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Research Article

Biosynthesis of Tin(IV) Oxide Nanoparticles (SnO_2 NPs) via *Chromolaena Odorata* Leaves: The Influence of Heat on the Extraction Procedure

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ABSTRACT

In this research, tin(iv) oxide nanoparticles (SnO_2 NPs) using leaf extract of *Chromolaena Odorata* was successfully synthesized. Although the traditional extraction method typically requires heating for collecting the extract, this study performed the extraction utilizing free heat. Subsequently, a comparative analysis was performed with the boiled version to recognize any distinctions in the formation of SnO_2 NPs. Leaves of *C. odorata* contain bioactive compounds, particularly polyphenolic flavonoids, which potentially serve as effective agents in green synthesis, acting as both reducing and capping agents for Sn^{4+}. The synthesis was conducted at ambient temperature, followed by calcination at 700°C. FESEM images revealed that the morphologies of SnO_2 NPs in both samples were uniform and spherical. The presence of O and Sn elements was further confirmed by EDX analysis, with an atomic composition of approximately 76% and 23%, respectively. XRD obtained the most prominent peaks of SnO_2 NPs which are (110), (101), and (211) with fair sharpness for both samples with tetragonal structure. Furthermore, the FTIR spectrum affirmed the presence of pertinent functional groups through the vibration and stretching pattern of SnO_2 and Sn-OH groups. Based on these findings, the heat-free treatment of *C. odorata* extract proves to be comparable to the boiled version in mediating biosynthesis. Nevertheless, the preference is towards the traditional process, as the use of heat enhances the extraction process by increasing the abundance of bioactive compounds without undergoing degradation. Additionally, bioactive compounds aid in stabilizing the structure of SnO_2 NPs and preventing agglomeration.

Keywords: Tin oxide nanoparticles, biosynthesis, *Chromolaena odorata*, extraction

1. INTRODUCTION

Oxide semiconductors (Al_2O_3, ZnO, CuO, TiO_2 and SnO_2) have drawn attention from scientists worldwide due to their economic simplicity in processing techniques and are less toxic. Among the oxide semiconductors, SnO_2 has a wide range of applications such as gas sensing (Basu et al., 2011), catalyst (Manjunathan et al., 2018), solar cells (Zainudin et al., 2019), lithium-ion batteries (Zoller et al., 2019), medical applications (Buniyamin et al., 2022) etc. Tin(iv) oxide nanoparticles (SnO_2 NPs) are excellent n-type semiconductors with an energy band gap of 3.6 eV and this nanomaterial possesses the efficiency of these applications due to the improved surface area (Jeevanandam et al., 2018). SnO_2 NPs have reportedly been prepared using several techniques such as electrodeposition (Li et al., 2009), sol-gel (Zhang & Gao, 2004), laser ablation (Liu et al., 2003), microwave heating (Nehru & Sanjeeviraja, 2014), hydrothermal (Akhir et al., 2016), co-precipitation (Tazikeh et al., 2014) and ball milling (Tan et al., 2004).

The industrial applications of SnO_2 NPs are restricted because it depends on the use of high cost and toxic chemicals which are commonly used in these techniques. Hence, it is very crucial to offer synthetic methods using green and economic reagents which are more environmentally friendly and cost effective. Furthermore, the green synthesis of metal oxide nanoparticles using plant extract has been of much interest in recent studies and future research (Buniyamin et al., 2023; Shankar et al., 2016). There are articles reporting studies of green synthesis or biosynthesis of SnO_2 NPs using plants, for example, *Aspalathus linearis* (Diallo et al., 2016), *Brassica oleracea* (Osuntokun et al., 2017), *Ficus carica* (Hu, 2015), *Calotropis gigantean* (Bhosale et al., 2018), *Persia Americana* (Elango & Roopan, 2016), *Daphne mucronata* (Haq et al., 2020), *Aquilaria malaccensis*, *Pandanus amaryllifolius* (Buniyamin et al., 2022), *Pruni spinosae* (Dobrucka et al., 2018), *Ziziphus jujube* (Honarmand et al., 2019), *Plectranthus amboinicus* (Fu et al., 2015), and *Cleistanthus Collinus* (Kamaraj et al., 2014).

