

## GREEN SYNTHESIS AND CHARACTERIZATION OF TiO<sub>2</sub> NANOPARTICLES USING BIO-WASTE EXTRACTS FOR PARACETAMOL PHOTODEGRADATION

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### ABSTRACT

Pharmaceutical residues such as paracetamol are increasingly detected in aquatic environments, posing ecological risks and demanding sustainable removal strategies. In this study, TiO<sub>2</sub> nanoparticles were synthesized via a green route using bio-waste such as avocado peel, petai pod, and pomelo peel ethanol extracts as natural capping and stabilizing agents. FT-IR analysis confirmed the presence of phenolic and flavonoid groups, while UV-DRS revealed slight variations in band gap energy (3.18 - 3.27 eV), with TiO<sub>2</sub>-EEPPo exhibiting the narrowest band gap. XRD patterns showed that all samples were dominated by the anatase phase with crystallite sizes of 9.9 - 17.7 nm, and FE-SEM/PSA analysis demonstrated nanoscale particle sizes (23 - 98 nm) with porous but agglomerated morphologies. Zeta potential values (-17.11 to -24.16 mV) indicated sufficient colloidal stability, while adsorption studies highlighted improved affinity in biowaste-modified TiO<sub>2</sub> due to hydroxyl, carboxyl, and phenolic groups. Photocatalytic degradation experiments demonstrated that photolysis alone removed only 36.52 % of paracetamol after 180 min. Biowaste-modified catalysts also exhibited high efficiencies, with TiO<sub>2</sub>-EEPA, TiO<sub>2</sub>-EEPPi, and TiO<sub>2</sub>-EEPPo degrading 91.75 %, 90.1 %, and 95.89 %, respectively, under UV irradiation. These findings confirm that agro-waste-derived extracts not only enhance the structural and photocatalytic properties of TiO<sub>2</sub> but also provide an eco-friendly approach for pharmaceutical wastewater remediation and bio-waste valorization.

**Keywords:** avocado, green synthesis, nanoparticles, petai, photocatalyst, pomelo, titanium dioxide.

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### INTRODUCTION

The increasing consumption of pharmaceuticals has raised serious environmental concerns due to the continuous release of active pharmaceutical ingredients (APIs) into aquatic ecosystems. Among these, paracetamol (acetaminophen) is one of the most

widely consumed analgesic and antipyretic drugs, with an annual global production of approximately 145 000 tons [1]. In Indonesia, monitoring studies have reported paracetamol concentrations of 610 ng L<sup>-1</sup> in Angke and 420 ng L<sup>-1</sup> in Ancol coastal waters [2]. Such contamination not only reduces water quality but also threatens aquatic organisms, as evidenced by histological and

hematological impairments in fish exposed to paracetamol [3, 4]. Moreover, the persistence of paracetamol in aquatic environments increases the potential for bioaccumulation and long-term ecological risks.

Several treatment methods have been investigated for paracetamol removal from wastewater, including adsorption, biosorption, and photolysis. Adsorption onto activated carbon has been shown to reduce paracetamol levels, but the method is constrained by limited adsorption capacity, strong pH dependency, and challenges in adsorbent regeneration [5, 6]. Biosorption using *Saccharomyces* sp. demonstrated effective removal but required long retention times [7]. UV photolysis can degrade paracetamol; however, it is highly dependent on light intensity and often yields incomplete mineralization [8]. These limitations highlight the need for more efficient and sustainable treatment approaches.

Heterogeneous photocatalysis employing titanium dioxide (TiO<sub>2</sub>) nanoparticles has emerged as a promising strategy for pharmaceutical wastewater treatment. TiO<sub>2</sub> is chemically stable, inexpensive, and non-toxic, and under UV irradiation it generates reactive oxygen species (ROS) such as hydroxyl radicals ( $\bullet\text{OH}$ ) and superoxide anions ( $\bullet\text{O}_2^-$ ), which mineralize organic pollutants into harmless products like CO<sub>2</sub> and H<sub>2</sub>O [9, 10]. Nevertheless, pristine TiO<sub>2</sub> suffers from intrinsic drawbacks, including rapid electron-hole recombination, nanoparticle agglomeration, and low absorption of visible light, all of which limit its photocatalytic efficiency [11].

