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Phyto mediated of SnO_2 nanoparticles using *Pandanus amaryllifolius* extract for photocatalytic degradation of methylene blue

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ABSTRACT

The development of environmentally friendly photocatalysts is crucial for the remediation of persistent textile dyes. This study reports a novel and simple green synthesis of ultrafine tin oxide (SnO_2) nanoparticles by utilising Pandanus amaryllifolius leaf extract as a bioreductor and dual function capping agent, thereby eliminating the need for toxic chemicals and high energy calcination. Comprehensive characterisation confirmed the formation of a pure tetragonal rutile phase with ultrafine crystallite sizes of 4 – 5 nm. Importantly, surface interactions mediated by phytochemicals intrinsically engineer the optical band gap to 3.50 eV, much narrower than bulk SnO_2 through the formation of defect states and mid-gap states, enhancing responsiveness to visible light. The photocatalytic efficacy was evaluated under challenging conditions using a high initial concentration of Methylene Blue (MB) (100 ppm). Green synthesised SnO_2 nanoparticles exhibited robust degradation performance, achieving a removal rate of 66.5% within 120 minutes. Kinetic analysis reveals a characteristic two stage pseudo first order behaviour, indicating a transition from the initial induction phase to the formation of reactive oxygen species at steady state. This research provides new insights into the engineering of semiconductor defects mediated by phytochemicals and offers highly scalable sustainable nanomaterial strategies that align with green chemistry principles for industrial wastewater treatment.

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1. INTRODUCTION

The increase in the release of persistent organic pollutants (POPs), particularly synthetic textile dyes like Methylene Blue (MB), poses a serious threat to aquatic ecosystems and human health due to their high toxicity, striking colour properties, and resistance to biodegradation [1, 2]. Conventional wastewater treatment often fails to completely mineralise these resistant compounds. Therefore, advanced oxidation processes (AOPs), particularly heterogeneous photocatalysis, have emerged as a highly efficient remediation strategy capable of degrading organic pollutants into harmless products [3, 4]. However, conventional chemical synthesis for photocatalysts heavily relies on toxic reducing agents, hazardous solvents, and high energy processes, which directly contradict the principles of green chemistry and the circular economy [5, 6].

SnO_2 is a highly promising n-type semiconductor photocatalyst due to its high electron mobility, excellent chemical stability, strong oxidation potential, and low cost [7, 8]. To address the environmental drawbacks of traditional fabrication, green synthesis utilising plant extracts as natural reducing and stabilising (capping) agents has garnered significant attention. Various botanical sources, such as *Tectona grandis* [9, 10], *Camellia sinensis* [11], and *Hibiscus sabdariffa* [8], have been successfully used to synthesise environmentally friendly SnO_2 nanoparticles for dye degradation. This phytochemically mediated approach not only eliminates the use of toxic chemicals but also provides surface functionalisation that can enhance catalytic activity and stability [12].

Despite these advancements, pure SnO_2 has an inherent drawback in the form of a wide bandgap (~3.6 eV), which limits its activation primarily to UV light and results in rapid electron-hole recombination [7, 13]. Although structural modification and doping are common solutions, these methods often involve complex and energy-intensive stages [14, 15]. There is a critical research gap in utilising specific botanical extracts that have not been optimally explored to intrinsically modify the band gap and morphology of SnO_2 at the early stages of green synthesis.

Pandanus amaryllifolius is a highly abundant and low-cost botanical resource, rich in unique phytochemicals such as flavonoids, alkaloids, phenolics, and 2-acetyl-1-pyrroline. Although Buniyamin et al. recently utilised pandan extract to synthesise SnO_2 for optoelectronic applications [5], its potential as a bioreductor and dual function stabilising agent for engineering oxygen vacancies and specifically narrowing the SnO_2 bandgap for photocatalytic wastewater remediation has not yet been fully explored. The specific molecular interactions between the unique phytochemicals of pandan and Sn precursors offer a clear advantage in creating surface defects that enhance visible light absorption without the need for post-synthesis chemical doping.

