PRELIMINARY STUDY ON SYNTHESIS OF TIN OXIDE NANOPARTICLE USING GAMMA RADIATION INDUCED METHOD

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ABSTRACT

Radiation induced synthesis provides a clean, scalable, and energy efficient route for producing metal oxide nanoparticles (NPs) with controlled characteristics. This work reports the successful synthesis of tin oxide (SnO_2) NPs through gamma irradiation of aqueous $SnCl_2$ solutions under nitrogen purged conditions. Seven formulations varying in precursor concentration, stabilizer content, and absorbed dose (0–100 kGy) were systematically evaluated using dynamic light scattering (DLS) and transmission electron microscopy (TEM). The optimized formulation 0.05 M $SnCl_2$ without PVP produced highly uniform NPs with a Z-Average of 9.367 nm and a PDI of 0.096. When 1% PVP was introduced, although the Z-Average revealed 25.90 nm and PDI was 0.312, the primary peak remained narrow at 10.43 nm, but additional peaks at 270.8 nm and 4452 nm indicated the formation of secondary agglomerates. These findings demonstrate that precursor concentration, stabilizer content, and irradiation dose critically influence nucleation and growth, enabling the reproducible production of sub 10 nm SnO_2 NPs suitable for integration into polymer-based nanocomposites and gas sensing applications.

ABSTRAK

Sintesis teraruh sinaran menyediakan laluan yang bersih, berskala dan cekap tenaga untuk menghasilkan nanozarah logam oksida (NP) dengan ciri terkawal. Kerja ini melaporkan kejayaan sintesis NP oksida timah (SnO_2) melalui penyinaran gamma larutan $SnCl_2$ berair di bawah keadaan dibersihkan nitrogen. Tujuh formulasi yang berbeza dalam kepekatan prekursor, kandungan penstabil, dan dos yang diserap (0–100 kGy) dinilai secara sistematik menggunakan penyerakan cahaya dinamik (DLS) dan mikroskop elektron penghantaran (TEM). Formulasi yang dioptimumkan 0.05 M $SnCl_2$ tanpa PVP menghasilkan NP yang sangat seragam dengan Z-Purata 9.367 nm dan PDI 0.096. Apabila 1% PVP diperkenalkan, walaupun Z-Average mendedahkan 25.90 nm dan PDI ialah 0.312, puncak utama kekal sempit pada 10.43 nm, tetapi puncak tambahan pada 270.8 nm dan 4452 nm menunjukkan pembentukan aglomerat sekunder. Penemuan ini menunjukkan bahawa kepekatan prekursor, kandungan penstabil dan dos penyinaran secara kritikal mempengaruhi nukleasi dan pertumbuhan, membolehkan pengeluaran boleh dihasilkan semula sub 10 nm SnO_2 NP yang sesuai untuk penyepaduan ke dalam nanokomposit berasaskan polimer dan aplikasi penderiaan gas.

Keywords: metal oxide nanoparticles, radiation induced synthesis, dynamic light scattering, PVP

INTRODUCTION

Tin oxide (SnO_2) nanoparticles are widely recognized for their significant applications in various fields such as catalysis, nanophotonic, biotechnology [1], sensors and energy storage due to their unique electrical, optical, and catalytic properties. Traditional methods of synthesizing tin oxide nanoparticles, including sol-gel [2], Ultraviolet-visible light or laser-induced photochemical reactions [3], and X-ray irradiation using synchrotron radiation [4], often require high temperatures, lengthy processing times, and the use of hazardous chemicals, which can be both energy-intensive and environmentally detrimental.

Radiation-based synthesis presents a promising alternative, potentially offering a cleaner, more efficient route to nanoparticle production. It offers a considerable control on the size distribution of the nanoparticles which can be carried out at room temperature [5]. However, there is little information to support this novel production method. This could be caused by the lack of irradiation facilities elsewhere. Nevertheless, the advantages of this method cannot be overlooked.

Conventional methods such as solvothermal synthesis, furnace techniques, and chemical vapor deposition (CVD) presents several significant challenges that impede their scalability and efficiency. Solvothermal synthesis often requires high pressures and temperatures, leading to complex and costly setups [6]. Furnace techniques, while effective, are energy-intensive and time-consuming, making them less viable for large-scale production. CVD, on the other hand, involves toxic precursors and high operational costs due to the need for sophisticated equipment and stringent control of reaction conditions. These traditional methods also frequently result in inconsistent particle sizes and purities, which can affect the performance of the nanoparticles in applications [8].