To address these challenges, green synthesis approaches using plant extracts have gained increasing attention as eco-friendly alternatives to conventional chemical methods. Plant-derived metabolites such as flavonoids, phenolics, alkaloids, terpenoids, and saponins act as natural reducing and capping agents, enabling the synthesis of stable and well-dispersed TiO<sub>2</sub> nanoparticles while simultaneously enhancing their surface reactivity [12, 13]. In addition, bio-waste extracts are renewable and widely available, making them sustainable precursors for nanomaterial production [14].

Agro-waste materials such as pomelo peel (*Citrus maxima*), avocado peel (*Persea americana*), and petai pod (*Parkia speciosa*) are particularly rich in bioactive compounds that can support nanoparticle synthesis. Pomelo peel contains cellulose and flavonoids that can enhance adsorption capacity and photocatalytic stability [15, 16]. Avocado peel is abundant in phenolics, flavonoids,

and lipids, which contribute to nanoparticle stability, radical scavenging, and surface modification [17 - 19].

This study focuses on the green synthesis of TiO<sub>2</sub> nanoparticles using ethanol extracts of pomelo peel, avocado peel, and petai pod, followed by their application in the photocatalytic degradation of paracetamol. The approach aims to develop a sustainable and cost-effective photocatalyst that not only addresses pharmaceutical pollution but also valorizes agricultural waste, thereby supporting environmental sustainability and circular economy principles [20].

## EXPERIMENTAL

### Materials

Fresh avocado peels (*Persea americana*), petai pods (*Parkia speciosa*), and pomelo peels (*Citrus maxima*) were collected from a traditional market in South Jakarta, Indonesia. The biowaste samples were washed thoroughly with distilled water to remove impurities, air-dried at room temperature, cut into small pieces, and ground into fine powder before extraction. Titanium isopropoxide (TTIP, 97 %, Merck), ethanol p.a (Merck), and isopropanol p.a (Merck) were used as received without further purification. Distilled water was employed for all preparation and washing steps.

### Preparation of avocado, petai and pomelo peel extract

The peels were thoroughly washed with distilled water to remove adhering impurities, air-dried, and subsequently cut into small pieces. The dried materials were ground and sieved through a 60 mesh screen and subsequently macerated in ethanol at a peel powder to solvent ratio of 1:10 (w/v) for 72 h under ambient conditions. The mixtures were filtered, and the resulting extracts were collected as natural reducing and capping agents. To obtain the extract, the maceration filtrate was transferred into a round-bottom flask and concentrated using a laboratory-scale rotary vacuum evaporator. The resulting EEPa, EEPp, and EEPo extracts were preserved under refrigeration until further application [21].

### Synthesis of nanoparticle TiO<sub>2</sub> NPs

The synthesis procedure was carried out by mixing 10 mL of titanium isopropoxide (TTIP) with 25 mL of isopropanol. Subsequently, 10 mL of distilled water

was added, followed by the dropwise addition of 5 mL EEPA/EEPPi/EEPPo under continuous stirring at 500 rpm for 24 h at room temperature. The resulting gel was centrifuged and washed three times with distilled water. The obtained precipitate was dried in an oven at 80°C and calcined at 500°C for 2 h to yield the white TiO<sub>2</sub>-PA/PPi/PPo nanopowder [22].

### Characterization of TiO<sub>2</sub> NPS

The total phenolic content (TPC) and total flavonoid content (TFC) were determined using gallic acid and quercetin as standard references, respectively. TPC was measured to evaluate the role of phenolic compounds as natural reducing agents, while TFC was analysed to assess the contribution of flavonoids to nanoparticle stabilization and enhancement of photocatalytic activity. The crystalline phase and structural properties of the synthesized TiO<sub>2</sub> were examined by X-ray diffraction (XRD, PANalytical EMPYREAN) with Cu K $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ), and the average crystallite size was estimated using the Scherrer equation. Particle morphology, surface features, and size distribution were characterized using field emission scanning electron microscopy (FE-SEM) coupled with energy-dispersive X-ray spectroscopy (EDS, ThermoScientific Quatro S). The particle size distribution was further quantified using a Particle Size Analyzer (PSA, Malvern Panalytical). Functional groups and bonding structures were identified by Fourier transform infrared spectroscopy (FT-IR, Bruker Tensor II). The optical band gap was determined by UV-Vis diffuse reflectance spectroscopy (UV-Vis DRS), while the surface charge properties were evaluated through zeta potential analysis (Malvern Panalytical) [23].

