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Synthesis and Characterization of Mixed SnO_2–ZnO Nano Composites Material to Study the Kinetics of Catalytic Reduction of Aromatic Nitro Compounds

Sayantan S. Pal[1], Janvi S. Sah[1], Nandini M. Gupta[1], Ashok N. Patange[2,*]

Abstract

A straightforward and cost-effective method was employed to synthesize zinc oxide (ZnO) nanostructures supported on tin oxide (SnO_2) for catalytic applications. This hybrid nanocomposite, formed by integrating ZnO with SnO_2, exhibited impressive catalytic activity in the reduction of various nitro compounds. The synergistic interaction between ZnO and SnO_2 significantly enhanced the surface area and active sites of the catalyst, thereby improving its overall efficiency. The resulting SnO_2–ZnO nanocomposite not only exhibited exceptional catalytic reactivity but also showcased excellent stability over multiple cycles, along with remarkable reusability, making it highly practical for long-term applications. Its consistent performance without significant loss of activity highlights its robustness under reaction conditions. Owing to these advantageous features, the SnO_2–ZnO nanocomposite emerges as a highly promising and eco-friendly catalyst for the efficient transformation of toxic nitro compounds into valuable amine derivatives. Such a conversion is crucial in both environmental remediation and the synthesis of pharmaceuticals, dyes, and agrochemicals, where clean and sustainable chemical processes are increasingly in demand. This research study creates a way to explore the strength of mixed metal oxide composite material in the catalytic reduction of aromatic nitro compounds. It was observed that the reduction of nitro compounds takes place at faster rate with increasing the yield of products as compared to the traditional reducing agents, like $NaBH_4$, the surface morphology of synthesized mixed SnO_2–ZnO composite was studied with the help of scanning electron microscopy by using WDS (JEOL Japan Model : JSM7600 F. kinetics of catalytic reduction process of nitro substituted aromatic compound's was studied with the help of UV spectrophotometer.

Keywords: Metal oxides, nanoparticles, nano composite, catalyst, reduction, nitro compounds, efficiency, SEM

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INTRODUCTION

Catalytic reduction of aromatic nitro compounds, viz., o-Nitrophenol, p-Nitrophenol, and m-Dinitrobenzene are very useful in pharmaceutical, research and development, dyes and pigment, explosive, chemical reactions [1]. These compounds can pose environmental hazards due to their toxicity and potential for contamination. These substances have the potential to pollute water bodies, threatening the health of aquatic organisms. Additionally, they can seep into the soil, disrupting plant growth and possibly making their way into the food chain, which can impact both ecosystems and human health. The production and use of these

compounds can lead to air pollution. These compounds are toxic and can pose health risks to humans and wildlife, including Exposure to high levels of these compounds can cause acute toxicity, leading to symptoms, such as headaches, dizziness, and nausea [2]. Prolonged exposure to these substances can result in chronic toxicity, which may contribute to serious health conditions, including cancer and reproductive disorders. The current situation needs efficient water treatment ways to eliminate the harmful pollutants or reducing its toxicity and then releasing in environment. There are several methods available for the degradation of nitro compounds, including chemical oxidation, photocatalytic reduction, coagulation, and others. Among these, catalytic reduction stands out as one of the most efficient and preferred techniques. This method is highly valued for its excellent selectivity, making it ideal for targeting specific nitro compounds without affecting other components in the system. Additionally, catalytic reduction is cost-effective, relatively simple to implement, and offers high efficiency under mild conditions. Its advantages make it a sustainable and practical approach, particularly in environmental remediation and industrial wastewater treatment, where safe and complete breakdown of toxic nitro compounds is essential. Creating a low-cost catalyst is very important for proper water treatment [3].

