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SPECIAL ISSUE ARTICLE

Oil Palm (*Elaeis Guineensis*) Leaf Extract-Mediated Green Synthesis of TiO_2 Nanoparticles and Their Optical Characteristics

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ABSTRACT

This work aims to explore the characteristics of TiO_2 NPs green-synthesized via an environmentally friendly method using oil palm (*Elaeis guineensis*) leaf extract as a green medium and capping agent by utilizing titanium tetra isopropoxide (TTIP) as precursor. The oil palm leaves were extracted with different solvent concentration variations. The natural extract was characterized using liquid chromatography-mass spectroscopy (LC-MS) and infrared spectroscopy for active chemical contents and functional groups. The obtained TiO_2 NPs were also characterized using infrared spectroscopy (FTIR) for the functional groups, ultraviolet spectroscopy (UV-DRS) for the optical characteristics, and X-ray diffraction (XRD) for the phase formation and crystallographic properties. More sophisticated equipment of field emission scanning electron microscopy equipped with energy dispersive X-ray spectroscopy (FESEM/EDX) and Raman spectroscopy were employed to reveal the characteristics of the obtained TiO_2 NPs. The results from X-ray diffraction showed that the obtained TiO_2 NPs are in pure anatase crystal structure. There is also a trend that the bandgap energy of TiO_2 NPs reduces with the use of oil palm leaves extract as a green medium. It is confirmed that the green medium affects the optical characteristics of the synthesized TiO_2 NPs by reducing the bandgap energy from 3.2 eV for commercial and the one synthesized using ethanol only to 3.12 eV by using the green medium of oil palm leaves extract. These findings will provide insight for more novel, environmentally friendly nanomaterials synthesis methods.

Keywords: Green synthesis, Nanoparticles, Natural plant extract, Oil palm, Titanium dioxide

Introduction

Titanium dioxide is an inorganic compound derived from titanium with the chemical formula TiO_2. It can be found naturally in ilmenite, rutile, and anatase. This material is widely used for its brightness and various applications, including cosmetics, paints, foods, air purification, and bacterial degradation.[1] For a specific application, such as in nanotechnology, TiO_2 can be synthesized via many routes. Several routes are available to synthesize TiO_2 nanoparticles, including chemical[2] and physical methods.[3] The main problem in almost all these chemical or

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physical processes is the less eco-friendly approach, and thus they may impose risks to the environment.[4,5] Because of that, an environmentally friendly method to synthesize TiO_2 NPs by utilizing natural resources in the form of extracts from plant parts[6,7] has attracted special attention due to its simple protocol, cost-effectiveness, and sustainability.[8] The process involves simple steps in selecting suitable plant parts, solvent, and non-toxic elements during production.[9] The utilization of these natural resources has many advantages, such as energy efficiency, product selectivity, and safety for human health and the environment.[10]

Several plant extracts have been used as media for the green synthesis of metal oxide nanoparticles, with a broad range of applications. Septiningrum et al. used mangosteen pericarp extract as a medium in green-synthesizing Ag/TiO_2 nanocomposites for pollutant degradation.[6] Hussain et al. have explored the use of *Morus nigra* leaf extract as a medium in green-synthesizing TiO_2 nanoparticles with biological potential.[11] Rajendhiran et al. have analyzed the characteristics of TiO_2 nanoparticles green-synthesized using *Terminalia* catappa and *Carissa carandas* fruit extracts.[12] Sofyan et al. used *Terminalia* catappa fruit extract as a medium for green-synthesizing TiO_2 nanoparticles and applied the resulting nanoparticles as a semiconductor layer in a dye-sensitized solar cell.[8] Patil and his coworker used *Bixa orellana* seed extract as a green medium to synthesize silver nanoparticles for pharmacological applications,[13] while Maurya et al. used it as a medium to synthesize green TiO_2 nanoparticles for solar cell applications.[14] Oleiwi et al. have explored the use of orange peel as a green medium for synthesizing TiO_2 nanoparticles, comparing it with the conventional sol-gel method.[15] Recently, Sofyan et al. have also investigated the properties of TiO_2 nanoparticles green-synthesized with *Uncaria gambir* extract for dye-sensitized solar cell applications.[16]

Oil palm (*Elaeis guineensis*) is a species of palm tree that is widely cultivated for its oil-rich fruits. It is well known that its leaves are rich in catechins and polyphenols.[17,18] Several investigators have used oil palm leaves as free radical scavengers,[19] antibacterials,[20] and adsorbers for pollution.[21] However, in the plantation, the leaves are mostly discarded and left to decompose between the oil palm trunks.[22] Considering the active contents of the oil palm leaves, there is a possibility that this extract from oil palm leaves could be useful as a medium in an eco-friendly synthesis of TiO_2 NPs. To the best of the authors' knowledge, no literature has been published on using oil palm leaves as a medium in the green synthesis of TiO_2 NPs. In this study, oil palm leaves were extracted using various solvents and concentrations. The extract was then utilized as a medium for the green synthesis of TiO_2 NPs. The primary objective of this work is to investigate the characteristics of TiO_2 NPs green-synthesized using oil palm leaf extract, which can later be employed in photocatalysis and/or energy harvesting applications.

Materials and methods

Materials

The chemicals include ethanol (Merck), acetylacetone ($C_5H_8O_2$, Sigma Aldrich), commercial TiO_2 (Merck), polyvinylidene fluoride (PVDF, Merck), N-Methyl-2-pyrrolidone (NMP, Merck), commercial dye N719 ($C_{58}H_{86}O_8N_8S_2Ru$, Solaronix), platinum paste (Platisol, Solaronix), and electrolyte solution (Iodolyte Z-50, Solaronix). The precursor for the synthesis of TiO_2 was titanium tetra isopropoxide (TTIP, $Ti\{OCH(CH_3)_2\}_4$, Sigma Aldrich). All chemicals are reagent grade and were used without further treatment.

