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Eco-Friendly Synthesis of Dibenzylidene Acetone Using Cobalt Oxide Nanoparticle Catalysts

Pallavi Bhondkar[1], Smita Tandale[2], Chetana Tumade[3], Gurumeet Wadhawa[4]
[1] Student, P.G. Department of Chemistry
[2], Professors, Department of Chemistry
[3],[4] Department of Chemistry
Veer Wajekar College, Phunde, Uran

Abstract: *The present study focuses on the green synthesis of cobalt oxide (Co_3O_4) nanoparticles using tamarind leaf extract and their application as heterogeneous catalysts in the synthesis of dibenzylidene acetone (DBA) derivatives. Green synthesis provides an eco-friendly, cost-effective, and sustainable alternative to conventional chemical methods by avoiding the use of toxic reducing agents and hazardous reaction conditions. Phytochemicals present in tamarind leaf extract act as natural reducing and stabilizing agents during the formation of cobalt oxide nanoparticles.*

The synthesized Co_3O_4 nanoparticles were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV–Visible spectroscopy. XRD analysis confirmed the crystalline cubic spinel structure of cobalt oxide nanoparticles, while SEM images revealed nearly spherical and porous nanoparticles with moderate agglomeration. UV–Visible spectroscopic analysis showed strong absorption in the UV region along with extended visible-light absorption, indicating the semiconducting behavior of the nanoparticles.

The synthesized cobalt oxide nanoparticles were successfully utilized as heterogeneous catalysts in Claisen–Schmidt condensation reactions between acetone and substituted aromatic aldehydes for the synthesis of dibenzylidene acetone derivatives. The catalytic system exhibited several advantages including higher product yields, reduced reaction time, improved selectivity, catalyst reusability, and lower environmental impact. The synthesized DBA derivatives were further characterized using UV–Visible spectroscopy, FTIR spectroscopy, and NMR spectroscopy. The study demonstrates that cobalt oxide nanoparticles synthesized through a green route are efficient, reusable, and environmentally benign catalysts for sustainable organic synthesis.

Keywords: Green synthesis, cobalt oxide nanoparticles, dibenzylidene acetone, Claisen–Schmidt condensation, nanocatalysis, UV–Visible spectroscopy, XRD, SEM.

I. INTRODUCTION

Nanotechnology has emerged as an important and rapidly developing field in modern chemical research due to the unique physicochemical properties exhibited by nanoparticles. Nanoparticles possess high surface area, unique optical properties, enhanced catalytic activity, and improved electronic behavior compared to their bulk materials. These remarkable characteristics make nanoparticles highly useful in various scientific and industrial applications. Alongside nanotechnology, green chemistry has gained considerable importance as it focuses on minimizing the use of hazardous chemicals, reducing waste generation, and developing environmentally sustainable synthetic methodologies. Green synthesis approaches provide eco-friendly alternatives to conventional chemical methods and support the principles of sustainable chemistry.

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Among various metal oxide nanoparticles, cobalt oxide nanoparticles have attracted significant attention because of their semiconducting properties, excellent redox behavior, high catalytic activity, visible-light absorption capability, and good chemical stability. Due to these advantageous properties, cobalt oxide nanoparticles are widely applied in photocatalysis, sensors, energy storage devices, environmental remediation, and heterogeneous catalysis. Their high catalytic efficiency and stability make them promising materials for sustainable organic synthesis and environmental applications.

Green Synthesis of Cobalt Oxide Nanoparticles

The green synthesis of cobalt oxide nanoparticles was carried out using tamarind leaf extract as a natural reducing and stabilizing agent. Tamarind leaves contain various phytochemicals such as polyphenols, flavonoids, tannins, and other reducing biomolecules, which play an important role during nanoparticle formation. These bioactive compounds act as reducing agents for the conversion of metal ions into nanoparticles, while simultaneously functioning as stabilizing and capping agents to prevent particle aggregation and improve nanoparticle stability.

The green synthesis approach offers several advantages over conventional chemical methods, including eco-friendly processing, low toxicity, cost-effective synthesis, and reduced generation of hazardous chemical waste. Therefore, plant-mediated synthesis of cobalt oxide nanoparticles represents a sustainable and environmentally benign method for nanoparticle production.

II. MATERIALS REQUIRED

Materials Required

The chemicals used in the present study included cobalt nitrate ([Co(NO_3)_2•6H_2O]), tamarind leaf extract, ethanol, distilled water, and sodium hydroxide. Cobalt nitrate was used as the precursor for the synthesis of cobalt oxide nanoparticles, while tamarind leaf extract served as a natural reducing and stabilizing agent during nanoparticle formation. Ethanol and distilled water were used for washing and preparation of solutions, whereas sodium hydroxide was used for pH adjustment during the synthesis process. All chemicals used were of analytical reagent grade.