### Photodegradation experiment

The photocatalytic performance of TiO<sub>2</sub> nanoparticles (NPs) - PA/PPi/PPo was assessed through the degradation of paracetamol. A suspension was prepared by adding the catalyst to a 10 ppm paracetamol

solution under continuous stirring until adsorption-desorption equilibrium was achieved. The mixture was then irradiated with UV C ( $2 \times 15 \text{ W}$ ), and aliquots were withdrawn every 30 min for up to 120 min. The catalyst was separated by centrifuging 4 mL of the collected solution at 10,000 rpm for 10 min. The residual concentration of paracetamol was quantified by UV-Vis spectrophotometer at 244 nm. The degradation efficiency was calculated using Eq. (1), where AY<sub>i</sub> and AY<sub>f</sub> denote the initial and final absorbance values, respectively [24].

$$\text{Removal (\%)} = \frac{\text{AY}_i - \text{AY}_f}{\text{AY}_i} \times 100 \quad (1)$$

## RESULTS AND DISCUSSION

### Total phenolic content (TPC) and total flavonoid content (TFC)

Biowaste represent valuable agro-industrial byproducts rich in bioactive compounds, particularly phenolics and flavonoids with strong antioxidant potential. The total phenolic content (TPC) (Table. 1) was highest in extract from pomelo (EEPPo) (8.3 mg GAE g<sup>-1</sup>), followed by petai (EEPPi) (7.0 mg GAE g<sup>-1</sup>) and avocado (EEPA) (1.6 mg GAE g<sup>-1</sup>), indicating EEPPo as a superior source of phenolics. In contrast, total flavonoid content (TFC) was markedly higher in EEPPi (424.7 mg QE g<sup>-1</sup>) compared to EEPPo (93.2 mg QE g<sup>-1</sup>) and EEPA (38.4 mg QE g<sup>-1</sup>). These results highlight the compositional variability among different biowastes, with petai pod being particularly rich in flavonoids, while pomelo peel predominates in phenolics, suggesting their distinct potential as natural antioxidants and functional additives in green nanomaterial synthesis [25].

### Fourier Transform Infrared Spectroscopy (FT-IR)

The FT-IR spectra of ethanol extracts from pomelo (EEPPo), petai (EEPPi), and avocado (EEPA) peels revealed characteristic functional groups associated with phenolics, flavonoids, and carbonyl compounds, which may act as reducing and stabilizing agents during TiO<sub>2</sub>

Table 1. TPC and TFC extract biowaste.

Parameter	EEPA	EEPPi	EEPPo
Total Phenolic Content (mg GAE/g)	1.6	7.0	8.3
Total Flavonoid Content (mg QE/g)	38.4	424.7	93.2

nanoparticle formation. Broad absorption bands (Fig. 1a) at  $3330\text{ cm}^{-1}$  assigned to O–H stretching of phenolic groups and adsorbed water [26, 27], while peaks at  $2973 - 2855\text{ cm}^{-1}$  indicate C–H stretching of aliphatic chains [28]. Peaks near  $1650\text{ cm}^{-1}$  assigned to carbonyl groups (C=O) stretching vibrations of flavonoids and amino acids [24, 29], The peak at  $1385\text{ cm}^{-1}$  shows that the C–H bending bond is an aldehyde [30], and peaks at  $1200 - 900\text{ cm}^{-1}$  are attributed to C–O–C/C–OH stretching typical of polysaccharides and flavonoids. Among the extracts, EEPPo exhibited higher intensities at  $3332$  and  $1380\text{ cm}^{-1}$ , suggesting a higher concentration of bioactive metabolites compared to EEPA, consistent with Beer-Lambert's principle [22].

After calcination (Fig. 1b),  $\text{TiO}_2$ -extract composites displayed dominant absorption bands at  $438 - 506\text{ cm}^{-1}$ , which are characteristic of Ti–O and Ti–O–Ti stretching in the anatase phase [21].  $\text{TiO}_2$ -EEPPo exhibited more pronounced peaks ( $440.9$ ,  $465.3$ , and  $506\text{ cm}^{-1}$ ), indicating stronger surface modification compared to  $\text{TiO}_2$ -EEPA and  $\text{TiO}_2$ -EEPPi. The disappearance of –OH and C=O peaks at higher wavenumbers suggests the decomposition or chemical bonding of phytochemicals during calcination. These interactions, particularly with phenolic hydroxyl groups, may act as surface capping agents that limit crystal overgrowth and reduce particle agglomeration, thereby contributing to the physicochemical stabilization of  $\text{TiO}_2$  nanoparticles [31]. Such stabilization is important to ensure uniform particle size distribution, enhanced dispersion in aqueous media, and improved photocatalytic efficiency.