Gamma radiation processing has emerged as a powerful tool for nanomaterial synthesis, capitalizing on the radiolysis of water to generate reactive radical species capable of driving oxidation, reduction, and nucleation without added chemicals [10]. This approach offers unique advantages such as low temperature processing, high penetration depth, and precise control over radical driven reactions. It simplifies the process and reduces the need for extensive purification steps that are typically required in conventional synthesis methods to remove residual chemicals. As a result, gamma-induced method not only more cost-effective but also environmentally sustainable, producing high-purity tin oxide nanoparticles suitable for large-scale production.

This study investigates the effect of $SnCl_2$ concentration, polyvinylpyrrolidone (PVP) stabilizer content, and gamma dose on SnO_2 NPs formation. The objective was to identify the optimal parameter set for producing stable, sub 10 nm SnO_2 NPs and to establish a reproducible synthesis protocol suitable for future scale up.

METHODS

Materials

Materials Tin (II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$, ≥99% purity) and polyvinylpyrrolidone (PVP, average molecular weight 40,000) were used as precursors and stabilizing agents, respectively. Deionized water was used as the solvent for all preparations. All chemicals were obtained from reputable suppliers and used without further purification.

Preparation of Tin Chloride Solutions

Aqueous solutions of tin chloride with varying concentrations (0.05 M and 0.1 M) were prepared by dissolving $SnCl_2 \cdot 2H_2O$ in deionized water. For selected samples, PVP was added as a stabilizer at concentrations of 1% (w/v) relative to the solution. The solutions were mixed thoroughly until complete dissolution was achieved. 1M HCl was added to ensure complete dissolution and to suppress premature hydrolysis. Solutions were then purged

with nitrogen. Each solution was transferred into sealed glass vials to minimize contamination and evaporation during irradiation.

Gamma Irradiation Procedure

The prepared solutions were irradiated in the SINAGAMA gamma irradiation facility of the Malaysian Nuclear Agency, using a cobalt-60 source. The total absorbed dose varies from 0 kGy up to 100 kGy under ambient temperature and pressure. This dose was chosen based on preliminary studies to ensure the generation of sufficient radicals for NPs formation. The irradiation process causes the radiolysis of water, producing reactive species such as hydroxyl radicals $\bullet OH$, hydrogen atoms $H\bullet$, and solvated electrons e^-aq, which are responsible for the reduction of Sn^{2+} ions to SnO_2 NPs

Characterization

Dynamic light scattering (DLS) was used to measure the particle size and polydispersity index (PDI), to identify the changes of the particle size after the synthesis process. Transmission electron microscopy provided direct images of the particles, showing their shape and how their sizes were distributed. In addition, atomic force microscopy was used to observe the surface features of the particles. Together, these three techniques provided a clear and reliable understanding of particle formation, size, and surface characteristics.

RESULT AND DISCUSSION

The AFM (figure 1) and TEM (figure 2-5) images clearly demonstrate that the prepared sample contains nanosized features. In the AFM image, small black dots can be seen across the surface, indicating that nanoparticles were successfully formed and distributed across the surface. The TEM image provides further confirmation by showing individual nanoparticles more clearly and allowing their size to be measured around 5.55 nm to 6.93 nm (figure 5). Together, these two techniques validate that the synthesis process produced nanoparticles at the expected scale and that they are present both on the surface and within the sample.

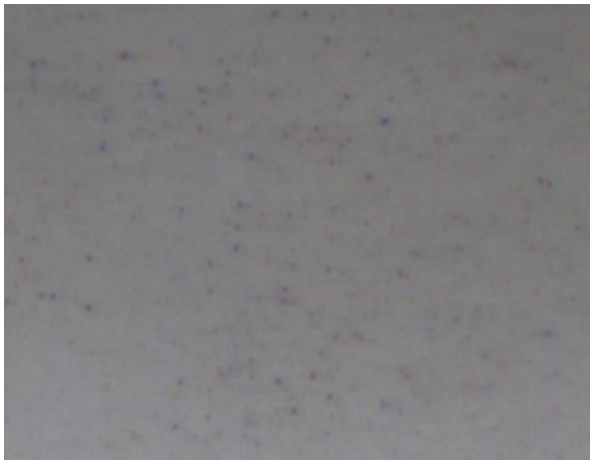

Figure 1: AFM image showing nanosized features (black dots) on the surface measured.