The use of SnO_2–ZnO nanocomposite as a catalyst provides numerous advantages in catalytic applications. One of the key benefits is its ability to significantly enhance the reaction rate and overall product yield, making the process more efficient and economically viable. This nanocomposite also enables reactions to occur under milder conditions – such as lower temperatures and pressures – thereby minimizing energy consumption and reducing the risk of undesirable side reactions. As a result, product selectivity is greatly improved, leading to higher purity and better quality of the desired output. Moreover, the synergistic effect between SnO_2 and ZnO in the composite structure contributes to improved surface area, enhanced electron mobility, and greater catalytic activity, which further boosts the overall performance of the reaction system. This makes SnO_2–ZnO nanocomposites an attractive and sustainable option for various industrial and environmental applications. The nanocomposite catalyst can be reused, making the process more sustainable and cost-effective.

The reduced compounds can be used in polymer synthesis, dyes pigment, pharmaceutical, and various chemical synthesis [4].

This work had basically explored the decrease in reduction time by combining two types of metal oxide which helps to reduce time, energy, and resources for effective reduction process.

EXPERIMENTAL METHODS

The Synthesis of SnO_2 Nanoparticles Using a Simple Co-Precipitation Method

Step 1: Preparation of Tin (II) Chloride Solution

The synthesis of SnO_2 nanoparticles began with the preparation of a 0.5 M Tin (II) Chloride solution [5]. Tin (II) chloride dihydrate ($SnCl_2.2H_2O$) was used as the precursor Material. The Tin (II) chloride dihydrate was carefully weighed and transferred to a Beaker, where it was dissolved in distilled water to obtain a 0.5 M solution [6]. The mixture was continuously stirred at room temperature for one hour to ensure that the precursor material was fully dissolved.

Step 2: Adjustment of pH and Precipitation

To initiate the precipitation reaction, the pH of the tin (II) chloride solution was adjusted to 9 by adding a 5% aqueous ammonia (NH_3) solution dropwise. The addition of Ammonia solution was done slowly while stirring the tin (II) chloride solution continuously. The solution was stirred for an additional hour to ensure the complete precipitation of tin (II) hydroxide [7].

Step 3: Nucleation and Aging

The precipitated tin (II) hydroxide was left at room temperature for 24 hours to allow for Nucleation and aging. During this period, the precipitate underwent a series of Transformations, including nucleation, growth, and aggregation, which ultimately led to the formation of SnO_2 nanoparticles.

Step 4: Filtration, Washing, and Drying

After the aging process, the precipitate was filtered using a Buchner funnel and washed continuously with double-distilled water and ethanol to remove any impurities. The washing procedure was carried out multiple times to thoroughly eliminate any remaining impurities. The filtered and washed precipitate was then dried at 80°C for 4 hours to obtain a dry powder [8].

Step 5: Annealing and Crystallization

The dried powder was then annealed at 600°C to obtain crystalline SnO_2 nanoparticles. The annealing process involved heating the powder in a furnace at a controlled temperature and atmosphere. The high-temperature annealing process allowed the SnO_2 nanoparticles to crystallize and obtain their desired structure and properties [9].

Preparation of SnO_2–ZnO Nanocomposite

Step 1: Preparation of Molar Solutions

To initiate the synthesis of the SnO_2–ZnO nanocomposite, two solutions were prepared:

1. *SnO_2 Molar Solution:* 7.299 grams of SnO_2 were dissolved in 100 ml of distilled water to obtain a 0.05 M solution.
2. *$ZnSO_4$ Molar Solution:* 7.189 grams of $ZnSO_4 \cdot 7H_2O$ were dissolved in 100 ml of Distilled water to obtain a 0.05 M solution.

Step 2: Synthesis of SnO_2–ZnO Nanocomposite

The synthesis of the SnO_2–ZnO nanocomposite involved the following steps:

1. *Sonication:* 100 mg of SnO_2 were added to 100 ml of the 0.05 M $ZnSO_4$ solution, and the mixture was sonicated for 5 minutes.
2. *Addition of NaOH:* 125 ml of 0.125 M NaOH solution were added dropwise to the sonicated mixture under magnetic stirring for 3 hours.
3. *Precipitation:* The formed precipitate was left undisturbed at room temperature for 12 hours to allow it to settle completely [10].