Oil palm leaf extract preparation

The oil palm leaf extract was prepared following the work of others[17] with slight modifications, as explained below: Oil palm leaves obtained from a farmer in Pangkalan, West Sumatra, were cleaned under running tap water and allowed to dry before being chopped with a sharp knife. Four different extract variations were prepared using ethanol with 0, 10, 30, and 50% acetylacetone. In this work, acetylacetone was used as a chelating agent to control the growth, stability, and crystallinity of TiO_2 nanoparticles.[23] For each variation, 20 g of chopped oil palm leaf was blended in 100 mL of solvent using a household blender for five minutes. Further, the mixture was filtered to separate the insoluble residue from the solvent, and extracts from the oil palm leaf with varying solvent concentrations were obtained and stored in a dark bottle. The oil palm leaf extract was analyzed for its chemical composition and functional groups using liquid chromatography-mass spectrometry (LC-MS; ACQUITY UPLC® H-Class System-Xevo G2-S QTof, Waters) and Fourier transform infrared spectroscopy (FTIR; PerkinElmer ATR Spectrum One).

Green synthesis of TiO_2 nanoparticles

The environmentally friendly synthesis of titanium dioxide in the oil palm extract medium followed the sol-gel method[24] with slight modification. The

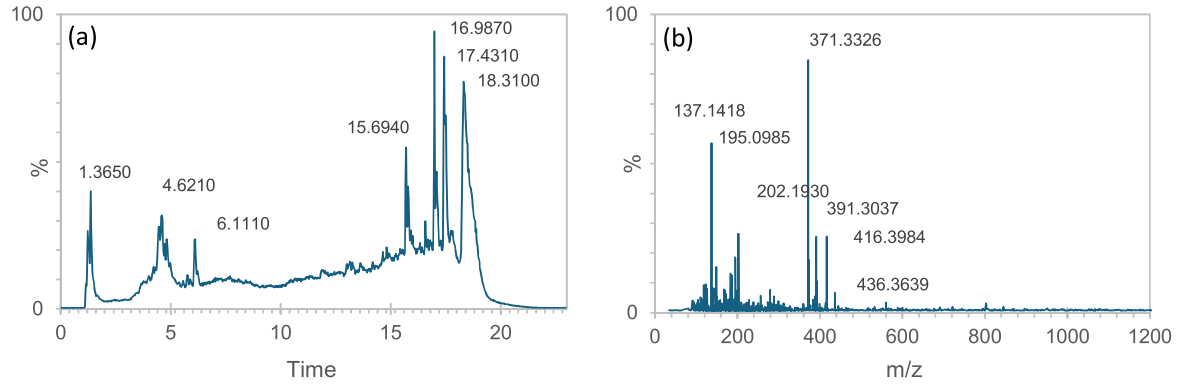

Fig. 1. Oil palm leaf extract (a) chromatogram and (b) spectrum obtained using LC-MS spectroscopy.

procedure is explained as follows. Five beaker glasses were prepared, and 10 mL of titanium tetra isopropoxide (TTIP, $Ti\{OCH(CH_3)_2\}_4$, Sigma Aldrich) was emptied into each beaker glass. Ten mL of ethanol was added dropwise to the first beaker glass (labelled as Eth), and ten mL of oil palm extract from various solvents explained in the previous section was added dropwise to the rest of the beaker glasses and labelled as P0, P10, P30, and P50, respectively. The mixture was agitated with a magnetic stirrer for 3 hours, then left overnight and filtered to obtain a wet gel. The gel was dried at 100 ˚C for 1 hour and calcined at 450 ˚C for 2 hours to obtain white powders of TiO_2 NPs. The obtained products were examined using FTIR, ultraviolet-visible spectroscopy (UV-Vis, Shimadzu UV2450), X-ray diffraction (XRD, AERIS Malvern PANalytical), Raman spectroscopy (HORIBA iHR320), and field emission scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (FESEM/EDX, FEI Inspect F50). For characterization using UV-Vis and XRD, the number of specimens was triplicate for statistical analysis, employing one-way analysis of variance (ANOVA) in MatLAB®.

Results and discussion

Active contents and functional groups

We obtained the chromatogram and mass spectrum of the active components in oil palm leaf extract using LC-MS. As can be seen from the chromatogram and spectrum given in Fig. 1a and 1b, respectively, oil palm leaf extract is detected to contain several active chemical compounds, most of which are dominated by tolylsulfonic acid at retention times 3.6–4.6, propenoic acid at retention times 5.6–6.1, and dicyclohexyl ethyl phosphate at retention times 14.4–15.7, as well as other unidentified active chemical components at higher retention times. These active compounds are expected to play an essential role in the green synthesis of TiO_2 nanoparticles.

To confirm the active compounds identified by LC-MS, the oil palm leaf extract was further analyzed by infrared spectroscopy. This infrared absorption can be further used to determine the functional groups present in the molecule. The functional group spectrum of the oil palm leaf extract obtained using infrared spectroscopy is given in Fig. 2a, whereas the infrared spectra of commercial TiO_2 NPs, TiO_2 NPs synthesized using ethanol only, and TiO_2 NPs synthesized via the green method using oil palm leaf extract are given in Fig. 2b. The infrared spectrum of oil palm leaf extract shows several absorption bands. In general, as can be seen from the figure, a broad signal is detected at 3000–3600 cm^{-1} from the O-H group as an indication of a phenol compound. The presence of the C=C group with the ring of aromatic compound is detected at 1570.12 cm^{-1}; the alkane compound with C-H stretching is detected at 1417.74 cm^{-1}; the type of alkene compound with C=C bending is detected at 751.31-872.83 cm^{-1}; whereas the typical alcohol compound with medium weak intensity of C-O stretching is detected at 1069.57-1181.45 cm^{-1}. The absorption peaks at the regions 3366 cm^{-1}, 1650 cm^{-1}, and 679 cm^{-1} are expected from the characteristics of flavonoids[25] and as an indication of the presence of quercetin and related compounds known as flavonoid compounds. The yellow colors in the oil palm leaf extract are expected to be due to hydroxy and methoxy derivatives of flavones and isoflavones[26] and are believed to influence the crystal growth of TiO_2 NPs during the green synthesis.[27]