These eco-friendly procedures have significant advantages such that it can be easily handled, abundantly available, inexpensive, reproducible, and only using aqueous media to replace toxic solvents (Buniyamin et al., 2023; Zulpahmi, 2023). *Chromolaena odorata* is a medicinal plant that is commonly grown as a perennial shrub in Malaysia. There were several phytochemical studies performed on the determination of bioactive compounds of *C. odorata* especially in their leaves which revealed the presence of bioactive compounds such as flavonoid, terpenoids and alkaloids that is believed to have good potential as reducing and capping agent towards the biosynthesis of SnO_2 NPs (Omokhua et al., 2016; Akinmoladun et al., 2007; Buniyamin et al., 2023). In this case, the main skeleton of the quercetin-type flavonoid (Figure 1) is recognized as one of the best candidates to carry the task based on the position of the hydroxyl group attached to the carbon aromatic ring that facilitates the capping action of the metallic Sn^{4+} (Gawade et al., 2017; Panche et al., 2016). It is understood that the thermal approach induces considerable alterations in the chemical composition and the number of bioactive compounds in plant leaves. Therefore, the utilization of heat during the extraction process of the leaves is anticipated to facilitate the degradation of bioactive compounds (Miglio et al., 2008).

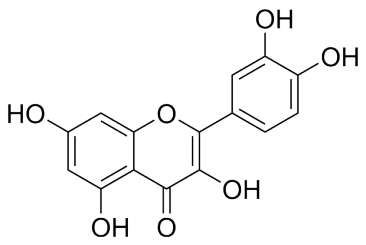

Figure 1. Quercetin-type flavonoid

Herein, we report the green synthesis of SnO_2 NPs using an extract of ground *C. odorata* leaves, whereby the extraction process was conducted with heat-free treatment, and a comparison was made to the boiled extract. The morphological, structural, and optical properties of the prepared SnO_2 NPs were investigated by using field emission scanning electron microscopy (FESEM), energy dispersive X-ray analysis (EDX), X-ray diffraction (XRD) and Fourier-transform infrared (FTIR). To the best of our knowledge, this is the first report ever made on the heat-free treatment of extraction of *C. odorata* leaves in the biosynthesis of SnO_2 NPs.

2. MATERIALS AND METHODS

2.1. Materials

Tin(iv) chloride pentahydrate ($SnCl_4. 5H_2O$) was purchased from Sigma-Aldrich and received without further purification. The leaf samples of *C. odorata* were collected from Kuala Selangor district, Malaysia, and Milli-Q water was used during the experiment.

2.2. Experimental

The synthesis process involved adding approximately 200 ml of *C. odorata* extract to the stirring solution containing 0.05M tin chloride pentahydrate. Some adjustments were made, including the omission of the boiling procedure for the ground leaves in this study's methodology. The stirred solution was allowed to sit for 3 hours at room temperature. Over time, the mixture gradually transformed into a jelly-like substance, which was then subjected to water removal in a conventional oven. The dried product underwent calcination at 700°C for 3 hours to produce SnO_2 NPs (Buniyamin et al., 2021). The SnO_2 NPs sample treated without heat for the extract was denoted as S1, while S2 was assigned to the sample treated with heat.

2.3. Characterizations

The green synthesized SnO_2 NPs was subjected to Scanning Field Emission Electron Microscopy (FESEM) using ZEISS Supra 40VP at 5 kV (80 K magnification), Energy Dispersive X-ray analysis (EDX) using phenom XL Benchtop Scanning Electron Microscope (BSEM), X-ray diffraction patterns using xpert pro pan analytica XRD with CuKα radiation (λ=1.54Å) with ca. 2θ=5º-90º with 45kV and scan speed of 0.417782*/sec with 40mA Fourier-transform Infrared (FTIR) using Perkin Elmer Spectrum 400 in the range 400 to 4000 cm^{-1} by ATR technique.

