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Short Communication

Controlled Reflux Synthesis of MnO_2 and SnO_2: Influence of Water Medium on Product Yield and Synthesis Efficiency

Anushita A. Moger*, Jyoti V. Gupta, Mrunal Marchant and Umesh Prajapati
Chemistry Laboratory, VIVA College of Arts, Science and Commerce, Virar (West), India
anushitatare@vivacollege.org

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Abstract

Metal oxide semiconductors have gained considerable attention due to their excellent chemical stability, redox activity and potential applications in gas sensing, catalysis, energy storage, and electronic devices. In the present study, manganese dioxide (MnO_2) and tin dioxide (SnO_2) nanoparticles were synthesized using a simple and economical reflux-assisted chemical method. The influence of aqueous reaction medium, specifically distilled water and deionized water on synthesis efficiency and product yield was investigated. MnO_2 was synthesized through an oxidation–precipitation reaction using manganese chloride tetrahydrate ($MnCl_2 \cdot 4H_2O$) and potassium permanganate ($KMnO_4$) under acidic conditions at controlled reflux temperature (95–100°C). SnO_2 was prepared by hydroxide precipitation of anhydrous tin chloride ($SnCl_2$) followed by oxidation using hydrogen peroxide under reflux conditions (80–90°C). The synthesized materials were obtained as brownish-black MnO_2 and white SnO_2 powders. The comparative study showed that the type of water medium significantly influenced the obtained yield indicating the important role of solvent purity in reaction kinetics and precipitation behavior. The reflux-based synthesis approach demonstrated good simplicity, reproducibility and scalability. The prepared metal oxides can be further explored for thin/thick film preparation and gas sensing applications.

Keywords: MnO_2, SnO_2, reflux synthesis, metal oxide semiconductor, distilled water, deionized water, precipitation method.

Introduction

Metal oxide semiconductors have attracted significant research interest due to their excellent electrical, optical and chemical characteristics allowing them to be employed in various fields such as gas sensing, energy storage, catalysis and electronics[1-3]. These materials exhibit high chemical stability, tunable electronic structures and excellent surface reactivity which contribute to their growing importance in materials science and nanotechnology[2,3].

Among the various metal oxide semiconductors, manganese dioxide (MnO_2) and tin dioxide (SnO_2) are considered promising functional materials because of their outstanding physicochemical properties and broad range of technological applications[2,3]. Manganese dioxide is a transition metal oxide that exists in several crystalline polymorphs and oxidation states providing excellent redox activity, chemical stability and catalytic performance. Consequently, MnO_2 has been extensively investigated for supercapacitors, batteries, catalysts, environmental remediation and gas sensing applications[2-4].

Tin dioxide (SnO_2) is n-type wide band-gap semiconductor well known for its high thermal stability, optical transparency, electrical conductivity and gas-sensing characteristics. SnO_2-based materials have been widely employed for detecting ammonia, hydrogen, carbon monoxide and other hazardous gases as the interaction between surface-adsorbed oxygen species and target gases leads to changes in the electrical resistance of the material[4-7]. The sensing performance of SnO_2 is strongly influenced by particle size, morphology, crystallinity, surface defects and synthesis conditions[5,6,8].

Metal oxide nanomaterials have been prepared using a variety of synthesis methods such as sol–gel processing, hydrothermal synthesis, spray pyrolysis, microwave-assisted methods and reflux-assisted chemical synthesis have been reported for preparing MnO_2 and SnO_2 nanomaterials[1,9]. Among these methods, reflux-assisted synthesis offers several advantages including uniform heating, improved mixing, controlled reaction kinetics, high reproducibility, low processing cost and simple experimental requirements[1,9].

The purity of the reaction medium also plays an important role during aqueous synthesis because dissolved ions and impurities influence nucleation, crystal growth, oxidation reactions, precipitation behavior and the final product yield[2,3]. Deionized water contains significantly fewer dissolved ionic species than distilled water, thereby providing a more controlled reaction environment during nanoparticle synthesis[2].

A simple reflux-assisted chemical route was employed to synthesize MnO_2 and SnO_2 nanoparticles. The influence of distilled water and deionized water as reaction media on the synthesis process, product yield and overall synthesis efficiency was systematically investigated to evaluate the role of solvent purity during metal oxide nanoparticle preparation[2,4,9].

Methodology

Materials: Analytical-grade manganese chloride tetrahydrate ($MnCl_2 \cdot 4H_2O$), anhydrous tin chloride ($SnCl_2$), potassium permanganate ($KMnO_4$), hydrogen peroxide (H_2O_2), hydrochloric acid (HCl), distilled water and deionized water were used as precursor materials for the synthesis of MnO_2 and SnO_2. All reagents were of analytical grade and were used directly without any additional purification[1,4].