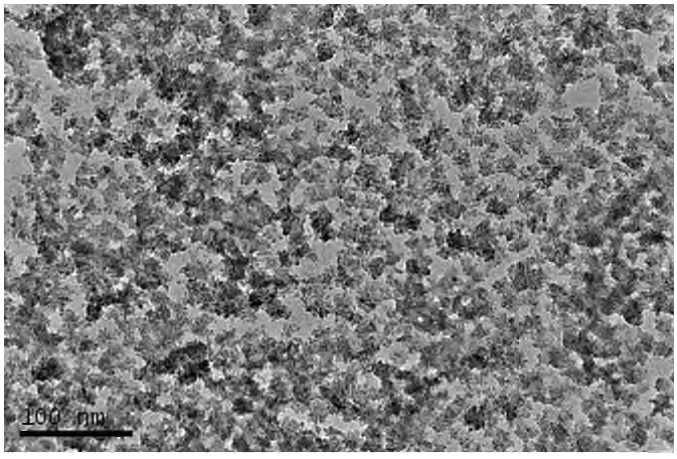

Figure 2: 40x magnification TEM image of SnO_2 NPs.

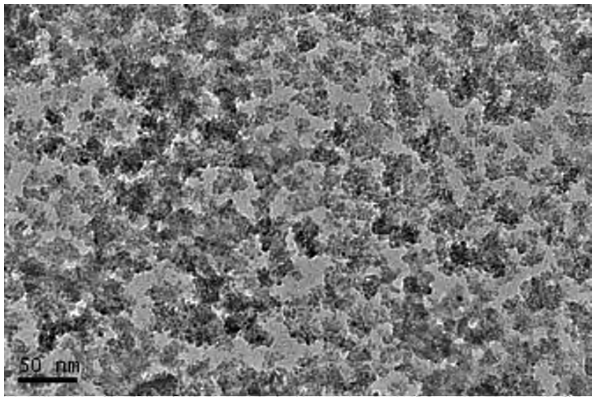

Figure 3: 80x magnification TEM image of SnO_2 NPs.

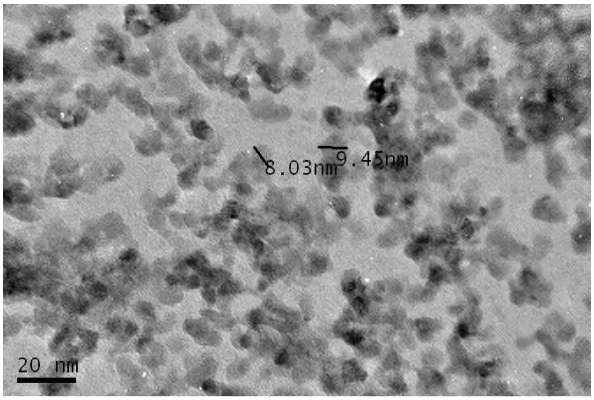

Figure 4: 150x magnification TEM image of SnO_2 NPs with the size of 8.03 nm and 9.45 nm being measured.

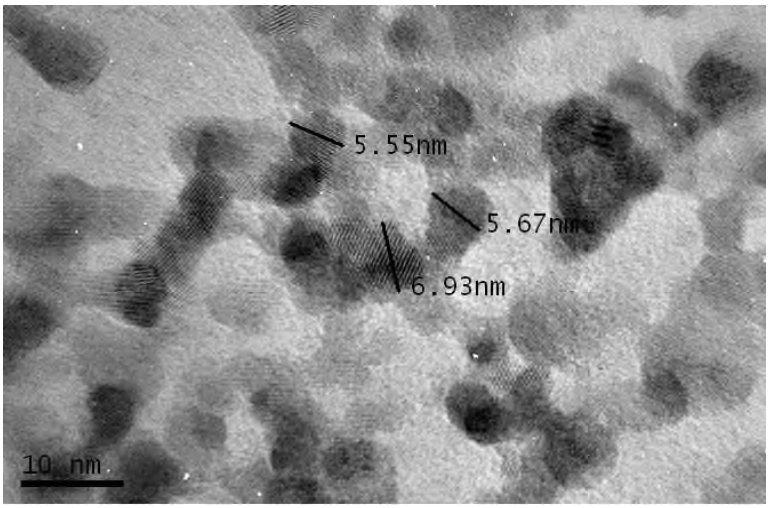

Figure 5: 200x magnification TEM image of SnO_2 NPs with the size of 5.55 nm, 5.67 nm and 6.93 nm being measured.