3. RESULTS AND DISCUSSION

3.1. Plausible Reaction Mechanism of SnO_2 NPs

The plausible reaction mechanism of SnO_2 NPs is displayed in Figure 2. When the precursor salt solution ($SnCl_4. 5H_2O$) was mixed with *C. odorata* leaf extract, the cation of Sn^{4+} was dispersed in the solution and developed a complex containing the active sites of the hydroxyl group quercetin-type flavonoid compound (Gomathi et al., 2021). Four dative bonds were formed when two quercetin molecules, each containing two adjacent hydroxyl groups, established coordination with the tetravalent Sn^{4+} cation. This network is purposely to keep the polyphenolic molecules together and prevent agglomeration. SnO_2 NPs were obtained after the calcination process of the complex.

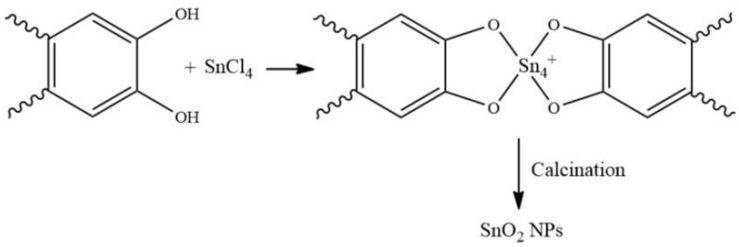

Figure 2. Plausible reaction mechanism for the synthesis of SnO_2 NPs

3.2. Field Emission Electron Microscopy (FESEM) and Energy Dispersive X-ray Analysis (EDX)

FESEM images depict the morphology of SnO_2 NPs generated from the extract without heat treatment (S1) and the extract subjected to heating (S2) in Figure 3 (a). S1 has spherical-like shape with slight agglomeration, leading to a particle size of 16 nm. In contrast, S2 in Figure 3 (b) exhibits tinier nanoparticles (6 nm) and a more uniform distribution, indicating reduced agglomeration. The influence of both extraction processes is evident, with the abundant availability of polyphenolic flavonoids from the heating treatment, potentially contributing to the prevention of agglomeration of SnO_2 NPs, as demonstrated by S2. While for EDX analysis, the presence of prominent peaks of O and Sn elements can be observed in the spectrum at 0.5 and 3.5 eV, with atomic weight percentages for S1 and S2 with an average of 76% for O and 23% for Sn (Table 1). Several foreign peaks were also observed, particularly the weak peaks of chlorine (Cl), nitrogen (N), and carbon (C) at 0.1, 0.2, and 0.3 eV. The findings of the morphological evidence and the elemental composition for both samples agree with previous literatures, thus verifying the pure construction of pure SnO_2 NPs (Gorai, 2018; Garrafa-Galvez et al., 2019; Matussin et al., 2020).