### Ultraviolet-Visible Diffuse Reflectance Spectroscopy (UV-DRS)

UV-DRS analysis revealed slight variations in the band gap energies of  $\text{TiO}_2$  modified with biowaste extracts:  $\text{TiO}_2$ -EEPA ( $3.25\text{ eV}$ ),  $\text{TiO}_2$ -EEPPi ( $3.29\text{ eV}$ ), and  $\text{TiO}_2$ -EEPPo ( $3.18\text{ eV}$ ). The narrower band gap of  $\text{TiO}_2$ -EEPPo (Fig. 2) suggests enhanced photocatalytic potential, as lower excitation energy facilitates electron transfer from the valence to the conduction band, thereby promoting visible-light absorption and efficient electron-hole ( $e^- - h^+$ ) generation for reactive oxygen species formation ( $\bullet\text{OH}$ ,  $\bullet\text{O}_2^-$ ) [32, 33]. This result is consistent with the phytochemical composition shown in Table 1, where EEPPo exhibited the highest total phenolic and flavonoid contents comparing to EEPA and EEPPi. The

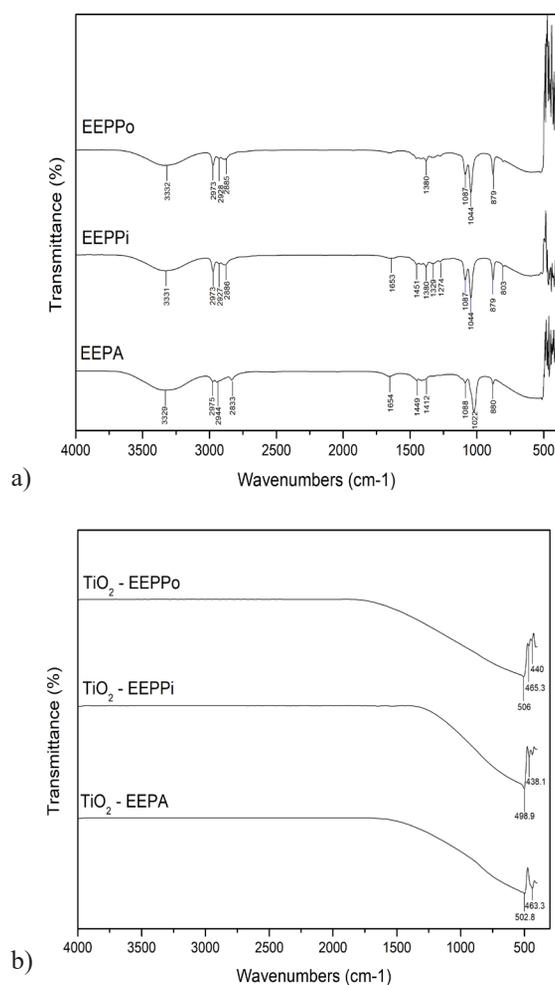


Fig. 1. (a) FT-IR spectra of biowaste extracts, (b) FT-IR spectra of  $\text{TiO}_2$ -extract after calcination at  $500^\circ\text{C}$ .

abundant bioactive compounds may contribute to surface functionalization or non-metal doping (C, N, S), which creates additional energy states within the  $\text{TiO}_2$  band structure, thus narrowing the band gap. In parallel, the strong interaction between phenolic hydroxyl groups and  $\text{TiO}_2$  surface can limit crystal overgrowth, leading to smaller particle sizes. Therefore, the higher TPC and TFC values of EEPPo are in good agreement with the reduced  $E_g$  and refined crystallite sizes of  $\text{TiO}_2$ , and these synergistic effects ultimately enhance the photocatalytic performance [34, 35].

### XRD patterns

XRD patterns confirmed that all  $\text{TiO}_2$  samples, biowaste-modified, were dominated by the anatase phase with a minor brookite peak influenced by