Step 3: Post-Synthesis Treatment

The precipitate was subjected to the following post-synthesis treatments:

1. *Filtration:* The precipitate was filtered using a Buchner funnel.
2. *Washing:* The collected precipitate was thoroughly washed several times using double-distilled water and ethanol to ensure the removal of any residual impurities.
3. *Drying:* The washed precipitate was dried carefully in a hot air oven at 80°C for 4 Hours.

Step 4: Obtaining the SnO_2/ZnO Nanocomposite

The resulting dried powder was the SnO_2/ZnO nanocomposite, which was obtained through the successful synthesis and post-synthesis treatments.

RESULT AND DISCUSSION

The investigation of surface morphology and elemental composition [8] surface of synthesized SnO_2, ZnO nano materials and mixed nano composite material SnO_2/ZnO was studied by using Field Emission Gun Scanning Electron Microscope (FEGSEM) with EDS and WDS (JEOL Japan Model: JSM7600 F. The JEOL field emission scanning electron microscope is a highly versatile instrument known for its superior high-resolution imaging capabilities. This instrument integrates two reliable technologies: an electron column equipped with a semi-in-lens objective lens that enables high-resolution imaging at low accelerating voltages, and a Schottky field emission gun (FEG) that generates electron emission using a strong electrostatic field. An FEG emitter produces a more coherent electron beam and offers significantly greater brightness compared to a traditional tungsten filament. Additionally, the instrument features a specialized Gentle Beam (GB) mode, which applies a negative voltage to the sample. This decelerates the incoming electrons just before they reach the specimen, allowing for enhanced resolution even at very low accelerating voltages [11].

Therefore, this technique is well-suited for examining fine structural details, particularly in low-density and non-conductive materials, even at high magnifications. It also enables efficient detection of low-angle backscattered electrons for enhanced imaging quality (Figure 1).