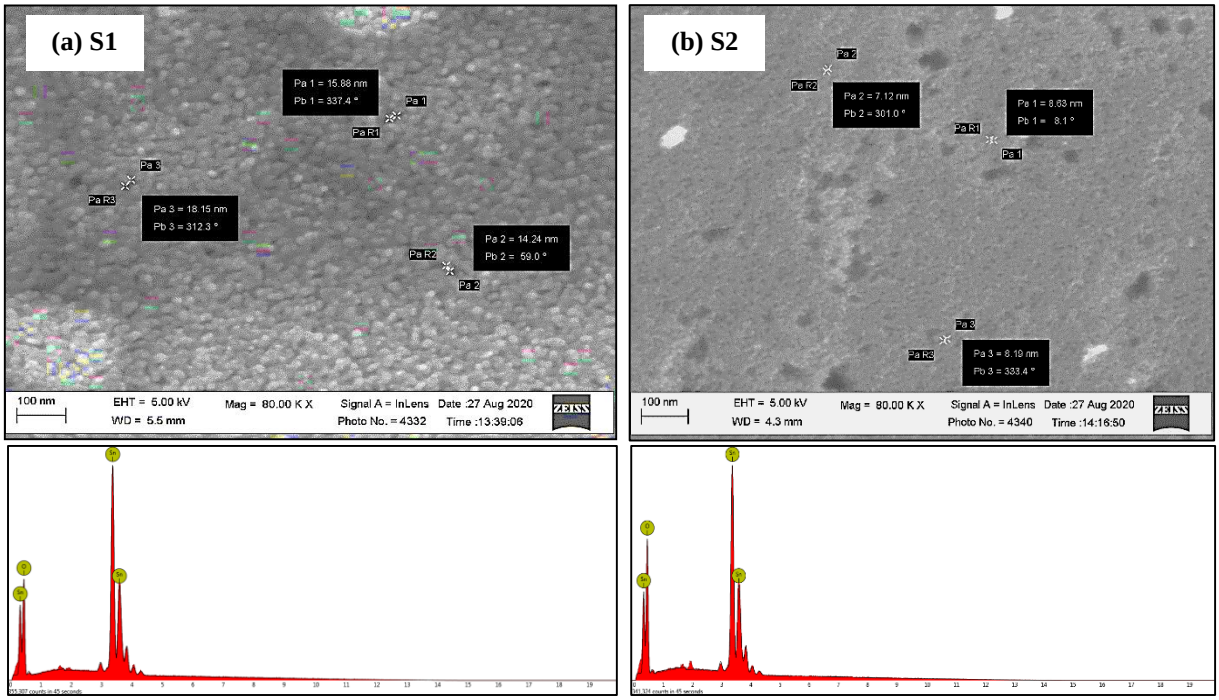

Figure 3. FESEM (a) and EDX pattern (b) images of S1(heat-free) and S2 (heated)

Table 1. The EDX measurement for atomic concentration for S1 and S2

Element	Atomic concentration %	
	S1	**S2**
O	74.78	79.15
Sn	25.22	20.85

3.3. X-ray Diffraction Analysis

XRD result shows the indexing pattern of the synthesized SnO_2 NPs as shown in Figure 4 to be evident during the formation. The most prominent peaks for SnO_2 NPs peaks at 2θ values of 26.5°, 33.7° and 51.5° correlated with (110), (101) and (211) planes signify the formation of SnO_2 with the tetragonal structure according to JCPDS card no.01-077-0452. All peaks are in accord with established data (Matussin et al., 2020; Sethumadhavan et al., 2014). From this result, the construction of SnO_2 NPs is revealed to be crystalline and tetragonal structure, and no other phases have been detected. In addition, the crystallite sizes for S1 and S2 based on the most intense plane (110) are 8.2 nm and 11.5 nm, which calculated from Scherrer's formula as shown in Eq.1, where λ is the X-ray wavelength of Cu Ka (λ=1.54Å), θ is the Bragg's angle, b is the full width at the half maximum (FWHM) in radians and K is unknown shape factor.

$$D = k\lambda/(\beta \cos \theta) \qquad (1)$$

Based on this result, the preparation of *C. odorata* extract, either the use of non-heat or boiled versions, proved to mediate the SnO_2 NPs formation as demonstrated by XRD diffraction peaks. The larger crystallite size born by S2 is presumably associated to its preference of the calcination temperature, in which the narrowness of the peak of (110) that slightly greater than S1 resulted in a slightly larger crystallite size (Głuchowski et al., 2022). Having XRD results for S1 and S2, the three principal peaks which are (110), (101), and (211), display a fairly sharpness pattern indicating good crystallinity which relates with the appropriate formation of SnO_2 NPs.

From this result, it is proven that the heat-free treatment on the preparation of *C. odorata* leaves extract, the prepared extract solution can still offer the bioactive compound sufficient concentration and assist the synthesis of the mechanism by forming the formation of meta-colloidal polyphenolic-Sn^{4+} complex molecule, comparable to the boiled extract. Regarding the potential mechanism, the bioactive compound of *C. odorata* leaves, particularly the polyphenolic flavonoid compound containing the hydroxyl group, has the capability of electron donation and metal chelation due to their nucleophilicity and aromatic rings are significant in contributing their overall electron density, resulting the formation of stable coordination complexes. They are efficient in reducing metals and act as stabilizers for metallic nanoparticles (Oza et al., 2020). The interaction between polyphenolic compounds with metals is presumed to be similar to phenolic acid, forming meta-stable colloidal nanoparticles (Amini & Akbari, 2019).

Reports on the biosynthesis of the metallic compound showed that the application of heat-free treatment could retain the content of such bioactive compounds and possess releasability from the cell leaves to water that later involves the biosynthesis mechanism (Vongsak et al., 2013; Butryee et al., 2009). As the synthesis process involves the reduction, nucleation, and growth of nanoparticles that correlate with excellent mono-dispersion, the retained release of the bioactive compound becomes an essential factor in constructing the nanoparticles (Peralta-Videa et al., 2016).