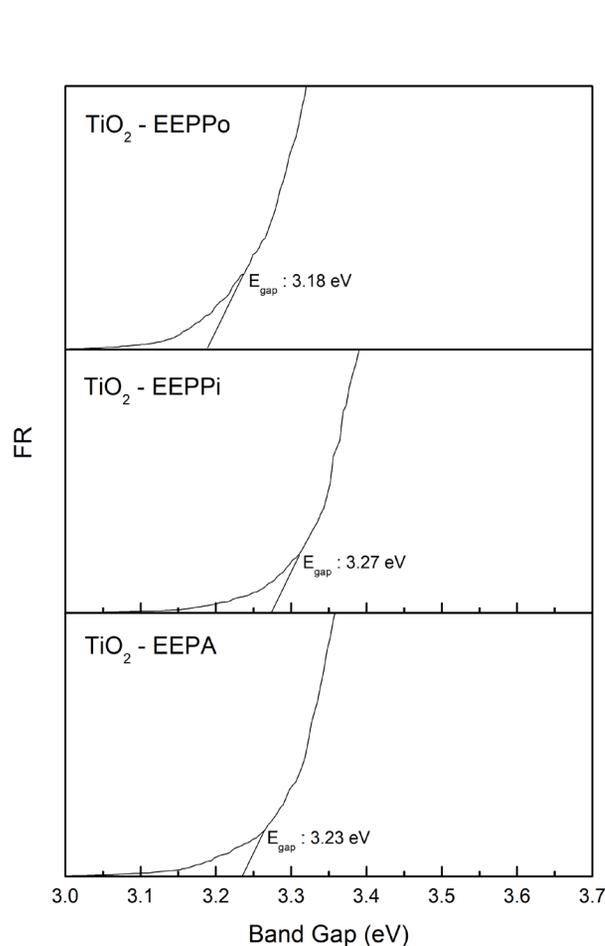


Fig. 2. UV-DRS spectra of TiO<sub>2</sub>-biowaste extracts.

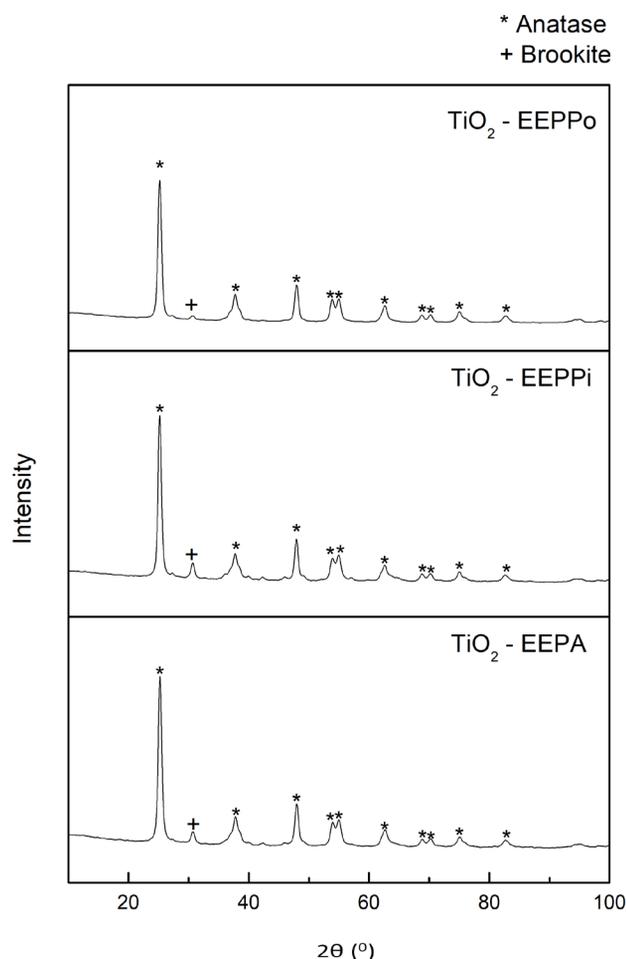


Fig. 3. XRD patterns of TiO<sub>2</sub>-biowaste extracts.

synthesis conditions. The crystallite sizes were 13.5 nm (TiO<sub>2</sub>-EEPA), 9.9 nm (TiO<sub>2</sub>-EEPPi), and 13.8 nm (TiO<sub>2</sub>-EEPPo), indicating that biowaste modification significantly reduced crystallinity compared (Fig. 3). The lower crystallinity, particularly in TiO<sub>2</sub>-EEPPi, is attributed to lattice defects induced by organic compounds, which increase e<sup>-</sup>/h<sup>+</sup> recombination sites and may alter the band gap through the quantum confinement effect, thereby lowering photocatalytic efficiency. These findings demonstrate that biowaste extracts strongly influence the structural and photocatalytic properties of TiO<sub>2</sub> [36 - 38].

