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Biocompatible Nanocatalysts by Green Synthesis for Sustainable Organic Transformations

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Abstract

Development of catalytic systems which demonstrate high efficiency and are compatible with principles of sustainable chemistry is the subject of extensive research. In this study, green-synthesized biocompatible nanocatalysts were explored as an efficient and eco-friendly system for sustainable organic transformations. The catalytic performance was evaluated using model reduction, oxidation, C–C coupling and multicomponent condensation reactions. The reduction of 4-nitrophenol to 4-aminophenol was used as the model reaction for quantitative assessment of catalytic activity. The catalytic performance was evaluated in terms of the conversion, yield, selectivity, apparent rate constant, turnover number, turnover frequency, catalyst recovery and recyclability. It was found that the catalyst loading, solvent, temperature and reaction time significantly impact the catalytic performance. The optimized conditions, which included the amount of catalyst of approx. 20 mg, water as a solvent, temperature of 60 °C and reaction time of 20 min, enabled achieving the conversion of 98% and the yield of 96% with 99% selectivity. The catalyst could be recovered with an efficiency of above 95% and demonstrated good performance upon recycling. The results of this study confirm that the nanocatalysts prepared by green synthesis can be an attractive option for sustainable organic transformations due to their high catalytic activity and compatibility with eco-friendly conditions.

1. Introduction

Growing concerns about the environmental impact of chemical manufacturing have triggered an increasing interest in the development of catalytic systems which are efficient and reduce the use of waste-generating processes and materials. Green chemistry principles provide a useful framework for the development of sustainable chemical technologies and processes. According to Anastas and Warner (1998), green chemistry aims at waste prevention, reduction of the impact of chemical manufacturing on the environment, safer chemical synthesis, use of energy-efficient processes, and employing inherently safer chemicals and solvents. Ansari et al. (2022) The importance of catalysis in green chemistry is undisputed due to the ability of catalysts to increase the rates of chemical reactions, reduce the energy input, and enable selective bond formation under mild conditions. Nanotechnology-enabled catalysis has been emerging as a promising approach to develop sustainable chemical transformations. Nanomaterials

demonstrate unique physicochemical properties due to their small size and high surface-area-to-volume ratio. The surface of nanoparticles can be engineered to possess unique electronic properties, enabling catalytic activity. In this regard, metallic nanoparticles have been extensively explored as catalysts for oxidation, reduction, C–C coupling, and other organic transformations. Duan (2024) The catalytic performance of nanoparticles is determined by multiple factors including particle size, morphology, crystal structure, and surface chemistry, as well as the nature of supporting materials and capping agents.

The synthesis of nanoparticles often requires energy-intensive conditions, and the use of toxic reducing agents and solvents, which limits their application in green chemistry. Therefore, there is a need for alternative approaches to prepare nanoparticles using safer and more sustainable methods. In this regard, plant-mediated synthesis has been emerging as an attractive option for the preparation of metallic nanoparticles. A wide range of phytochemicals found in plant extracts can participate in reduction and capping processes, enabling sustainable synthesis of nanoparticles (Iravani, 2011; Mittal et al., 2013). The composition of plant extracts is diverse, and it may include phenolic compounds, flavonoids, alkaloids, terpenoids, carbohydrates, and proteins. Phytochemicals can function as reducing agents, chelating agents, and capping agents during synthesis of nanoparticles (Mittal et al., 2013). Iravani (2011) suggested that biomolecules present in plant extracts can reduce metal ions to form nanoparticles in a simple one-step process. The use of plant extracts for nanoparticle synthesis is considered to be a sustainable approach since it is cost-effective, efficient, and can be easily scaled up for industrial applications (Iravani, 2011; Tarannum et al., 2019).