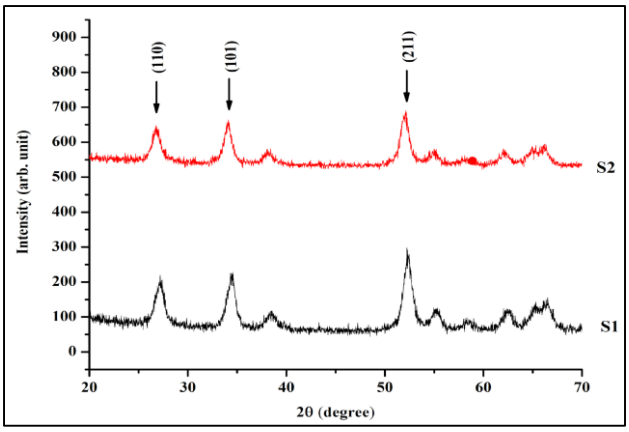

Figure 4. X-ray diffraction patterns of S1(heat-free) and S2 (heated)

3.4. Fourier-transform Infrared (FTIR) Analysis

FTIR analysis as displayed in Figure 5 shows the important transmittance peaks associated with functional groups involved in SnO_2 NPs construction. Of which, the transmittance at the region within 1754 to 1577 cm^{-1} relates to Sn-OH stretching and the vibration of SnO_2 is detected at region 1092 to 941 cm^{-1} (Vidhu & Daizy, 2015; Buniyamin et al., 2022). This implies that the extraction of ground leaves from *C. odorata* carried out at room temperature can still yield polyphenolic flavonoid compounds, similar to the effectiveness observed with the boiled extract. However, it is worth noting that the boiled extract exhibited a slightly higher efficacy in facilitating biosynthesis. This is evidenced in the FTIR spectrum, where S2 (boiled) demonstrated more neat transmittance bands compared to S1 (heat-free).

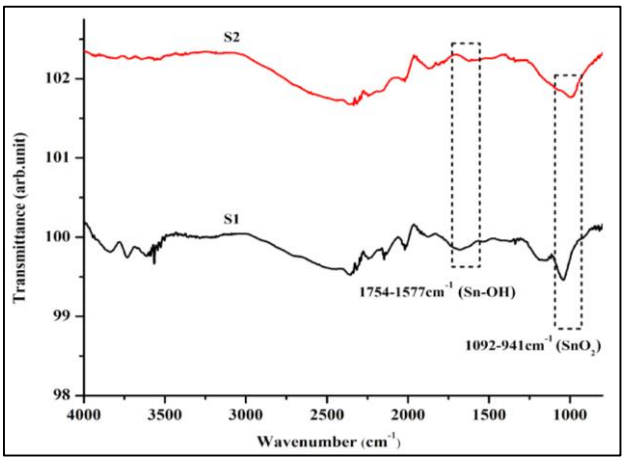

Figure 5. Transmittance evidence of biosynthesized SnO_2 NPs

4. CONCLUSION

Tin(iv) oxide nanoparticles (SnO_2 NPs) have been effectively prepared via a green and eco-friendly approach using heat-free treatment and boiling procedure of *Chromolaena odorata* leaves. FESEM images showed an agglomerated spherical-like shape with a uniform distribution of SnO_2 NPs produced from heat-free extract (S1), presumably influenced by the deficient of polyphenolic flavonoids, that play a role as capping and reducing agent. Elemental compositions of Sn and O were confirmed by EDX analysis with 76% and 23% atomic percentage. XRD results showed the tetragonal structure of SnO_2 NPs with the presence of prominent planes (110), (101), and (211). The construction of SnO_2 NPs was further confirmed

by the FTIR analysis of the transmittance peak of SnO_2 and Sn-OH groups. These results clearly suggest that the extraction process involving heat is preferable, as it ensures a sufficient present of polyphenolic flavonoids to inhibit the agglomeration of SnO_2 NPs. Further investigation is scheduled aiming to offer deeper insights into the optimization of extraction, including the consideration of techniques like the freeze-dried method.

Declaration of Interest
Hereby, the authors declare that there is no conflict of interest in the current work.

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