#### FE-SEM

FE-SEM characterization showed that all TiO<sub>2</sub> samples exhibited porous morphology with particle agglomeration yet remained within the nanoscale (< 100 nm). Image-J analysis revealed particle sizes of

23.3 nm (TiO<sub>2</sub>-EEPA), 28.8 nm (TiO<sub>2</sub>-EEPPi), and 25 nm (TiO<sub>2</sub>-EEPPo) (Fig. 4). The reduced particle size in TiO<sub>2</sub>-EEPA suggests that avocado peel extract inhibited crystal growth during calcination, potentially enhancing surface area and photocatalytic activity. However, excessive agglomeration, particularly in TiO<sub>2</sub>-EEPPi, may hinder light diffusion and charge transfer, thereby lowering efficiency. These results indicate that biowaste modification significantly affects particle size, aggregation, and photocatalytic behavior of TiO<sub>2</sub> [31, 39, 40].

#### Particle Size Analyzer (PSA)

Particle Size Analyzer (PSA) results (Fig. 5) showed average particle sizes of 91.9 nm (TiO<sub>2</sub>-EEPA), 94.64 nm (TiO<sub>2</sub>-EEPPo), and 92.33 nm (TiO<sub>2</sub>-EEPPi). Smaller particle size enhances surface area, which is beneficial for photocatalytic reactivity [41].

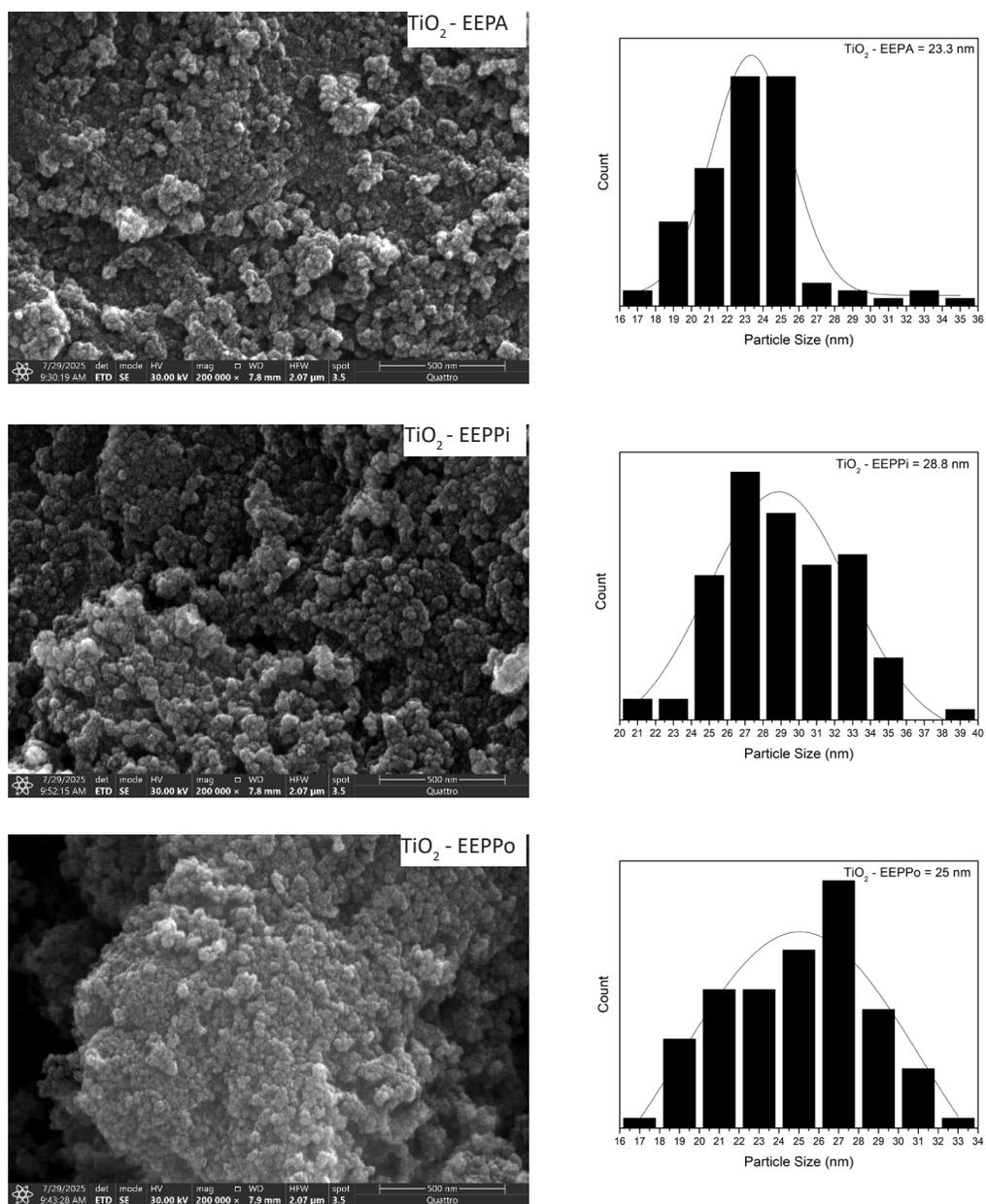


Fig. 4. FE-SEM image of  $\text{TiO}_2$ -biowaste extracts.