The synthesis method using gamma radiation was able to produce SnO_2 NPs for all concentrations of the solutions of tin chloride. The particle size results show that both concentration and radiation dose strongly affect NPs formation. At the lower concentration of 0.05 M without any stabilizing agent, increasing the dose from 25 to 50 kGy caused the Z-average to increase, suggesting the formation of larger aggregates. However, at 100 kGy the particle size dropped sharply to below 10 nm, indicating that higher doses promoted further breakdown and formation of smaller NPs. This can be seen in sample 3 with 0.05M and 0% PVP produces 9.322 $\pm$ 2.9 nm with a PDI of 0.079 as per Table 1.

When 1% stabilizing agent was added at the same concentration and dose (100 kGy), the particle size remained small for most of it. DLS measurements as per figure 1 revealed a Z-average diameter of 25.90 $\pm$ 2.26 nm with a PDI of 0.312. Larger size peaks can also be seen with the average size of 270.8 $\pm$ 82.78 nm and 4452 nm $\pm$ 975.6 nm. This indicated the formation of secondary agglomerates.

Table 1: Dynamic light scattering (DLS) results for the analyzed samples.

Sample	Concentration (M)	Stabilizing agent (%)	Dose (kGy)	Average Size (nm)	PDI
1	0.05	0	25	190.9	0.262
2	0.05	0	50	298.2	0.598
3	0.05	0	100	9.367	0.096
4	0.05	1	100	25.90	0.348
5	0.1	0	25	62.71	1
6	0.1	0	50	182.2	0.461
7	0.1	0	100	796.7	0.646

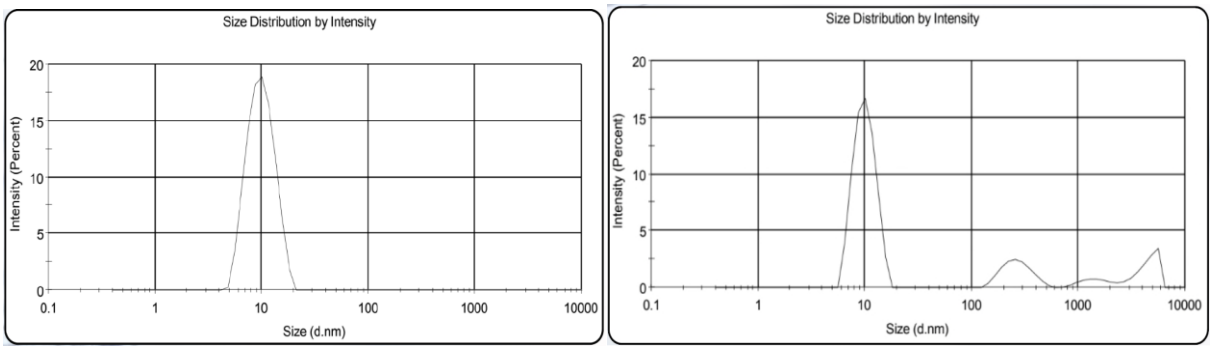

Figure 6: Dynamic light scattering (DLS) measurements comparison between sample 3 (0.05M, 0% PVP and 100 kGy) vs sample 4 (0.05M, 1% PVP and 100 kGy)

At the higher concentration of 0.1 M, the samples behaved differently. At 25 kGy the particle size was moderate, but at 50 kGy the particles became smaller. When the dose reached 100 kGy, the Z-average increased sharply, showing significant aggregation at high concentration. The PDI values also support this trend, where higher dose and higher concentration samples generally show broader size distributions, indicating less stable particle formation.

CONCLUSIONS

This proves that gamma radiation can successfully produce SnO_2 NPs, demonstrating that the technique is effective for NPs formation. The results show that both concentration and radiation dose strongly influence particle size. At 0.05 M concentration without stabilizer, from 25 to 50 kGy led to larger particles, indicating early aggregation, while a further increase to 100 kGy produced much smaller NPs. In conclusion, achieving uniform SnO_2 NPs requires balanced optimization of concentration, stabilizer amount, and radiation dose.

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