We hypothesise that the complex phytochemical profile of *Pandanus amaryllifolius* extract will effectively reduce tin precursors while stabilising the newly formed nanoparticles, thereby inducing oxygen vacancies and surface defects that significantly narrow the optical bandgap of SnO_2. As a result, the structural tuning mediated by these phytochemicals will enhance charge separation and promote the solar powered photocatalytic degradation of MB. Therefore, the specific objectives of this research are to (1) synthesise SnO_2 nanoparticles through a simple green route using *Pandanus amaryllifolius* leaf extract; (2) comprehensively characterise the structural, morphological, and optical properties of the biosynthesised nanoparticles; (3) evaluate their photocatalytic efficiency in degrading MB under simulated solar irradiation, including kinetic modelling; and (4) assess the catalyst's recyclability and propose a plausible degradation mechanism.

The significance of this research has two aspects. Scientifically, this research clarifies the structure-property relationship between *Pandanus amaryllifolius* phytochemicals and SnO_2 crystal growth, providing new insights into defect engineering through green chemistry. Practically, this research offers scalable, cost-effective, and environmentally friendly nanomaterials for industrial wastewater treatment, which directly aligns with sustainable development goals (SDGs) and the 5R principles (redesign-reduction-recovery-recycle-reuse) in the circular economy [6].

2. RESEARCH METHOD

Fresh *Pandanus amaryllifolius* leaves are thoroughly washed with running tap water to remove dust and dirt, followed by rinsing with deionised (DI) water. The leaves were shade-dried at room temperature to preserve thermolabile phytochemical compounds, then ground into a fine powder. For the preparation of the extract, 30 g of leaf powder is mixed with 300 mL of DI water and heated at 60°C for 60 minutes under continuous magnetic stirring. The resulting mixture is filtered using Whatman No. 1 filter paper, and the clear filtrate obtained is stored at 4 °C for subsequent use as a reducing and stabilising agent.

2.1. Green Synthesis of SnO_2 Nanoparticles

Dihydrate tin (II) chloride ($SnCl_2·2H_2O$, 99.9%) is used as a precursor without further purification. In a typical synthesis procedure, a 0.1 M aqueous solution of $SnCl_2·2H_2O$ is added dropwise to an extract of *Pandanus amaryllifolius* leaves at a volume ratio of 1:2. The pH of the mixture is adjusted to 9 using a solution [mention the substance, e.g., 1 M NaOH to facilitate the precipitation and stabilisation of SnO_2 perfectly. The mixture was heated to 80°C for 2 hours under continuous stirring. The formed precipitate was collected by centrifugation at 8000 rpm for 15 minutes, repeatedly washed with DI water and absolute ethanol to remove unbound organic residues, and dried in a hot air oven at 200°C for 2 hours. The dried powder was slowly ground using an agate mortar to obtain the final green synthesised SnO_2 nanoparticles.

2.2. Characterisation Techniques

The crystal structure and phase purity of the synthesised SnO_2 nanoparticles were analysed using X-ray diffraction (XRD, Shimadzu XRD-7000) with Cu Kα radiation ($\lambda = 1.5406$ Å) in the 2θ angle range of 20° to 80°. The optical properties and bandgap energy were evaluated using a UV-Vis diffuse reflectance spectrophotometer (UV-Vis DRS, Shimadzu UV-2600). Functional groups on the surface and the interaction between phytochemicals and the SnO_2 surface were identified using Fourier Transform Infrared Spectroscopy (FTIR, Thermo Fisher Nicolet iS5) in the wavenumber range of 4000-400 cm^{-1}. Surface morphology and elemental composition were examined using Scanning Electron Microscopy equipped with Energy Dispersive X-ray spectroscopy (SEM-EDX, JEOL JSM-6510A).

2.3. Photocatalytic Activity

The photocatalytic performance of SnO_2 nanoparticles is evaluated through the degradation of MB under simulated solar irradiation using a 300 W UV lamp. In a typical experiment, 50 mg of SnO_2 nanoparticles were dispersed into 100 mL of an aqueous MB solution with an initial concentration of 100 ppm. Before the irradiation process, the suspension is magnetically stirred in the dark for 30 minutes to establish the adsorption-desorption equilibrium. At certain time intervals, an aliquot of 3 mL is taken, centrifuged to separate the catalyst particles, and the residual MB concentration is analysed using a UV-Vis spectrophotometer at its maximum absorption wavelength (λmax = 664 nm). Degradation efficiency is calculated using the equation Degradation (%) = $[(C_0 - Ct) / C_0] \times 100\%$, where C_0 and Ct are the initial concentration and the concentration of MB at a given time (t), respectively.