To observe how oil palm leaf extract affects the characteristics of TiO_2 NPs after green synthesis, researchers collected the spectra of TiO_2 NPs and those from commercial TiO_2, which are provided in Fig. 2b. For commercial TiO_2 NPs, weak stretching vibrations of hydrogen-bonded water molecules, bending vibrations of the C=C group, and the Ti-O-Ti bridging

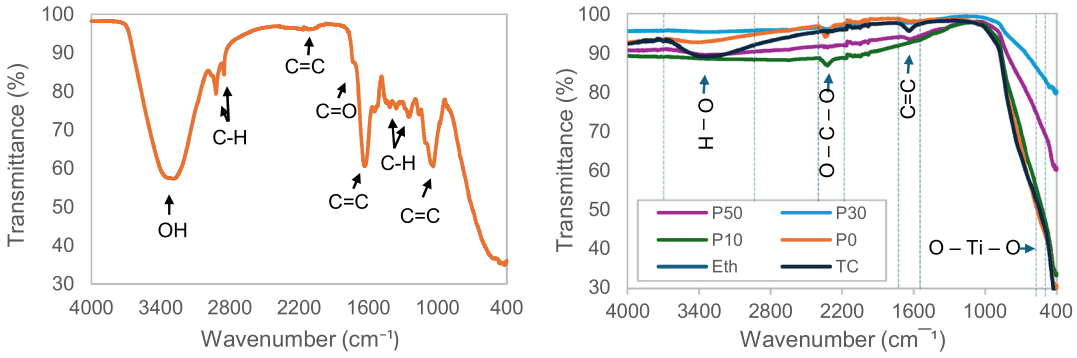

Fig. 2. Infrared spectra of (a) oil palm leaf extract and (b) TiO_2 NPs: commercial (TC), synthesized using ethanol (Eth), and green-synthesized using oil palm leaf extract (P0).

stretching mode are detected at around 3370, 1630, and 650 cm^{-1}, respectively.[28] The presence of OH groups is expected due to the interaction of TiO_2 NPs with environmental moisture.[29] The same characteristics were also found in the spectrum of TiO_2 obtained from the synthesis using pure ethanol (Eth) and the green synthesis using extracts from oil palm leaves. An additional weak absorption at 2326 cm^{-1} is also observed, possibly due to O-C-O linear vibrations, as reported in another study.[8] The presence of a certain amount of carbon in TiO_2 NPs may affect morphology, crystallinity, and bandgap energy as reported by others.[30,31]

Optical characteristics

UV-Vis spectroscopy is primarily used to measure a material's light absorption and determine its optical properties, such as the band gap energy. In this work, the optical characteristics of commercial TiO_2 NPs and those obtained from the synthesis were analyzed using UV–Vis DRS. The obtained reflectance spectra (not shown) were further analyzed to determine their bandgap energies using the Kubelka−Munk relation.[32] Firstly, the data was fitted in the Tauc plot $(F(R\infty).hc/\lambda)^{1/2}$ vs hc/λ, assuming the sample has an indirect allowed electron transition.[33] The results from this fitting are illustrated in Fig. 3. By extrapolating the linear part of the Tauc plot to the intersection point of the x-axis, an estimated value of the band gap energy for each TiO_2 NPs was obtained to be 3.20(0.00) eV, 3.20(0.02) eV, and 3.12(0.08) eV for commercial, synthesized using ethanol, and green-synthesized using oil palm leaf extract, respectively. Based on the obtained bandgap energies, the bandgap decreases when a green medium is used. This decrease is expected due to the active chemical contents in the oil palm extract during the synthesis.

For the optical characteristics of TiO_2 NPs green-synthesized with the addition of acetylacetone as a chelating agent, as shown in Fig. 3d, 3e and 3f, the bandgap energy decreases with increasing acetylacetone concentration, i.e., 3.08(0.09) eV, 3.00(0.07) eV, and 2.99(0.08) eV for 10%, 30%, and 50% acetylacetone concentrations, respectively. The values in parentheses indicate the standard error. Statistical analysis shows a significant difference in bandgap energy (p-value = 0.01), which is smaller than the alpha-value of 0.05. It is believed that the decrease in bandgap energy is due to the use of a chelating agent during the green synthesis of TiO_2 NPs. In terms of applications, reduced bandgap energy will play a crucial role in photocatalysis and light-harvesting applications, such as pollutant degradation and dye-sensitized solar cells. A lower bandgap allows materials to absorb a broader spectrum of light, particularly in the visible range, thereby significantly improving the efficiency of converting solar energy into chemical or electrical energy.[34]

Surface morphology and chemical composition

The surface morphology and chemical composition of the obtained TiO_2 NPs were examined using FESEM coupled with EDX. The results, in terms of chemical composition and surface morphology, of commercial TiO_2 NPs synthesized using ethanol only and green-synthesized using oil palm extract are presented in Figs. 4 and 5, respectively. The EDX spectra show that the main peaks of titanium and oxygen are clearly visible. This means that TiO_2 is present and that the green synthesis process worked. In addition, the weight fraction of titanium (62-67%) and oxygen (32-38%) confirms the successful green synthesis of TiO_2 NPs using oil palm leaf extract as a medium and capping agent.[35,36] Before FESEM characterization, the TiO_2 NPs were coated using Au/Pd[37] to increase their conductivity, which is confirmed as unlabelled peaks around 2 keV in the EDX spectra.[38] The presence of the carbon element, as detected using FTIR, cannot