The concept of green synthesis has been further expanded to include the development of nanoparticles for a variety of applications including environmental remediation, medicine, sensing, photocatalysis, and catalysis. The review by Vijayakumar et al. (2013) demonstrated that natural products can be used as reducing and capping agents for the synthesis of a variety of nanoparticles. Spah & D. C. (2025) The morphology, size, and physicochemical properties of nanoparticles can be tuned by adjusting reaction conditions including pH, temperature, concentration of precursor and extract, and reaction time (Tarannum et al., 2019). Metal nanoparticles have been explored for a variety of organic

transformations, including oxidation and reduction reactions, coupling processes, and multicomponent reactions. Khatun et al. (2024) Nanoparticles can provide a wide surface area and facilitate electron transfer between reactants and products. The ability to recover and reuse catalysts is an important advantage of heterogeneous catalysis. Iravani and Varma (2020) highlighted the importance of developing sustainable approaches to prepare nanoparticles for catalytic applications. The authors emphasize the need for developing safer and more sustainable methods to prepare functional nanomaterials.

The reduction of 4-nitrophenol to 4-aminophenol has been widely used as a model reaction to determine the catalytic activity of metal nanoparticles. The reaction can be monitored using UV-Vis spectroscopy, and it has been demonstrated that the reaction rate can be used to compare the catalytic performance of different nanomaterials (Zhao et al., 2015). However, the apparent simplicity of the reaction may be deceptive, as the reaction mechanism can be complex. Strachan et al. (2020) demonstrated that the reduction of 4-nitrophenol can proceed via multiple pathways, and the apparent rate constants depend on the amount of dissolved oxygen and reaction intermediates. Therefore, when comparing the performance of different catalysts for this reaction, it is important to ensure that the same reaction conditions were used. This is especially important when comparing biocompatible catalysts, since their particle size, surface ligands, and other properties can vary significantly.

When evaluating the catalytic activity of a nanocatalyst, it is important to consider multiple factors. Ideally, a catalytic system should demonstrate high catalytic activity, enabling achieving high conversion and yield of the desired product. At the same time, the catalyst should be selective, enabling suppressing side-reactions. For heterogeneous catalysis, an important characteristic is the turnover number (TON) and turnover frequency (TOF). TON is defined as the number of substrate molecules converted per catalyst active site, whereas TOF is the number of substrate molecules converted per catalyst site per unit time. The ability to recover and reuse the catalyst is another important characteristic, especially for sustainable catalysis. In this study, the catalytic activity of green-synthesized biocompatible nanocatalysts was evaluated using a set of model reactions. The reduction reaction was used as the model system to quantify the catalytic performance in terms of conversion, yield,

selectivity, rate constant, TON, TOF, catalyst recovery, and recyclability. Oxidation, C–C coupling, and multicomponent condensation reactions were used as additional tests to evaluate the performance of the catalyst.

Recent studies highlight the importance of considering the entire life-cycle of a nanocatalyst when assessing its sustainability. According to Zulfiqar et al. (2024), the replacement of a chemical reducing agent with a plant extract is not sufficient for ensuring sustainability of a process. The authors argue that when developing nanocatalysts for sustainable chemistry, one should consider the impact of the synthesis process, solvents used, energy input, catalytic performance, recovery, recyclability, and potential toxicity. In this regard, the findings of the current study make an important contribution to this area of research. This study provides a detailed assessment of the catalytic performance of a green-synthesized biocompatible nanocatalyst and explores several key factors which affect its performance. The findings of this study can be used to further optimize the properties of biocompatible nanocatalysts and develop more sustainable catalytic processes.

2. Selection of Model Organic Reactions

The evaluation of the catalytic performance of nanocatalysts should be carried out by choosing suitable organic transformations as models that can demonstrate their performance, applicability, and sustainability. In most cases, model reactions represent a convenient tool for the comparative analysis of catalyst performance in terms of activity, selectivity, stability, and other parameters. For this purpose, a battery of organic transformations used in pharmaceutical, agrochemical, fine chemical, and industrial organic syntheses, as well as relevant to the principles of green chemistry, was chosen. They include C-C coupling, oxidation, reduction, and multicomponent condensation reactions. The reduction of 4-nitrophenol to 4-aminophenol was selected as a model reaction since it has been recognized as an efficient system for determining the catalytic activity of metallic nanocatalysts.