3. RESULTS AND DISCUSSIONS

3.1. Optical Properties

The UV-Vis spectrum of *Pandanus amaryllifolius* leaf extract (Figure 1a) displays a strong absorption band at 280 nm, confirming the dominance of phenolic compounds and flavonoids that act as natural reducing and stabilising agents [16]. SnO_2 nanoparticles exhibit a dominant absorption band at 348 nm, corresponding to the characteristic band to band electronic transition of SnO_2 [1].

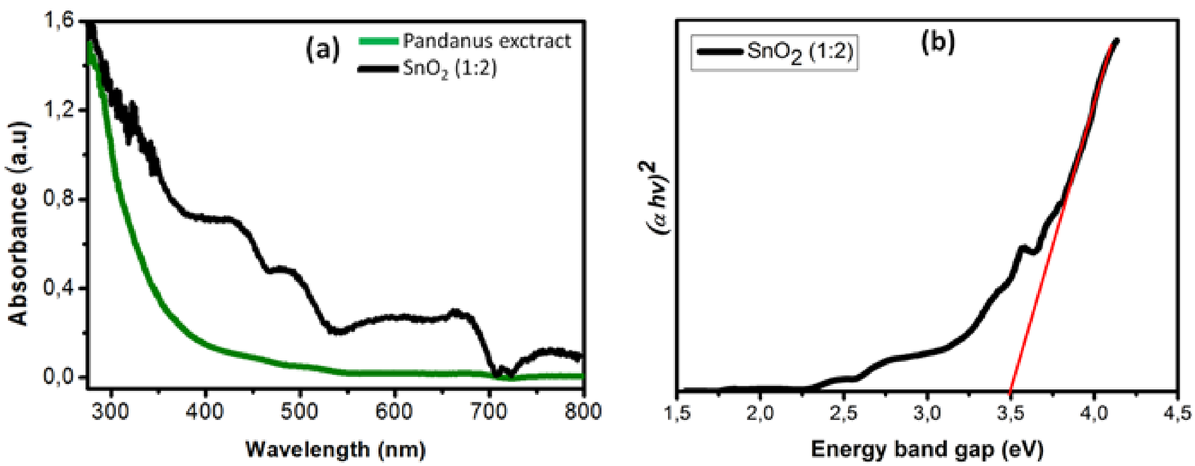

Figure 1. (a) UV-Vis spectroscopy of *Pandanus amaryllifolius* leaf extract and SnO_2 nanoparticles, (b) energy band gap of SnO_2 nanoparticles.

Tauc analysis (Figure 1b) reveals an optical bandgap value of 3.50 eV. This value is narrower compared to bulk SnO_2 (3.60 eV). Unlike the quantum confinement effect, which generally widens the bandgap at ultrafine sizes, this narrowing strongly indicates the formation of defect states (such as oxygen vacancies) and electronic interactions between the SnO_2 surface and the phytochemical functional groups of *Pandanus amaryllifolius* that create mid-gap states [12, 17].

Interestingly, this 3.50 eV band gap is narrower compared to green synthesised SnO_2 using *Camellia sinensis* (3.79-4.02 eV) [11], and is highly competitive compared to pandan extract SnO_2 which requires 2 hours of calcination at 600 °C to achieve 3.40 eV [5]. This proves that the phytochemicals in pandan are intrinsically capable of engineering the band gap of SnO_2 without requiring high energy treatment.

3.2. Structural and Morphological Analysis

The X-ray diffraction pattern (Figure 2) confirms the tetragonal rutile crystal phase of SnO_2 (JCPDS No. 41-1445) without the presence of secondary phase peaks, indicating high phase purity [10]. The crystallite size calculated using the Scherrer equation is in the range of 4-5 nm. This ultrafine size is comparable to SnO_2 quantum dots synthesised using Ulva sp. algae extract (<5 nm) [16] and smaller than green SnO_2 from kiwi extract (5-10 nm) [18]. This indicates that biomolecules from pandan extract are very effective as a capping agent in inhibiting crystal growth.