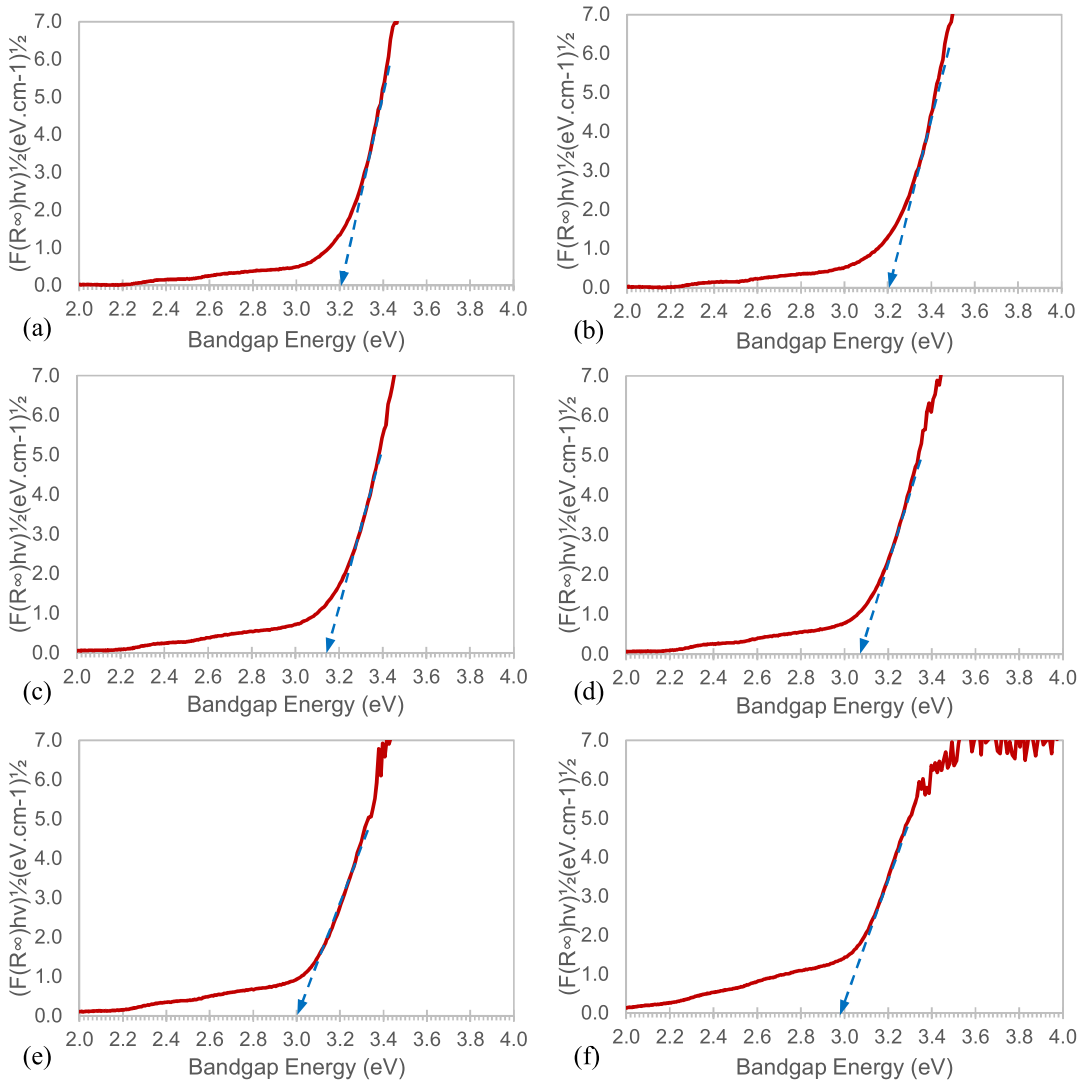

Fig. 3. Tauc plots for bandgap energy determination of (a) commercial TiO_2, (b) TiO_2 synthesized using ethanol only, (c) TiO_2 synthesized using oil palm leaf extract only, (d) TiO_2 synthesized using oil palm leaf extract with 10%, (e) 30%, and (f) 50% acetylacetone.

be identified in the EDX spectrum, probably due to the limitations of the EDX technique.

As shown in Fig. 5, the electron image of commercial TiO_2 (TC) reveals homogeneous sphere-like particles but tends to be agglomerated. Measurement using ImageJ® revealed an average particle size of 190 nm. For the synthesis using ethanol only (Eth), the round morphology is maintained, but the tendency to agglomerate makes the particles appear larger than those of the commercial one, with an average particle size of 345 nm. With the addition of oil palm leaf extract as a medium in the green synthesis, as shown in Fig. 4, the round-shaped particles remain, with a particle size smaller than that of TiO_2 synthesized using ethanol only and resembling that of commercial TiO_2 with an average particle size of 200 nm. Since the medium is the only difference in the synthesis methods, it is confirmed that oil palm leaf extract affects the particle growth of TiO_2 NPs during the green synthesis. For the samples with the addition of acetylacetone as a chelating agent, as shown in Fig. 5d, 5e and 5f, the electron images indicate that the particle size tends to agglomerate and form large particles at low (10%) and high (50%) acetylacetone concentrations with an average particle size of 486 nm and 625 nm, respectively. The tendency to agglomerate appears low at a 30% acetylacetone concentration, with an average particle size of 430 nm.