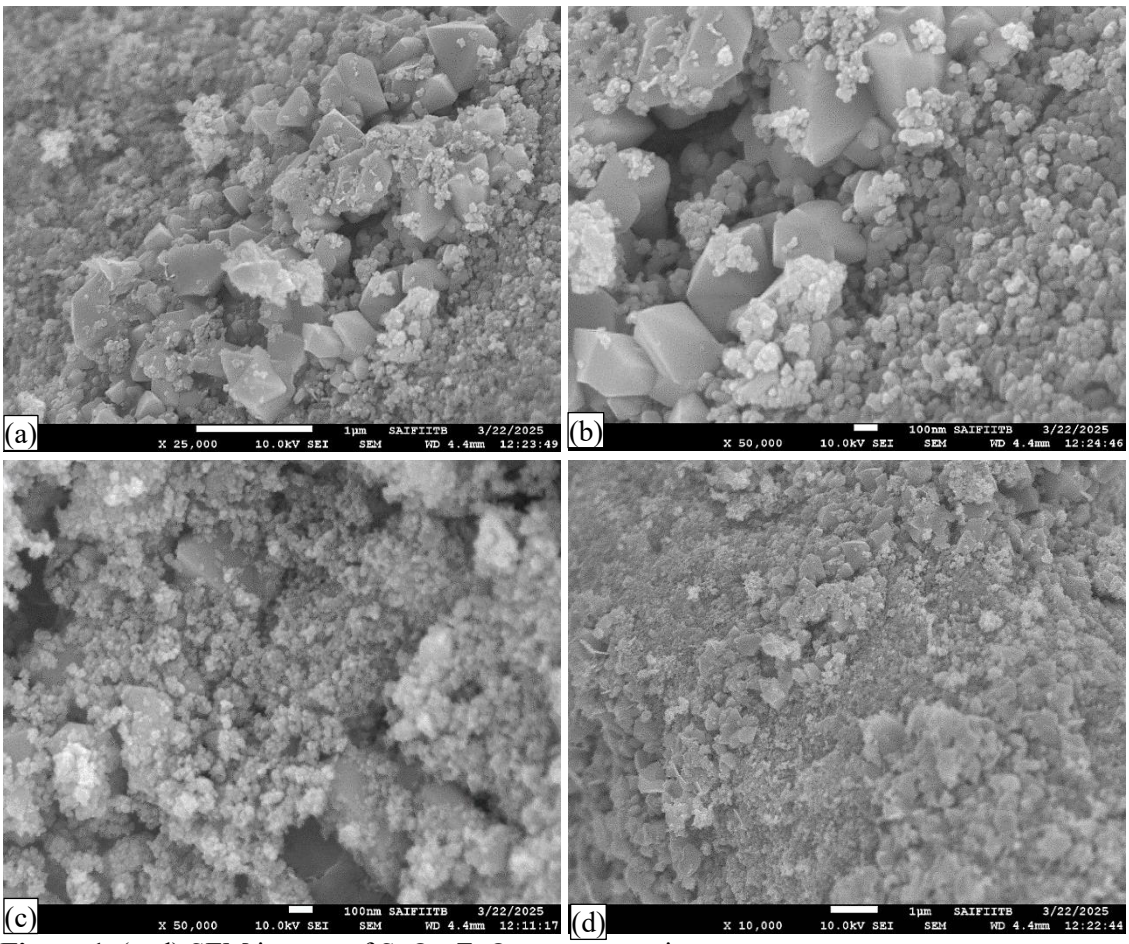

Figure 1. (a–d) SEM images of SnO_2–ZnO nanocomposite.

Catalytic Study

The ability of SnO_2–ZnO nanocomposite to catalyse the reduction of o-nitrophenol, p-nitrophenol, m-dinitrobenzene was studied by using UV-visible spectrophotometer.

MECHANISMS

- *Step 1:* Adsorption of reactants nitro compounds and $NaBH_4$ on the surface of SnO_2–ZnO nanocomposite
- *Step 2:* The catalyst SnO_2–ZnO nanocomposite surface activates the nitro groups, and the $NaBH_4$ reaction with water results in release of Hydrogen gas on the surface of catalyst which is used in the reduction process.

To monitor the reaction here UV-visible spectrophotometer was used.

In Figure 2, we can see the decrease in absorbance of o-nitrophenol is observed. Here, we set the wavelength at 345 nm (maximum absorbance was observed here) with respect to time the absorbance goes on decreasing due to formation of o-aminophenol.

In Figure 3, we can see the decrease in absorbance of p-nitrophenol is observed here. We set the wavelength at 320 nm (maximum absorbance was observed here) with respect to time the absorbance goes on decreasing due to formation of p-aminophenol.