Zeta potential analysis (Fig. 6) revealed values of  $-17.11$  mV ( $\text{TiO}_2$ -EEPA),  $-21.88$  mV ( $\text{TiO}_2$ -EEPPi), and  $-21.45$  mV ( $\text{TiO}_2$ -EEPPo), suggesting that all samples exhibited sufficient colloidal stability in aqueous suspension. More negative zeta potential values reflect stronger electrostatic repulsion, reducing agglomeration, while lower values indicate higher aggregation tendency [42]. These results highlight that both particle size reduction and colloidal stability strongly influence the photocatalytic efficiency of  $\text{TiO}_2$  [43, 44].

#### Adsorption paracetamol of extract biowaste $\text{TiO}_2$ - NPS

The adsorption (Fig. 7) shows that biowaste-modified  $\text{TiO}_2$  (EEPA, EEPPi, EEPPo) displayed initial positive adsorption (0.5 - 0.8 % at 30 min) followed by a gradual decrease. The enhanced adsorption in modified samples is attributed to the presence of surface functional groups from biowaste extracts (-OH, -COOH, phenolic) that provide additional binding sites through electrostatic interactions and hydrogen bonding [41]. Reduced

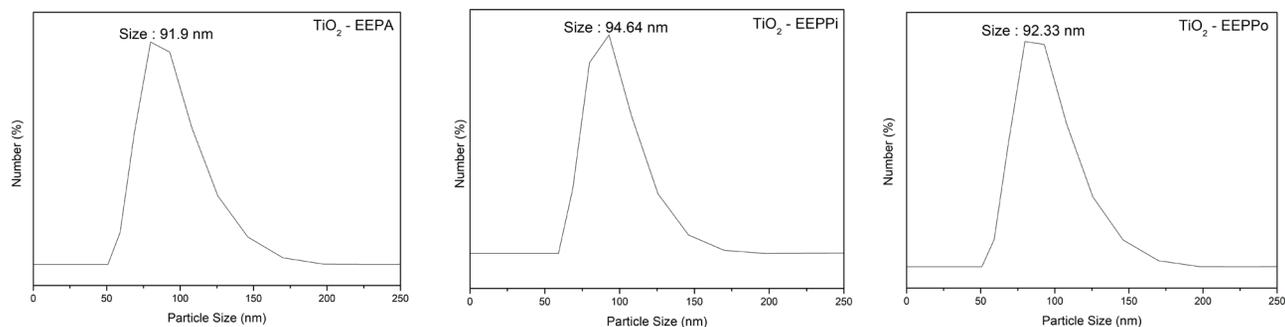


Fig. 5. Size of  $\text{TiO}_2$ -biowaste extracts at aqueous colloidal states generated from Particle Size Analyzer.

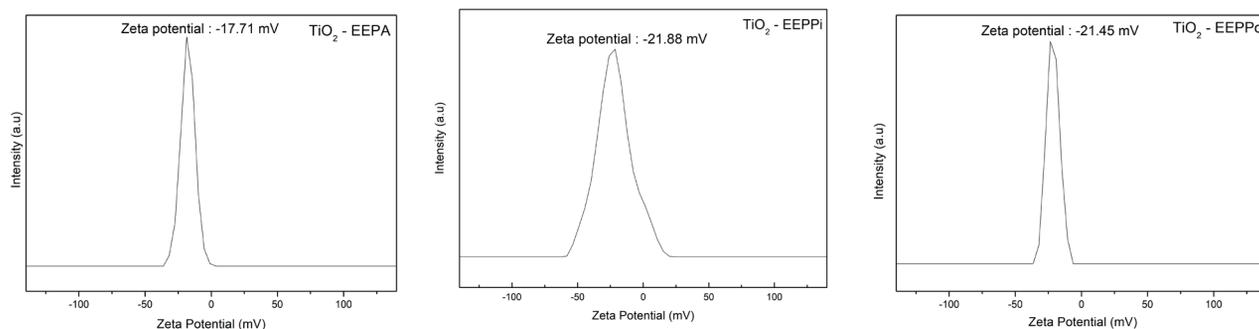


Fig. 6. Zeta potential of  $\text{TiO}_2$ -biowaste extracts at aqueous colloidal states generated from Particle Size Analyzer.