Crystallographic properties

X-ray diffraction is a powerful method for determining a material's crystallographic properties. In this

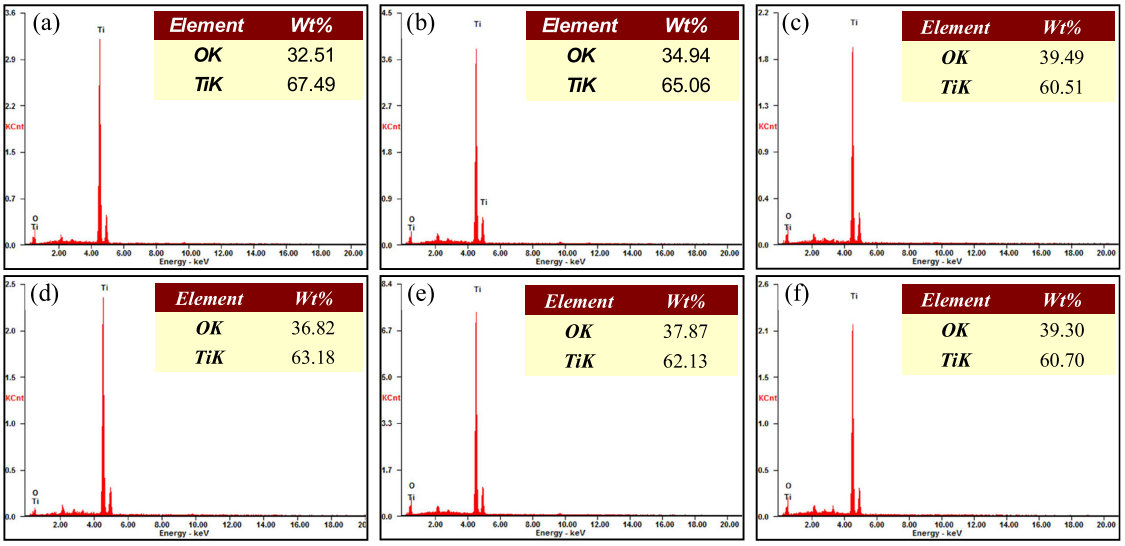

Fig. 4. Energy dispersive X-ray spectra of (a) commercial TiO_2 NPs, (b) TiO_2 synthesized using ethanol only, and TiO_2 NPs green-synthesized using oil palm leaf extract with (c) 0%, (d) 10%, (e) 30%, and (f) 50% acetylacetone. Elemental compositions are given in the inset table.

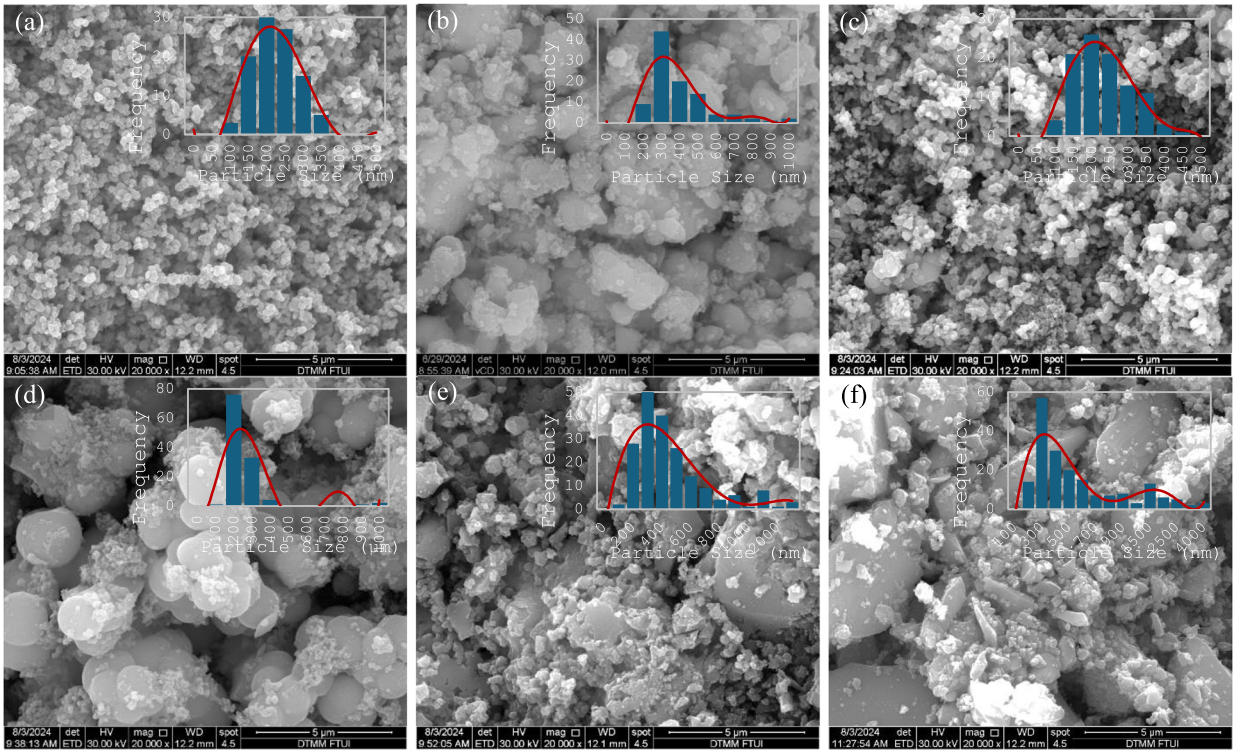

Fig. 5. Secondary electron images of (a) commercial TiO_2, (b) TiO_2 NPs synthesized using ethanol only, (c) TiO_2 NPs green-synthesized using oil palm leaf extract with acetylacetone concentration variations (c) 0%, (d) 10%, (e) 30%, and (f) 50%. The inset graph is the particle size distribution.

work, powder X-ray diffraction was used to analyze the crystallographic properties of TiO_2 NPs synthesized via green methods. As comparisons, commercial TiO_2 NPs and TiO_2 NPs synthesized using ethanol only were also examined. The results are provided in Fig. 6. The diffractogram of commercial TiO_2 NPs shows very sharp peaks around 2θ 25.0°, 36.6°, 37.5°, 38.3°, 47.7°, 53.6°, 54.8°, 62.4°, 68.5°, 70.2°, and 74.8°, indicating the presence of (101), (103), (004), (112), (020), (105), (211), (204), (116), (220), and

