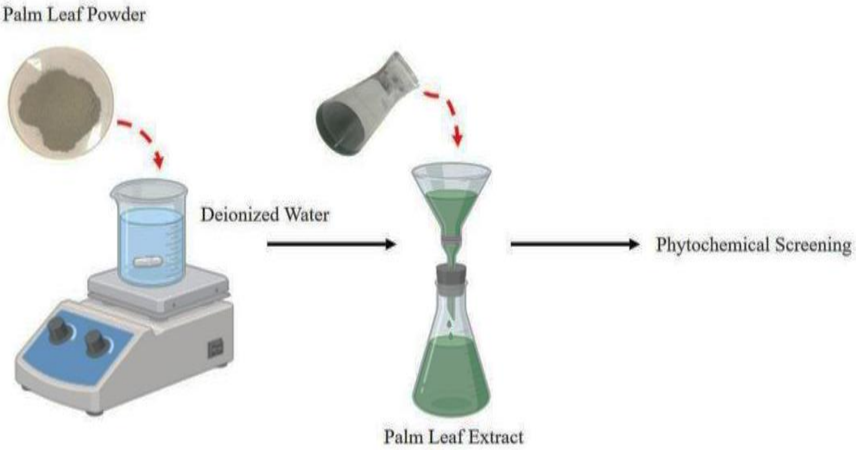

Figure 1. Palm Leaf Extract

2.3 Biosynthesis of TiO_2

Synthesis of TiO_2 using palm leaf extract refers to research conducted by [17]. The TiO_2 biosynthesis process begins by adding 3.5 M TTIP to 10 mL of distilled water. Stir with a magnetic stirrer at 200 rpm for 4 hours Then, 15 mL of palm leaf extract was slowly added. For the synthesis of Ba-doped TiO_2 with concentrations of 1 %, 2 % and 3 %, $Ba(NO_3)_2$ was added at this stage. The solution was allowed to gel for 24 hours, then centrifuged for 20 minutes at 2500 rpm.The characterization of the final product was carried out by means of Fourier transform infrared spectroscopy (FTIR).

3. Result and Discussion

3.1 Phytochemical Screening

To identify the content of bioactive compounds in palm leaf extract, qualitative phytochemical analysis was performed as an initial screening method. The purpose of this phytochemical analysis is to detect the presence of secondary metabolites believed to be present in palm leaves. The phytochemical analysis performed includes tests for the presence of alkaloids, flavonoids, terpenoids, saponins, tannins and steroids. The results of the phytochemical analysis are presented in Table 1 and Figure 2.

Table 1. Phytochemical Screening Results of Palm Leaf Extract (*Elaeis guineensis Jacq*.)

No	Test	Reagent	Theoretical Result	Result of Observation	Result
1.	Alkaloid	Mayer	Yellowish precipitate	Yellowish precipitate	+
		Bouchardart	Brick Red Precipitate	Brick Red Precipitate	+
2.	Flavonoid	$FeCl_3$ 5%	Black Colloid	Black Colloid	+
		H_2SO_4 98%	Yellowish Orange Solution	Yellowish Orange Solution	+
3.	Terpenoid	Salkowsky	Red Solution	Red Solution	+
		Liebermann-Burchard	Bluish Green Solution	Bluish Green Solution	+
4.	Saponin	Aquadest	Forms a stable froth	Forms a stable froth	+
5.	Tanin	$FeCl_3$ 5%	Black Colloid	Black Colloid	+
6.	Steroid	Salkowsky	Red Solution	Yellowish precipitate	-
		Liebermann-Burchard	Bluish Green Solution	White Solution	-

Description : + (Detected)
- (Not Detected)

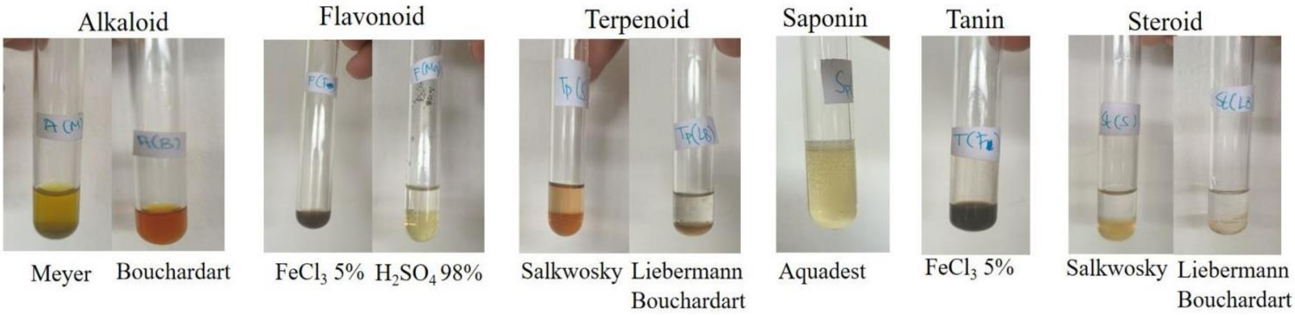

Figure 2. Pythochemical Screening of Palm Leaf Extract (*Elaeis guineensis Jacq.)*