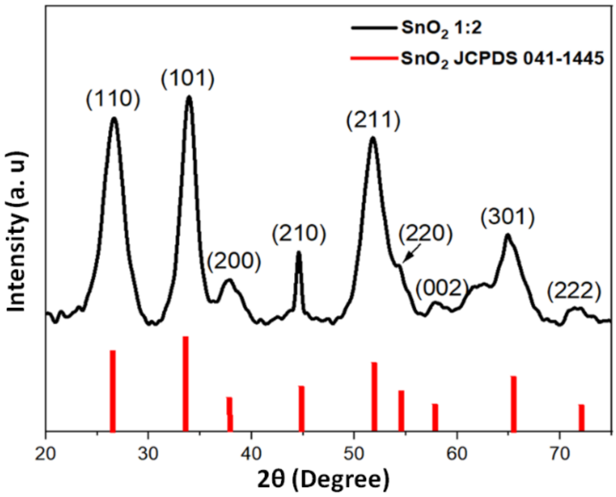

Figure 2. XRD pattern of SnO_2 nanoparticles.

SEM analysis (Figure 3a) shows that the particles tend to agglomerate with quasi-spherical to irregular morphology, with aggregate sizes reaching hundreds of nanometres (Figure 3b). This agglomeration phenomenon commonly occurs in green synthesis due to the presence of an organic layer from plant extracts that envelops the particles and high surface energy [9, 11]. It is important to note that the photocatalytic performance highly depends on the size of the primary crystallites (4-5 nm from XRD) and their active surface area, rather than the size of the macroscopic aggregates observed in SEM [19].

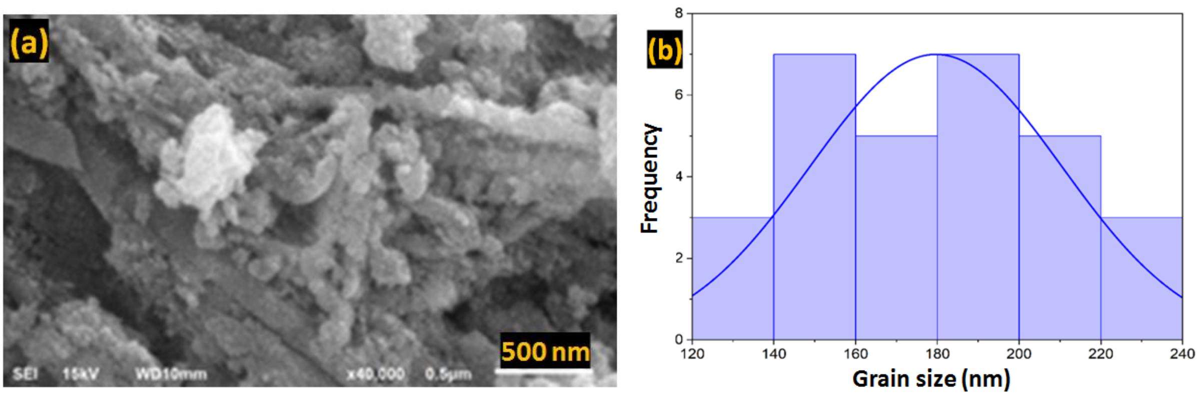

Figure 3. (a) SEM image and (b) grain size distribution of SnO_2 nanoparticles.

3.3. Elemental Composition

The EDX spectrum (Figure 4) confirms the presence of elemental peaks of tin (Sn) and oxygen (O) as the main components, with a mass percentage of Sn at 82.50%. The presence of carbon (C) and other minor elements confirms the biomolecular residues from the *Pandanus amaryllifolius* extract bound to the nanoparticle surface, which act as a natural capping agent [20]. Quantitative analysis of oxygen through EDX is not used to directly claim the absence of oxygen due to the detector's accuracy limitations for light elements; evidence of oxygen absence has been comprehensively validated through band gap narrowing in the UV-Vis section.