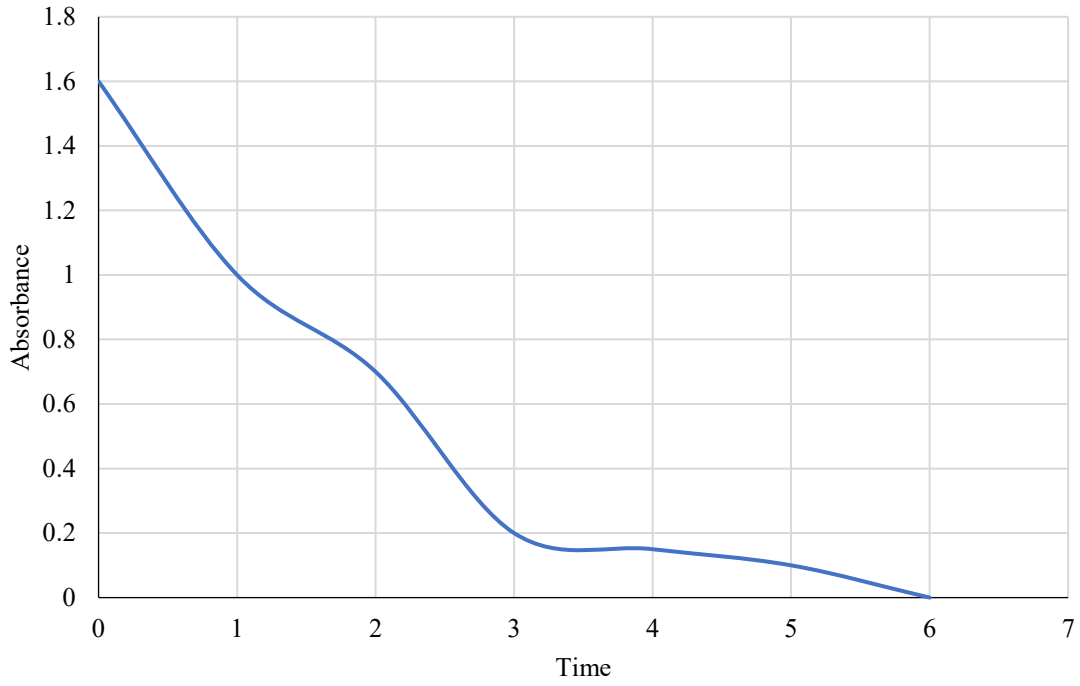

Figure 2. Gradual decline in absorbance over time, suggesting a multi-phase reaction or stepwise degradation process.

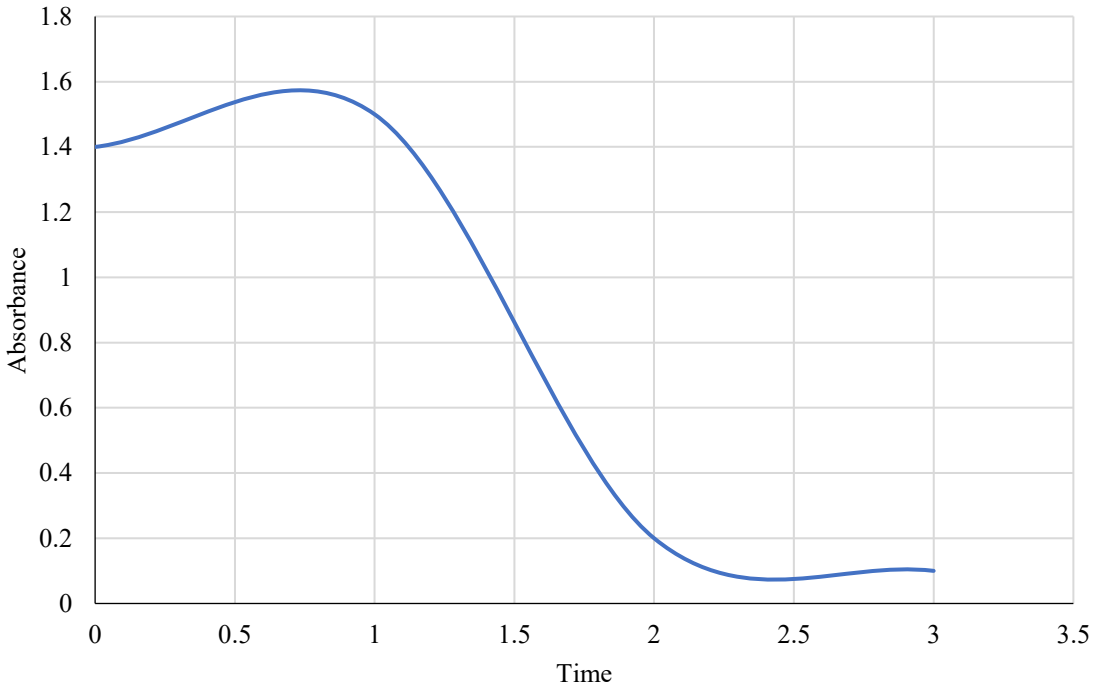

Figure 3. Rapid decline in absorbance over a short time interval, indicating a fast reaction or degradation process.