In the biosynthesis of nanoparticles (NPs), plant extracts act as reducing agents and stabilizers of the nanoparticles [18]. Palm leaf extract contains secondary metabolites such as flavonoids, tannins and terpenoids [11]. Flavonoids and saponins have been reported to be capable of controlling the kinetics of nanostructure formation and preventing aggregation [19].

3.2 Results of TiO_2 Biosynthesis

Green synthesis TiO_2 was performed using TTIP precursors with barium (Ba) doping variations of 0%, 1%, 2%, and 3% (w/v). The morphology of the green synthesized TiO_2 product is shown in Figure 3. The results of the synthesis were white TiO_2 solids with a mass of 2.2544 grams, 2.294 grams, 2.315 grams, and 2.385 grams, respectively. The increase in solid mass along with the increase in Ba doping concentration indicates the successful insertion of Ba atoms into the TiO_2 crystal structure. This is in line with the [20] which shows that the increase in Mg atoms in TiO_2 results in an increase in the mass of TiO_2 and a decrease in the composition of Ti in the TiO_2 structure. This also shows the success of doping by replacing Ti cations by Mg^{2+}.

Figure 3. TiO_2 Powder

By using *Elaeis guineensis Jacq* extract as a reducing agent and TTIP precursor. The reaction steps are as follows [21]: TTIP, ($Ti[OCH(CH_3)_2]_4$), Titanium (Ti) in TTIP has an oxidation number is +4.Titanium hydroxide $Ti(OH)_4$ is produced when TTIP reacts with water in a hydrolysis reaction. The reaction that occurs is:

$$Ti[OCH(CH_3)_2]_4 + 4H_2O \rightarrow Ti\,(OH)_4 + 4OH\text{-}CH(CH_3)_2$$

Furthermore, the palm leaf extract is a reducing agent that transfers electrons to titanium hydroxide, leading to a decrease in Ti's oxidation state from +4 to +3 :

$$Ti(OH)_4 + e^- \rightarrow Ti(OH)_3 + OH^-$$

Redox reactions involving oxygen cause the core of Ti^{3+} to evolve into TiO_2 NPs. Under these reaction conditions, which can determine the correct nucleation and growth mechanism. During the reduction and growth process, secondary metabolites in the extract, such as flavonoids, can be adsorbed on the surface of the formed TiO_2 nanoparticles. TiO_2 NPs form NP molecules with various shapes, such as triangular, cubic, spherical, pentagonal, hexagonal, etc. At this stage, the plant extract acts as a stabilizer to maintain a stable morphology [22]. The reaction for the formation of TiO_2 nanoparticles is as follows:

$$Ti[OCH(CH_3)_2]_4 + 4H_2O + ne^- \rightarrow TiO_2 \text{ nanoparticles} + 4OH\text{-}CH\,(CH_3)_2$$

3.3 FTIR Analysis of TiO_2

The FTIR spectroscopy technique confirmed the analysis of the functional groups of the synthesized material. The FTIR spectra of pure TiO_2 and Ba doped TiO_2 are presented in Figure 4. The organic hydroxyl functional group, O-H, displays a stretching vibration at wave number 3440 cm^{-1}.This group appears due to the absorption process of deionized water absorbed on the surface of the sample [23]. This is in line with research [24]. The absence of an absorption peak at 2900 cm^{-1}, associated with C-H stretching vibrations, indicates that organic compounds have been removed after calcination at 400°C for 4 hours [7].