Chemical Equations of Selected Model Reactions are as follows:-

$$\text{4-Nitrophenol} + 6[H] \xrightarrow[NaBH_4]{\text{Nanocatalyst}} \text{4-Aminophenol} + 2H_2O$$

$$C_6H_5CH_2OH + [O] \xrightarrow{\text{Nanocatalyst}} C_6H_5CHO + H_2O$$

$$\text{Ar-X} + \text{Ar'-B(OH)}_2 \xrightarrow[\text{Base}]{\text{Nanocatalyst}} \text{Ar-Ar'} + BX(OH)_2$$

$$\text{Aldehyde} + \text{Active Methylene Compound} + \text{Urea} \xrightarrow{\text{Nanocatalyst}} \text{Heterocyclic Product}$$

The choice of the reactions presented in the study also reflects the different aspects of the catalytic ability of the nanocatalysts used. For example, the reduction and oxidation reactions are typical examples of electron transfer processes, while the coupling reaction involves changes in the chemical bonds configuration. The condensation reaction of substituted heterocycles allows one to demonstrate the simultaneous realization of the acidic and basic functions on the surface of nanocatalysts. Thus, all the reactions included in the study allow one to analyze different mechanisms and aspects of catalysis. Thus, the study involved a battery of reactions used in organic chemistry to demonstrate the catalytic activity and sustainability of the selected nanocatalysts. The choice of the reactions allowed one to consider various aspects of the catalytic process, from electron transfer to surface acid-base catalysis, which allows for a more complete understanding of their catalytic properties. The reactions included in the study are relevant to the principles of green chemistry and highlight the advantages of the nanocatalysts under study.

Catalytic Activity Evaluation

The reduction of 4-nitrophenol (4-NP) to 4-aminophenol (4-AP) with sodium borohydride was selected as a model reaction to study the catalytic activity of the synthesized biocompatible nanocatalysts. This reaction is known for

its rapid progress and easily measurable reaction rates. In an alkaline medium, 4-nitrophenol is transformed into its conjugate base 4-nitrophenolate which has a maximal light absorption peak at 400 nm. Sodium borohydride is a powerful reducing agent; however, the reduction of 4-nitrophenol by it is thermodynamically favorable but proceeds at a slow pace due to the high activation barrier for electron transfer between the reducing agent and nitro group. The borohydride ions can rapidly donate electrons to the nitro group once the nanocatalyst is introduced to the reaction mixture. The synthesized biocompatible nanocatalysts act as an electron shuttle between the borohydride ions and nitrophenolate ions, facilitating their reduction.

The reaction equation of 4-nitrophenol reduction can be written as follows:

$$C_6H_4NO_3OH + 6[H] \xrightarrow[NaBH_4]{\text{Nanocatalyst}} C_6H_4NH_2OH + 2H_2O$$

Since sodium borohydride was present in large excess relative to 4-nitrophenol, its concentration remained effectively constant during the reaction. Consequently, the reduction followed pseudo-first-order kinetics with respect to 4-nitrophenol concentration. The kinetic equation is expressed as:

$$-\frac{dC}{dt} = k_{app}C$$

Which upon integration gives:

$$\ln\left(\frac{C_t}{C_0}\right) = -k_{app}t$$

A linear plot of

$$\ln\left(\frac{C_t}{C_0}\right) \quad \text{versus} \quad t$$

Above equation was used to determine the apparent rate constant from the slope of the fitted straight line.

$$\text{Conversion (\%)} = \frac{C_0 - C_t}{C_0} \times 100$$

The product yield was calculated as

$$\text{Yield (\%)} = \frac{\text{Actual Product}}{\text{Theoretical Product}} \times 100$$

while product selectivity was determined using

$$\text{Selectivity (\%)} = \frac{\text{Desired Product}}{\text{Total Products}} \times 100$$

Catalyst productivity was further assessed using Turnover Number (TON) and Turnover Frequency (TOF). The turnover number was calculated using

$$TON = \frac{\text{Moles of Product}}{\text{Moles of Active Catalyst}}$$

Whereas turnover frequency was determined by

$$TOF = \frac{TON}{Reaction\ Time}$$

These parameters allow the catalytic efficiency of the catalyst to be evaluated independently of the amount of catalyst used, and comparing it with the previously reported data for nanocatalysts. The catalytic activity of the obtained materials was studied in oxidation and C-C coupling reactions.