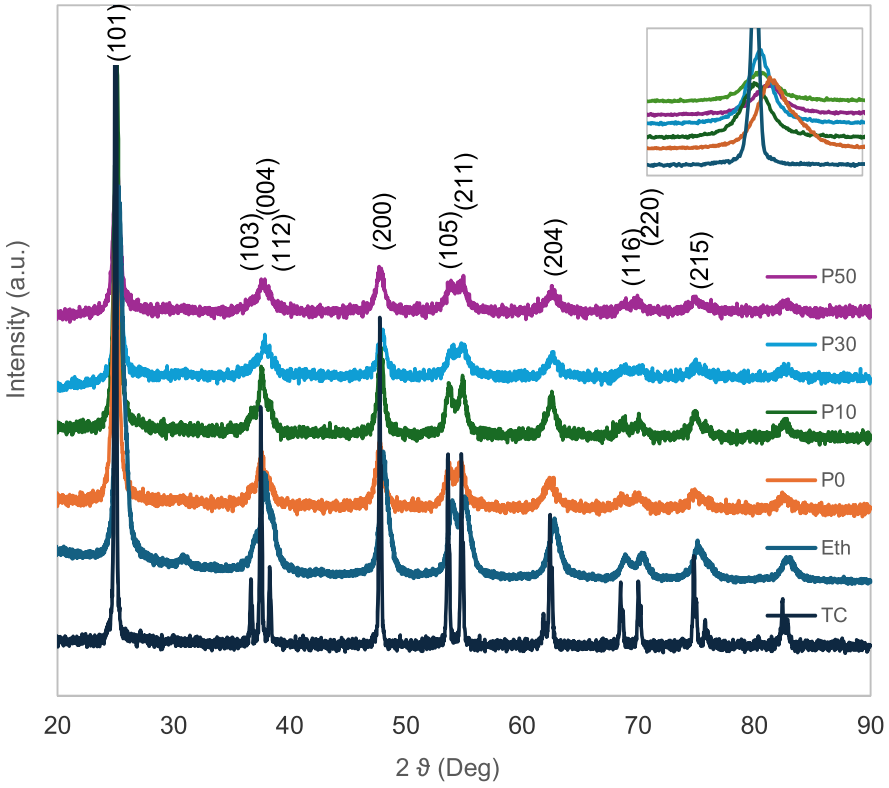

Fig. 6. X-ray diffractograms of TiO_2 NPs (TC), synthesized using ethanol only (Eth), and green-synthesized using oil palm leaf extract with 0% (P0), 10% (P10), 30% (P30), and 50% (P50) acetylacetone. The inset shows a peak shift.

(215) planes, respectively. No other impurities were detected. The analysis indicated that these peaks belong to the anatase phase (ICSD 98-000-9853). At the same time, the diffractograms of the obtained TiO_2 NPs also reveal the same prominent peaks as the commercial TiO_2 NPs (ICSD 98-015-4604), also with no other phase detected except for the one synthesized using ethanol only at around 31.6° due to the presence of the brookite phase (ICSD 98-015-4605).[8,39] These findings agree with the results obtained by others.[40]

As shown in the diffractogram, the obtained TiO_2 NPs exhibit high crystallinity, as indicated by sharp, intense peaks. No new peaks indicative of phase formation were detected in the green medium with the green extract. It can be confirmed that using a green medium has not affected crystal structure formation during the green synthesis. However, as can be inspected in the plane (101), a slight shift is observed in all peaks of the synthesized TiO_2 NPs to the higher angle as compared to that of commercial TiO_2 NPs. This shift is expected due to crystallite size and lattice strain,[41] which affect the Bragg peak and shift the peak angle accordingly. This shift was also observed in our previous work.[8] To further deepen our understanding of the effect of green medium solvent variations on the crystallographic properties of TiO_2 NPs, Rietveld analysis was performed using Highscore Plus[42,43] on all TiO_2 NPs diffractograms. The results, summarized after Rietveld refinement, are presented in Table 1.

The refinement indicated that there is no effect of the green medium or the chelating agent on the lattice parameters and, consequently, on the unit cells. For the crystallite size, there is a tendency for it to decrease with the use of palm leaf extract compared with commercial TiO_2 and with ethanol-only synthesis. With the addition of acetylacetone, the crystallite size increases at low concentration and then decreases again with the increase of acetylacetone concentration. One-way analysis of variance shows that there is indeed a significant difference in crystallite size (p-value = 0.0267), which is smaller than the alpha-value of 0.05.

To compare with the results obtained in this work, Table 2 presents the media used for the green synthesis of TiO_2 NPs and their key parameters across different plant sources.

Raman spectroscopy

Raman spectroscopy is a powerful technique to identify chemical structures and compositions by analyzing how light interacts with molecular bonds. In

Table 1. Lattice parameters of TiO_2 NPs from the green synthesis and the commercial one.

Sample	Lattice parameter			Goodness of fit (χ^2)	Crystallite size (nm)
	a (Å)	c (Å)	V (Å)^3		
TC	3.7806(1)	9.5017(3)	135.8053	1.0533	96.4616(0.00)
Eth	3.7858(5)	9.4990(2)	136.1514	1.9600	10.7745(0.75)
P0	3.7864(5)	9.4990(2)	136.1845	1.0000	9.5606(0.07)
P10	3.7857(5)	9.5069(1)	136.2284	1.2763	11.5779(0.01)
P30	3.7920(1)	9.5000(3)	136.6238	1.9209	9.8167(0.15)
P50	3.7883(9)	9.4880(3)	136.1611	1.4748	9.2924(0.38)

Table 2. A comparative table of key parameters from the green synthesis of TiO_2 NPs across plant sources.