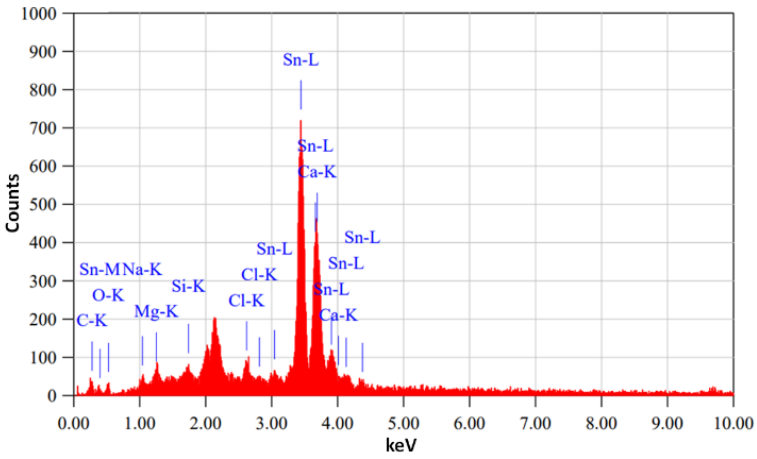

Figure 4. EDX spectrum of SnO_2 nanoparticles.

3.4. FTIR Analysis

The FTIR spectrum (Figure 5) provides direct evidence of the role of *Pandanus amaryllifolius* phytochemicals. The pandan extract (Figure 5a) shows a broad band at 3337 cm^{-1} (O-H stretch), as well as peaks at 1637 and 1996 cm^{-1} (C=C and C=O stretches from flavonoids/phenols). In the SnO_2 nanoparticles spectrum (Figure 5b), the presence of bands at 3400 cm^{-1} (O-H), 2850 cm^{-1} (C-H), 1780 cm^{-1} (C=O), and 1050 cm^{-1} (C-O) indicates that the hydroxyl and carbonyl functional groups from the *Pandanus amaryllifolius* extract have successfully electrostatically bound to the SnO_2 surface [8, 12]. The characteristic peak at 572 cm^{-1} confirms the Sn-O-Sn stretch vibration [18]. The shift and preservation of these organic functional groups prove that the phytochemistry of pandan not only reduces Sn^{4+} ions but also forms a capping layer that prevents agglomeration and stabilises surface defects.

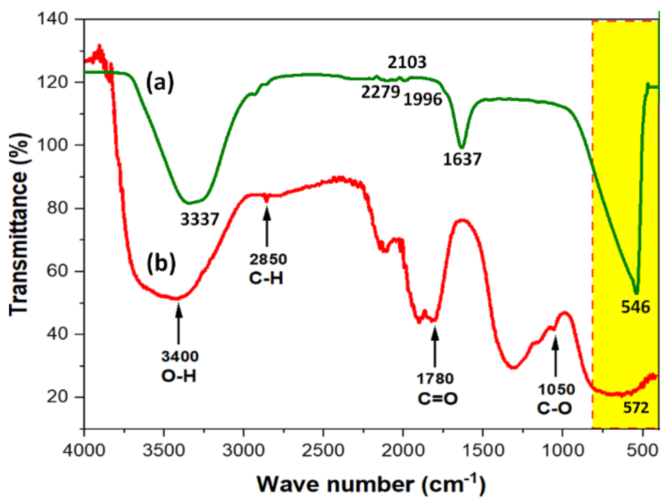

Figure 5. FTIR spectrum of (a) *Pandanus amaryllifolius* extract and (b) SnO_2 nanoparticles.

3.5. Photocatalytic evaluation

Photocatalytic evaluation was conducted under challenging conditions, namely a high initial concentration of MB (100 ppm). As shown in Figure 6a, SnO_2 nanoparticles achieved a degradation of 66.50% in 120 minutes. Although this degradation percentage appears lower compared to the literature using lower concentrations, the absolute degradation at a concentration of 100 ppm demonstrates a very robust catalytic capacity. The decrease in degradation percentage at high concentrations is thermodynamically caused by the inner filter effect (light penetration hindered by dense MB molecules) and saturation of active sites on the catalyst surface [4].