Synthesis of MnO_2: MnO_2 nanoparticles were prepared using an oxidation–precipitation route with manganese chloride tetrahydrate ($MnCl_2 \cdot 4H_2O$) and potassium permanganate ($KMnO_4$) as the precursor chemicals[1-3]. To evaluate the effect of the reaction medium, 5 g of $MnCl_2 \cdot 4H_2O$ was dissolved separately in distilled water and deionized water to prepare two independent reaction systems. Hydrochloric acid was then added to acidify the solution, after which the mixture was heated under reflux at 95–100°C while being continuously stirred using a magnetic stirrer[1].

A freshly prepared $KMnO_4$ solution was introduced slowly into the refluxing mixture under constant stirring. As the reaction progressed, a brownish-black precipitate gradually appeared confirming the formation of MnO_2 nanoparticles through the oxidation process[1,3]. Upon completion of the reaction, the precipitate was separated by filtration and washed several times with distilled water to remove any remaining ions and unreacted precursor species. The resulting product was dried in a hot-air oven and stored for subsequent characterization and yield evaluation[1,2].

Synthesis of SnO_2: SnO_2 powder was synthesized using anhydrous $SnCl_2$ as the precursor through a hydroxide precipitation method followed by oxidation under reflux conditions[4,10]. For the synthesis, 5g of $SnCl_2$ was dissolved separately in distilled water and deionized water to prepare two reaction systems.

The solution was subjected to hydroxide precipitation, followed by the controlled addition of hydrogen peroxide to promote oxidation of the intermediate tin hydroxide into tin dioxide. The reaction mixture was refluxed at 80–90°**C** under continuous stirring to ensure complete oxidation and uniform particle formation[4,9,10].

After the reaction was completed, the resulting white precipitate was collected by filtration and thoroughly washed with distilled water to remove any soluble impurities and residual reactants. The purified product was then dried in a hot-air oven to obtain SnO_2 powder. After drying the powder was stored in airtight containers for subsequent characterization and product yield analysis[4,6,9].

Results and Discussion

Effect of Water Medium on Synthesis of MnO_2 and SnO_2: The effect of the aqueous reaction medium on the synthesis efficiency of manganese dioxide (MnO_2) and tin dioxide (SnO_2) was investigated using distilled water and deionized water as reaction solvents. Both metal oxides were successfully synthesized through a reflux-assisted chemical method under controlled temperature conditions[1,2,9].

During the synthesis of MnO_2, manganese chloride tetrahydrate ($MnCl_2 \cdot 4H_2O$) and potassium permanganate ($KMnO_4$) were used as precursor materials under acidic reflux conditions. In both distilled water and deionized water media, a brownish-black precipitate was obtained confirming the successful formation of MnO_2. The experimental yield of MnO_2 was found to be 3.15–3.22 g.

Only a slight variation in product yield was observed between the two reaction media. This difference may be attributed to the presence of dissolved ions and trace impurities, which can influence nucleation, oxidation kinetics, crystal growth and precipitation behaviour during MnO_2 formation[2,3]. The controlled reflux temperature of 95–100°C provided sufficient thermal energy for the oxidation of Mn^{2+} ions and promoted the formation of crystalline MnO_2[1,3].

For the synthesis of SnO_2, anhydrous $SnCl_2$ was employed as the precursor material and hydroxide precipitation followed by oxidation using hydrogen peroxide was carried out under reflux conditions[4,9]. A white SnO_2 powder was successfully obtained after filtration, washing and drying. The experimental yield varied between 1.58 and 3.16g depending on the reaction medium employed.

The comparatively larger variation in SnO_2 yield may be associated with the sensitivity of tin hydroxide precipitation and its subsequent oxidation to solvent purity, ionic concentration, reaction kinetics and pH conditions[4,6,9].

Deionized water contains a significantly lower concentration of dissolved ionic species than distilled water and therefore provides a more controlled reaction environment for nanoparticle synthesis. Reduced ionic interference can promote more uniform nucleation, improved crystal growth and higher precipitation efficiency, resulting in improved product recovery[2,3]. The present observations are therefore consistent with previous reports describing the influence of reaction medium purity on the synthesis behaviour and physicochemical properties of metal oxide nanomaterials[2,3].

Overall, the reflux-assisted synthesis method employed in the present work proved to be a simple, economical, reproducible and scalable route for the preparation of MnO_2 and SnO_2 powders. Similar advantages of reflux-assisted and solution-based synthesis methods have been reported in earlier investigations on metal oxide nanomaterials[1,2,9].

Conclusion

In the present study, MnO_2 and SnO_2 powders were successfully synthesized using a reflux-assisted chemical method. The effect of the aqueous reaction medium was examined by comparing the synthesis carried out in distilled water and deionized water.

The synthesis of MnO_2 showed nearly similar yields in both reaction media while SnO_2 exhibited a noticeable variation in yield depending on the purity of the solvent used. These observations suggest that the chemical nature of the reaction medium influences precipitation, oxidation behaviour and overall product formation.

The developed synthesis approach is simple, economical and reproducible, making it suitable for the preparation of metal oxide materials for further studies related to sensing and other functional applications.

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