Medium	Variation	Phase	Bandgap (eV)	Crystallite Size nm)
Mangosteen pericarp (MP)[6]	TiO_2 P25	Anatase/rutile	3.17	-
	Ag/TiO_2 5 mM	Anatase/rutile	3.09	-
	Ag/TiO_2 15 mM	Anatase/rutile	3.06	-
	Ag/TiO_2 45 mM	Anatase/rutile	3.00	-
Tropical almond (TA) fruit[8]	TiO_2 P25	Anatase/rutile	3.19	17.03
	No TA	Anatase/brookite	3.19	12.53
	0.6% TA	Anatase	3.14	10.43
	0.8% TA	Anatase	3.12	12.93
	No TA + 0.0017% graphene oxide (GO)	Anatase	3.18	16.30
	0.6% TA + 0.0017% GO	Anatase	3.16	11.83
	0.8% TA + 0.0017% GO	Anatase	3.16	9.31
	No TA + 0.16% GO	Anatase	3.16	18.32
	0.6% TA + 0.16% GO	Anatase	3.15	13.61
	0.8% TA + 0.16% GO	Anatase	3.15	11.62
Tropical almond (TA) and *Carissa carandas* (CC)[12]	0.2% TA	Anatase	3.28	21.00
	0.4% TA	Anatase	3.26	17.00
	0.6% TA	Anatase	3.24	12.00
	0.8% TA	Anatase	3.21	8.00
	0.2% CC	Anatase	3.30	20.00
	0.4% CC	Anatase	3.28	19.00
	0.6% CC	Anatase	3.26	15.00
	0.8% CC	Anatase	3.24	10.00
Bixa Orellana (BO)[14]	No BO	Anatase/brookite	3.20	13.00
	With BO	Anatase	2.90	9.00
Orange peel (OP)[15]	No OP	Anatase/brookite	2.85	30.20
	With OP	Anatase	3.25	24.90

this work, Raman spectra were used to confirm the X-ray diffraction analysis. As shown in Fig. 7a, the spectrum of commercial TiO_2 NPs shows a strong characteristic as indicated by the peaks centering at 143 cm^{-1}, 197 cm^{-1}, 396 cm^{-1}, 514 cm^{-1}, and 637 cm^{-1}. These peaks are attributed to the anatase phase.[44,45] Hence, it is confirmed that the commercial TiO_2 NPS contains 100% anatase. The spectrum of TiO_2 NPs synthesized using ethanol only (Eth) shows the same characteristics as that of commercial TiO_2 NPs, but with the addition of a tiny peak at around 326 cm^{-1}. This little peak is due to a small brookite phase, as detected by XRD analysis mentioned in the previous section.[46] The Raman spectra of green-synthesized TiO_2 NPs follow the same trend as the commercial one and are present in a pure tetragonal anatase phase. A slight blue shift as illustrated in Fig. 7b, however, is detected as indicated by the corresponding five characteristics with the principal peak locating around 144 cm^{-1} followed by other peaks at 197-199 cm^{-1}, 397 cm^{-1}, 516 cm^{-1}, and 638 cm^{-1}. This blue shift is expected due to variations in crystallite size, as reported by others.[47] The characteristics obtained from these Raman spectra are consistent with the findings revealed by the X-ray diffraction analysis.

Reaction mechanism

To better understand the mechanism behind the green synthesis of TiO_2 nanoparticles (NPs) using oil palm leaf extract, we present a proposed formation mechanism that focusses on both the hydrolysis reaction alone and the hydrolysis reaction in conjunction with the green medium. As has been previously mentioned, with no green medium from the oil palm leaf

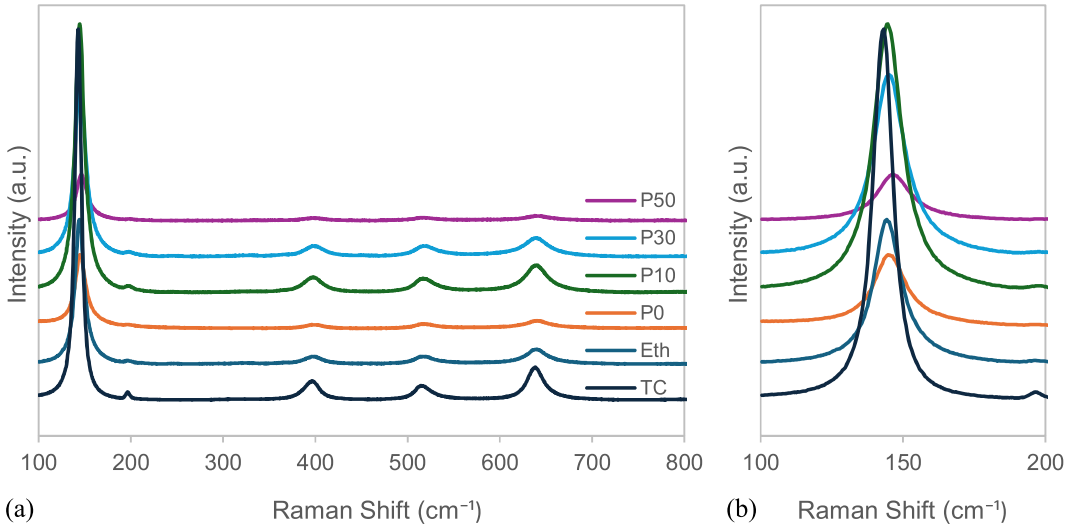

Fig. 7. (a) Superimposed Raman spectra of TiO_2 NPs (TC), synthesized using ethanol only (Eth), and green-synthesized using oil palm leaf extract with 0% (P0), 10% (P10), 30% (P30), and 50% (P50) acetylacetone at 800-100 cm^{-1}, and (b) the region zoomed at 200-100 cm^{-1} shows the blue shift.

extract, the formation of TiO_2 is merely controlled by hydrolysis and condensation reactions represented by the following reaction: [48]

$$Ti[OCH(CH_3)_2]_4 + 2H_2O \rightarrow TiO_2 + 4C_3H_7OH \quad (1)$$

In the presence of oil palm leaves, other factors influence the growth of TiO_2 NPs during the green synthesis, i.e., the active components in the oil palm leaves. In this instant, the reduction of TTIP to TiO_2 involved several reaction steps: [49]

- 1. Formation of titanium hydroxide species:

$$Ti[OCH(CH_3)_2]_4 + 4H_2O \rightarrow Ti(OH)_4 + 4\,OH - CH(CH_3)_2 \quad (2)$$

- 2. Reduction of titanium precursor:

$$Ti\,(OH)_4 + e^- \rightarrow Ti\,(OH)_3 + OH^- \quad (3)$$

- 3. Nucleation and growth:

During nucleation and growth, reduced titanium species form nuclei and subsequently grow into TiO_2 NPs, involving a series of redox reactions that take oxygen from the surrounding environment.