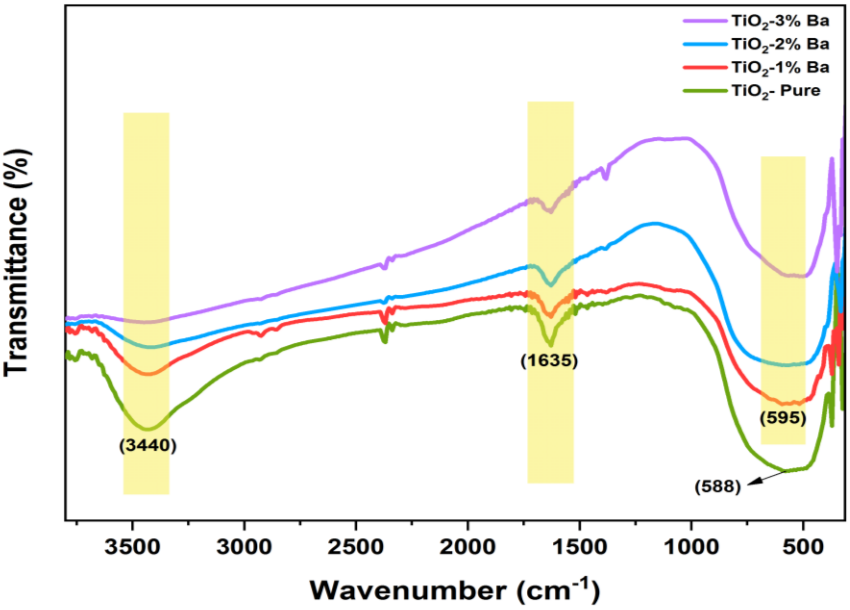

Figure 4. FTIR spectra of pure TiO_2 and Ba doped TiO_2

In wave number 1635 cm^{-1} shows carbonyl functional group, C=O with *streaching* vibration. The palm leaf extract may be absorbed by the sample, causing this cluster to appear. Furthermore, at wave number 588 cm^{-1} [2, 25] on pure TiO_2 shows Ti-O bonds, indicating the formation of titanium oxide. The absorption peak shifts in the range of 548-595 cm^{-1} in Ba-doped TiO_2, indicating the overlap of Ti-O and Ba-O bond vibrations, which indicates the formation of a barium titanate phase. The similar vibrational frequencies and coupling interactions between Ti-O and Ba-O bonds in the crystal structure can explain this phenomenon.

3.4. XRD Analysis of TiO_2

XRD was employed to examine the crystal size of both Ba-doped and undoped TiO_2. Figure 5 illustrates the XRD diffractogram of pure TiO_2 and Ba-doped TiO_2. The XRD peaks of both undoped and Ba-doped TiO_2 are almost entirely consistent, located at 2θ = 25.41, 25.41, 26.49, 57.71, 60.18, 84.38, 85.30, and 89.73, corresponding to the (101), (104), (200), (121), and (204) planes, in accordance with JCPDS card No. 78-2486, which indicates the anatase phase of TiO_2 [15]. It can also be observed that the diffraction peak position of TiO_2 (101) shifted to a lower angle after the addition of Ba, suggesting an increase in lattice parameters due to the successful incorporation of Ba^{2+} ions into the TiO_2 lattice [23,26].

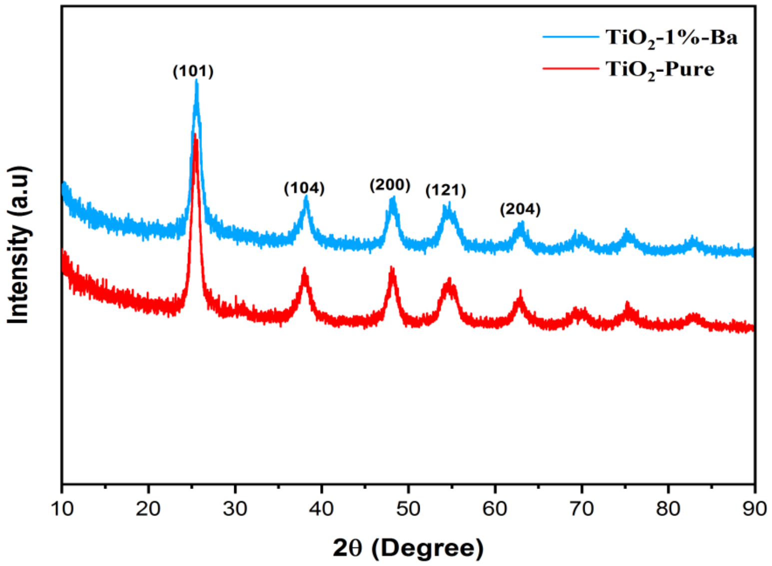

Figure 5. XRD diffractogram of pure TiO_2 and Ba doped TiO_2

The incorporation of Ba into the TiO_2 lattice resulted in an increased crystallite size of 1.65 nm, attributed to the larger ionic radius of Ba^{2+} 1.65 nm compared to that of pure TiO_2 1.17 nm. The presence of Ba within the TiO_2 lattice causes lattice distortion, which hinders nucleation growth [27]. This aligns with research [28] indicating that Ba doped TiO_2 1.42 nm exhibits a larger crystallite size compared to undoped TiO_2 0.74 nm.

4. Conclusion

This study successfully developed a green synthesis method of TiO_2 using palm leaf extract, which is an environmentally friendly and economical approach. The doping with barium has been shown to be effective in modifying the properties of the TiO_2 produced. The development of more sustainable and efficient nanotechnology materials that have the potential to be applied in various fields is a result of this research.

5. Acknowledgement

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6. Conflict of Interest

The authors declare that there are no competing interests in relation to this article.

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