Various analytical instruments and laboratory apparatus were employed for synthesis and characterization of the nanoparticles. A UV–Visible spectrophotometer was used to study the optical properties of the synthesized nanoparticles, while X-ray diffraction analysis was carried out using an X-ray diffractometer to determine their crystalline structure and phase purity. Surface morphology and particle size were analyzed using a scanning electron microscope (SEM). A magnetic stirrer was used for continuous mixing during synthesis, and a furnace was used for calcination to obtain crystalline cobalt oxide nanoparticles.

III. SYNTHESIS OF COBALT OXIDE NANOPARTICLES

Green Synthesis of Cobalt Oxide Nanoparticles

Fresh tamarind leaves were thoroughly washed with distilled water to remove dust and impurities. The cleaned leaves were boiled in distilled water for about 20–30 minutes to extract the bioactive phytochemicals present in the leaves. The obtained extract was filtered and allowed to cool to room temperature before use in nanoparticle synthesis.

A 0.1 M cobalt nitrate solution was prepared separately using distilled water. The tamarind leaf extract was then added dropwise into the cobalt nitrate solution under continuous stirring conditions. The reaction temperature was maintained between 60–80°C throughout the synthesis process. During the reaction, a gradual color change from pink to dark brown was observed, indicating the formation of cobalt oxide nanoparticles.

The reaction mixture was centrifuged to collect the synthesized nanoparticles, and the obtained precipitate was repeatedly washed with distilled water and ethanol to remove impurities and unreacted materials. The purified product

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was dried at 80–100°C and subsequently calcined at 400–500°C to obtain crystalline cobalt oxide (Co_3O_4) nanoparticles with improved crystallinity and stability.

IV. CHARACTERIZATION OF COBALT OXIDE NANOPARTICLES

X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) analysis was carried out to determine the crystalline structure and phase purity of the synthesized cobalt oxide nanoparticles. The analysis is based on Bragg's law, n\lambda = 2d\sin\theta where (n) represents the order of diffraction, (\lambda) is the wavelength of X-rays, (d) is the interplanar spacing, and (\theta) is the diffraction angle. The diffraction peaks observed at 2θ values of 31.3°, 36.8°, 44.8°, 59.3°, and 65.2° corresponded to the crystal planes (220), (311), (400), (511), and (440), respectively. These characteristic peaks confirmed the formation of crystalline cobalt oxide nanoparticles with cubic spinel Co_3O_4 structure. The broadening of diffraction peaks indicated nanoscale crystallite size, while the absence of impurity peaks confirmed the phase purity of the synthesized nanoparticles.

Scanning Electron Microscopy (SEM)

SEM analysis was performed to study the surface morphology and particle distribution of the synthesized cobalt oxide nanoparticles. The SEM images revealed nearly spherical nanoparticles with porous surface morphology and slight agglomeration. The nanoparticles were found to be uniformly distributed with nanoscale dimensions. The porous morphology of the nanoparticles provides high surface area, which enhances catalytic efficiency and improves adsorption behavior during catalytic reactions.

UV–Visible Spectroscopy of Co_3O_4 Nanoparticles

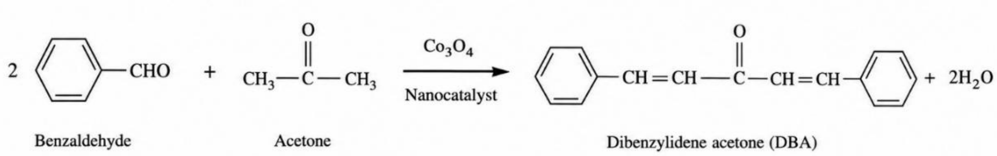
UV–Visible spectroscopic analysis was carried out to investigate the optical properties and electronic transitions of cobalt oxide nanoparticles. UV–Visible spectroscopy measures the excitation of electrons from the valence band to the conduction band in semiconductor nanoparticles. The synthesized Co_3O_4 nanoparticles showed strong absorption in the UV region around 250–350 nm along with extended absorption in the visible region between 400–600 nm. These absorption characteristics confirmed the semiconducting behavior of cobalt oxide nanoparticles and indicated their potential application in visible-light photocatalysis and heterogeneous catalytic reactions.

V. SYNTHESIS OF DIBENZYLIDENE ACETONE (DBA)

Reaction Principle

- DBA synthesized via Claisen–Schmidt condensation.
- Benzaldehyde reacts with acetone in presence of Co_3O_4 nanocatalyst.