- 4. Growth control and stabilization:

During the nucleation and growth process, the active compounds in the oil palm leaf extract, such as quercetin and gallic, can be adsorbed onto the surface of the forming TiO_2 NPs, which will, in turn, control and stabilize the formation of TiO_2 NPs by preventing agglomeration and providing surface modifications.

- 5. The overall formation reaction of TiO_2 NPs using the oil palm leaf extract can then be summarized as:

$$Ti[OCH(CH_3)_2]_4 + 2H_2O \rightarrow TiO_2NPs + 4OH - CH(CH_3)_2 \uparrow \quad (4)$$

Conclusion

Researchers successfully carried out the environmentally friendly green synthesis of TiO_2 NPs using oil palm leaf extract as reducing and capping agents. All characterizations confirmed the successful outcome of the environmentally friendly synthesis. Although the presence of carbon was detected in the obtained TiO_2 product, it does not alter the TiO_2 NPs crystal structure. There is a tendency for the crystallite size to decrease with the use of palm leaf extract compared with commercial TiO_2 and that synthesized with ethanol only; however, the crystallite size is independent of the chelating agent concentration. The use of a green medium in the synthesis of TiO_2 NPs has also been shown to control the optical characteristics by reducing the bandgap energy, i.e., 3.2 eV for commercial TiO_2 and for TiO_2 synthesized using only ethanol, to 3.12 eV with the green medium of oil palm leaf extract. The bandgap energy of TiO_2 NPs green-synthesized in the presence of acetylacetone also reduces with an increase of acetylacetone concentration, i.e., 3.08 eV, 3.00 eV, and 2.99 eV for 10%, 30%, and 50% acetylacetone concentrations, respectively. A lower bandgap allows materials to absorb a broader spectrum of light,

particularly in the visible range. Hence, reduced bandgap energy will play an essential role in photocatalysis and light-harvesting applications, such as pollutant degradation and dye-sensitized solar cells. The future of green synthesis also aligns with the global drive for sustainable, eco-friendly technologies because green synthesis methods use naturally occurring reducing and capping agents, eliminating the need for toxic chemicals and making the process safer for both the environment and human health.

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the figures and tables in the manuscript are ours. Furthermore, any figures and images that are not ours have been included with the necessary permission for republication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at Universitas Indonesia, Depok 16424, Indonesia.

Authors' contribution statement

N.S.: conceptualization, formal analysis, funding acquisition, methodology, validation, writing – original draft, review, and editing. A.: data curation, funding acquisition, investigation. Y. R.: data curation, funding acquisition, investigation. F.A.: data curation, investigation, and visualization. M. M.: data curation and investigation. M.: data curation and investigation. A. R.: data curation, investigation, and visualization. A.H. Y.: writing – review and editing. D.D.: writing – review and editing.

Data availability

Data will be made available upon request.

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استخلاص أوراق نخيل الزيت (Elaeis guineensis) كوسيط في التخليق الأخضر لجزيئات ثاني أكسيد التيتانيوم (TiO_2) وخصائصها البصرية

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الملخص

يهدف هذا العمل إلى استكشاف خصائص جزيئات ثاني أكسيد التيتانيوم النانوية (TiO_2 NPs) المصنعة بطريقة صديقة للبيئة باستخدام مستخلص أوراق نخيل الزيت (Elaeis guineensis) كوسط أخضر وعامل تغليف، من خلال استخدام رباعي إيزوبروبوكسيد التيتانيوم (TTIP) كمادة أولية. تم استخلاص أوراق نخيل الزيت باستخدام تركيزات مختلفة من المذيبات. وقد تم تمييز المستخلص الطبيعي باستخدام الكروماتوغرافيا السائلة مع مطياف الكتلة (LC-MS) والطيفية تحت الحمراء لاكتشاف المحتويات الكيميائية النشطة والمجموعات الوظيفية. تم أيضًا تمييز جزيئات TiO_2 النانوية المتحصل عليها باستخدام الطيفية تحت الحمراء (FTIR) للمجموعات الوظيفية، والطيفية فوق البنفسجية للخصائص البصرية(UV-DRS) ، والانحراف بالأشعة السينية (XRD) لتكوين الطور والخصائص التبلورية. تم استخدام أجهزة متطورة مثل مجهر إلكترون المسح الانبعاثي الميداني المجهز بمطياف الأشعة السينية المشتت للطاقة (FESEM/EDX)وطيفية رامان للكشف عن خصائص جزيئات TiO_2 النانوية المتحصل عليها. أظهرت نتائج الانحراف بالأشعة السينية أن جزيئات TiO_2 النانوية المتحصل عليها تمتلك بنية كريستالية من نوع الأناتاز النقي. كما لوحظ اتجاه في انخفاض طاقة الفجوة الإلكترونية لجزيئات TiO_2 النانوية مع استخدام مستخلص أوراق نخيل الزيت كوسط أخضر. تم التأكيد على أن الوسط الأخضر يؤثر على الخصائص البصرية لجزيئات TiO_2 النانوية المصنعة من خلال تقليل طاقة الفجوة من 3.2 إلكترون فولت للعينات التجارية وتلك المصنعة باستخدام الإيثانول فقط إلى 3.12 إلكترون فولت باستخدام الوسط الأخضر من مستخلص أوراق نخيل الزيت. توفر هذه النتائج رؤى لطرق تصنيع نانوية صديقة للبيئة وأكثر ابتكارًا.

الكلمات المفتاحية: التخليق الأخضر، الجزيئات النانوية، مستخلص النبات الطبيعي، زيت النخيل، ثاني أكسيد التيتانيوم.