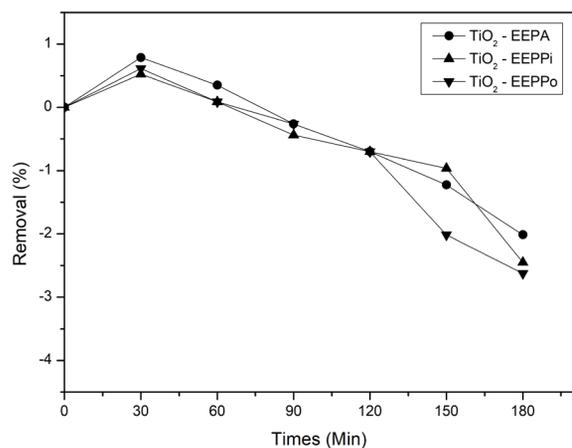


Fig. 7. Adsorption paracetamol using  $\text{TiO}_2$ -biowaste extracts.

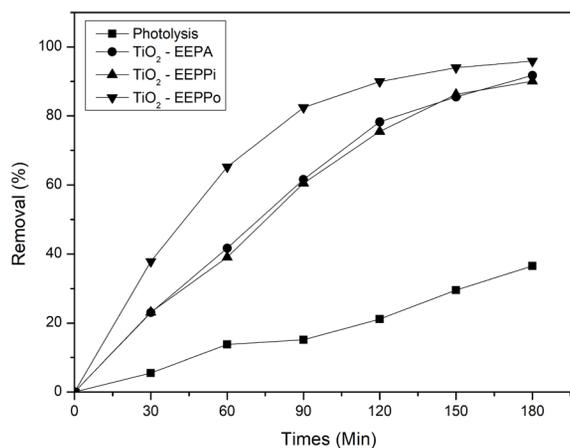


Fig. 8. Photocatalytic paracetamol using  $\text{TiO}_2$ -biowaste extracts.

particle size and increased surface heterogeneity, as confirmed by FE-SEM and PSA, also contribute to the improved adsorption [42]. However, the decline over time suggests site saturation and partial desorption of weakly bound molecules [45].

#### Photocatalytic activity of extract biowaste $\text{TiO}_2$ - NPS

The photodegradation (Fig. 8) shows that photolysis

alone achieved only 36.52 % degradation after 180 min, confirming the limited effect of UV-C without a photocatalyst. Biowaste extract-modified catalysts ( $\text{TiO}_2$ -EEPA,  $\text{TiO}_2$ -EEPPi,  $\text{TiO}_2$ -EEPPo) also exhibited high efficiencies (> 90 %), with  $\text{TiO}_2$ -EEPPo (95.89 %) performing the highest photocatalytic activity. This improvement can be ascribed to phenolic compounds and surface functional groups that enhanced charge

transfer and reduced recombination [32]. However, TiO<sub>2</sub>-EEPA and TiO<sub>2</sub>-EEPPi displayed slower initial rates, likely due to particle agglomeration and surface heterogeneity that limited light absorption [42]. Biowaste extract-assisted TiO<sub>2</sub> improved photocatalytic degradation by introducing additional adsorption sites and prolonging electron-hole lifetimes [45], though the efficiency strongly depends on the chemical nature of the biowaste extracts.

## CONCLUSIONS

TiO<sub>2</sub> was successfully synthesized using avocado peel, petai pod, and pomelo peel ethanol extracts as capping and stabilizing agents. Characterization confirmed the anatase structure with nanoscale size, good colloidal stability, and porous morphology. Photocatalytic tests showed high efficiency, with TiO<sub>2</sub>-EEPPo achieving the best performance at 95.89 % paracetamol degradation, highlighting bio-waste as a sustainable precursor for green photocatalyst development.

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## Authors' contributions

*N.C.: Experimental work, Writing original draft, Formal analysis, Design of the research; K.K.: Conceptualization, Methodology, Validation, Formal analysis, Writing - review & Editing, Design of the research, Project management and funding acquisition; J.S.: Writing - review & Editing and Validation; A.H., R.AP, M.A.M, and T.O: Formal analysis and Validation.*

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