In Figure 4, we can see the decrease in absorbance of m-dinitrobenzene is observed here. We set the wavelength at 285 nm (maximum absorbance was observed here) with respect to time the absorbance goes on decreasing due to formation of Meta phenylenediamine (m-PDA).

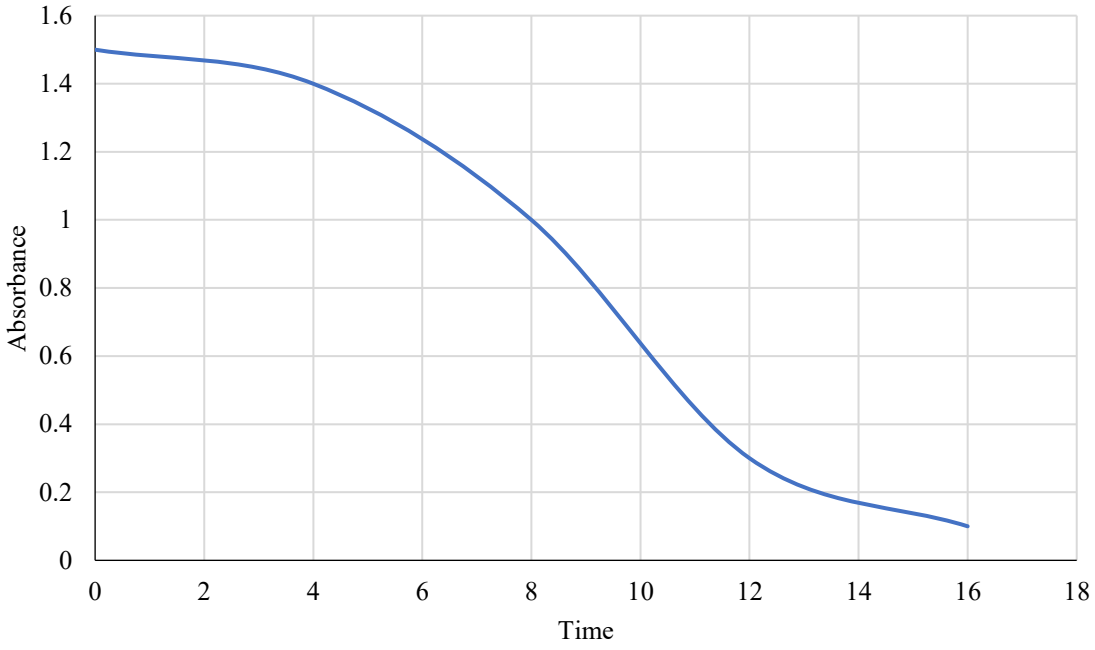

Figure 4. Decrease in absorbance over time indicating a potential degradation or reaction progression.

CONCLUSIONS

The kinetics of catalytic reduction process of aromatic nitro compounds was studied with $NaBH_4$ as a traditional reducing agents and newly synthesized SnO_2–ZnO nanocomposite materials, the reduction of nitro compounds takes place faster rate with increasing the yield of products as compared to the traditional reducing agents, like $NaBH_4$, catalyst enhances the reaction rate by decreasing the activation energy, by activating the nitro groups so that easy reduction takes place. By using catalyst the reduction of nitro compounds had been very fast compared to traditional methods where traditionally o-nitrophenol used to take 30 minutes but by using the mixed metal oxide nanocomposite it is done in 7 minutes, in the same way p-nitrophenol used to take 15 minutes but by using the metal oxide nanocomposite it is done in 3 minutes and for m-dinitrobenzene used to take 45 minutes but by using the metal oxide nanocomposite it is done in 15 minutes.

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