Overall reaction:



$$2C_6H_5CHO + CH_3COCH_3 \xrightarrow[\text{Nanocatalyst}]{Co_3O_4} C_6H_5CH{=}CHCOCH{=}CHC_6H_5 + 2H_2O$$

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VI. EXPERIMENTAL PROCEDURE

Reaction Steps

Benzaldehyde and acetone were initially mixed in ethanol to prepare the reaction mixture, and the synthesized Co_3O_4 nanoparticles were added as a heterogeneous catalyst. The reaction mixture was then stirred continuously at 50–60°C, and sodium hydroxide solution was added to initiate the Claisen–Schmidt condensation reaction. During the course of the reaction, the appearance of yellow to orange coloration indicated the formation of dibenzylidene acetone (DBA) derivatives. After completion of the reaction, the mixture was poured into ice-cold water to precipitate the product. The resulting solid was filtered, purified by recrystallization, and dried to obtain pure DBA derivatives.

VII. OBSERVATION TABLE

Aldehyde Used	Product	Yield (%)	Melting Point (°C)
Benzaldehyde	DBA	85–90	110–112
4-Methylbenzaldehyde	p-Methyl DBA	80–88	120–123
4-Methoxybenzaldehyde	p-Methoxy DBA	78–85	135–138
4-Chlorobenzaldehyde	p-Chloro DBA	82–89	140–145
4-Nitrobenzaldehyde	p-Nitro DBA	75–82	160–165

VIII. CHARACTERIZATION OF DBA

UV–Visible Spectroscopy

UV–Visible spectroscopic analysis of the synthesized dibenzylidene acetone (DBA) derivatives showed characteristic absorption peaks corresponding to different electronic transitions. Absorption bands observed in the range of 240–260 nm were assigned to $\pi \rightarrow \pi^*$ transitions, while peaks between 300–330 nm corresponded to conjugated $\pi \rightarrow \pi^*$ transitions. Additional absorption observed in the range of 340–360 nm was attributed to $n \rightarrow \pi^*$ transitions of the conjugated carbonyl system. The observed bathochromic shift and extended absorption confirmed the presence of a highly conjugated α,β-unsaturated ketone system and indicated successful formation of DBA derivatives.

FTIR Analysis

FTIR spectroscopic analysis confirmed the formation of dibenzylidene acetone derivatives through characteristic absorption bands. The strong absorption peak observed in the range of 1660–1680 cm^{-1} corresponded to C=O stretching vibrations of the α,β-unsaturated ketone group. Peaks appearing between 1600–1625 cm^{-1} were assigned to C=C stretching vibrations of the conjugated double bond system, while absorption bands in the range of 3020–3080 cm^{-1} indicated aromatic C–H stretching vibrations. The absence of aldehydic C–H stretching peaks confirmed the complete conversion of benzaldehyde into the desired DBA product.

NMR Analysis

The ^1H NMR spectra of the synthesized DBA derivatives exhibited characteristic signals corresponding to aromatic and olefinic protons. Signals observed in the range of δ 7.2–7.8 ppm were assigned to aromatic protons, whereas peaks between δ 7.5–7.7 ppm corresponded to olefinic protons of the conjugated α,β-unsaturated system. The observed spectral signals confirmed the formation of conjugated DBA structures, while the absence of aliphatic proton signals indicated high purity of the synthesized compounds.

Discussion

The synthesized Co_3O_4 nanoparticles played an important catalytic role in the Claisen–Schmidt condensation reaction due to their high surface area, active catalytic sites, enhanced reaction kinetics, and improved selectivity. Compared with conventional catalysts, cobalt oxide nanoparticles exhibited several advantages including shorter reaction time, higher product yield, better selectivity, lower environmental impact, and excellent catalyst reusability. While conventional catalysts generally provide yields in the range of 60–80% with longer reaction times and poor recyclability, Co_3O_4 nanoparticles achieved higher yields of about 80–90% under milder and environmentally friendly reaction conditions.

Conclusion

The present study successfully demonstrated the green synthesis of cobalt oxide nanoparticles using tamarind leaf extract through an eco-friendly and sustainable approach. Characterization studies using XRD, SEM, and UV–Visible spectroscopy confirmed the formation of nanoscale crystalline Co_3O_4 nanoparticles with good structural properties. The synthesized nanoparticles acted as efficient heterogeneous catalysts in the synthesis of dibenzylidene acetone derivatives, providing high yields and reduced reaction times under mild conditions. Spectroscopic characterization further confirmed the successful formation of DBA derivatives. Overall, the study highlights the importance of nanotechnology and green chemistry in sustainable organic synthesis and environmentally benign catalytic applications.

Future Scope

Future research in this field may focus on photocatalytic applications of cobalt oxide nanoparticles, large-scale industrial synthesis, detailed catalyst recyclability studies, and biomedical applications of synthesized nanomaterials. Further investigations may also explore the development of advanced nanocomposite materials with improved catalytic and functional properties for various environmental and industrial applications.

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