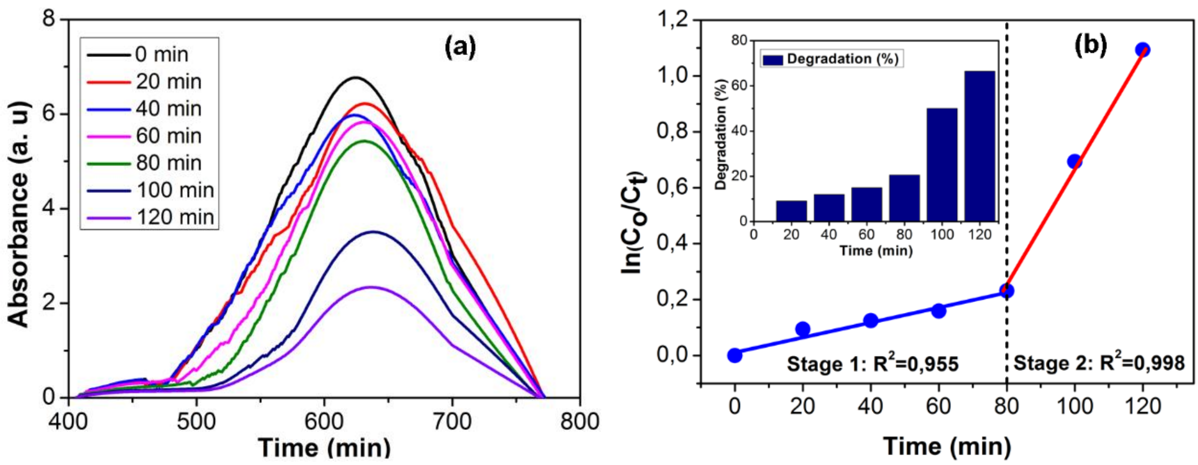

Figure 6. (a) Photocatalytic degradation of MB, (b) kinetic plot of ln(C_0/Ct) of MB.

Kinetic analysis (Figure 6b) reveals a two-stage pseudo-first-order kinetic behaviour. The first stage (0-80 minutes) with a rate constant (k) of 0.002683 min^{-1} (R2 = 0.955) represents the induction phase, where reactive oxygen species (ROS) begin to form and fill the oxygen vacancies as electron trapping centres. The second stage (80-120 minutes) shows a significant acceleration in the reaction rate with k = 0.02139 min^{-1} (R^2 = 0.998). This acceleration indicates that after the active sites are saturated, the intermediates resulting from the first stage of degradation act as photosensitisers, or the formation of ROS (such as O_2^- and OH) has reached a steady state that massively degrades MB [1,19]. These findings confirm that SnO_2 nanoparticles possess superior kinetic stability even under high pollutant loads, making them a promising candidate for large scale industrial wastewater remediation.

4. CONCLUSIONS

This research has successfully demonstrated the green synthesis of SnO_2 nanoparticles that are simple and environmentally friendly using Pandanus amaryllifolius leaf extract as a reducing agent and natural capping agent. Structural and optical analyses confirm the formation of a pure tetragonal rutile phase with ultrafine crystallite sizes (4-5 nm). Significantly, the electronic interaction between the phytochemical functional groups of pandan and the surface of SnO_2 nanoparticles has been shown to effectively engineer the bandgap to 3.50 eV, narrower than bulk SnO_2, through the formation of defect states and mid-gap states, without the need for high-temperature calcination treatment or conventional chemical doping.

In photocatalytic applications, these green SnO_2 nanoparticles demonstrate very robust performance even under challenging conditions, degrading 66.50% of MB at a high initial concentration (100 ppm) within 120 minutes. The observed pseudo first order two stage kinetic behaviour underscores the superior catalytic stability, where the formation of reactive oxygen species (ROS) reaches a steady state capable of overcoming kinetic limitations under high pollutant loads. These findings not only provide new insights into bandgap engineering mediated by phytochemicals but also offer sustainable strategies for fabricating high performance photocatalysts in line with green chemistry principles.

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