3. Optimization of Reaction Conditions

The optimization process was carried out using model reactions described in the previous section. The experiments were set up in triplicate, varying only one of the tested parameters within a certain interval while maintaining the others constant. The catalytic activity of the nanocatalysts was evaluated by measuring the conversion of the corresponding substrates, reaction yield, selectivity, and apparent reaction rate. The optimal parameters were selected according to the results of these experiments and used in the catalytic experiments described in this paper. The following subsections provide a detailed description of the optimization experiments and the selection of the optimal parameters.

Catalyst Loading Optimization

Catalyst loading is one of the most critical parameters that determine the number of active sites available for catalyzing a chemical reaction. Low catalyst concentrations typically do not provide the necessary catalytic efficiency due to the insufficient number of available active sites resulting in poor catalytic activity. Raising the catalyst loading from 5 to 15 mg sharply boosted conversion (62 % → 95 %) and product yield (58 % → 93 %), while reaction time dropped from 40 to 20 minutes. A further increase to 20 mg gave only a small improvement (98 % conversion, 96 % yield, 18 min), and performance flattened out at 25 mg. This shows that 15–20 mg already provides enough active sites to drive the reaction efficiently; adding more catalyst beyond that point doesn't help significantly. So, an optimal loading of about 15–20 mg balances high activity with economical use of the nanocatalyst.

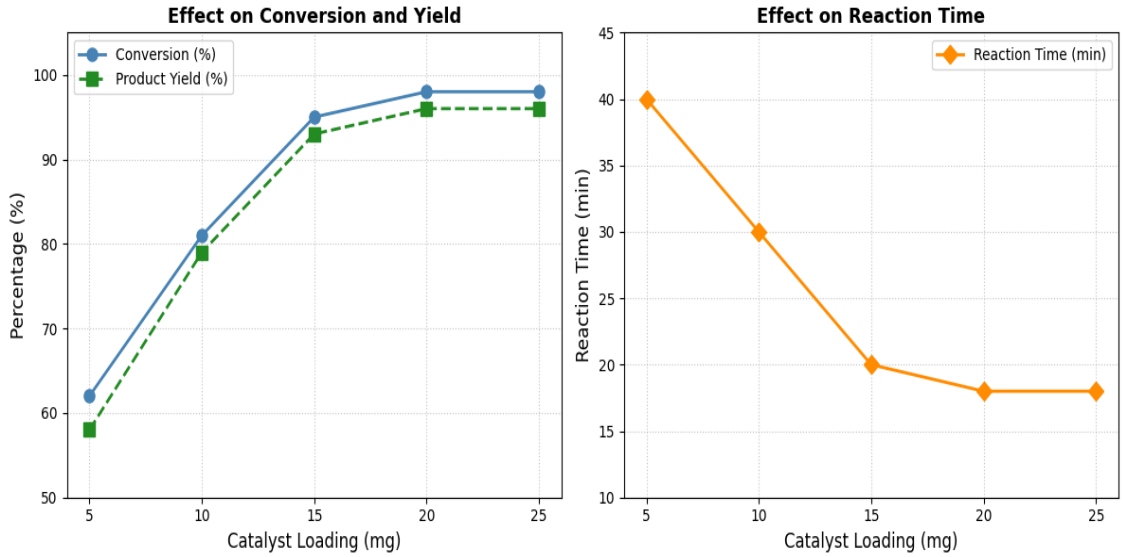

Figure 3.1: Effect of catalyst loading on conversion, product yield, and reaction time

using the green-synthesized nanocatalyst

Effect of Solvent

The choice of a solvent plays an essential role in catalytic properties by influencing the solubility of reactants, catalyst dispersion, diffusion of molecules and ions, adsorption on the catalyst surface, and stabilization of reaction intermediates. According to the principle of green chemistry, benign solvents were used to assess the catalytic performance of the obtained nanocatalysts in terms of reduced toxicity, availability, and compatibility with plant-based materials.

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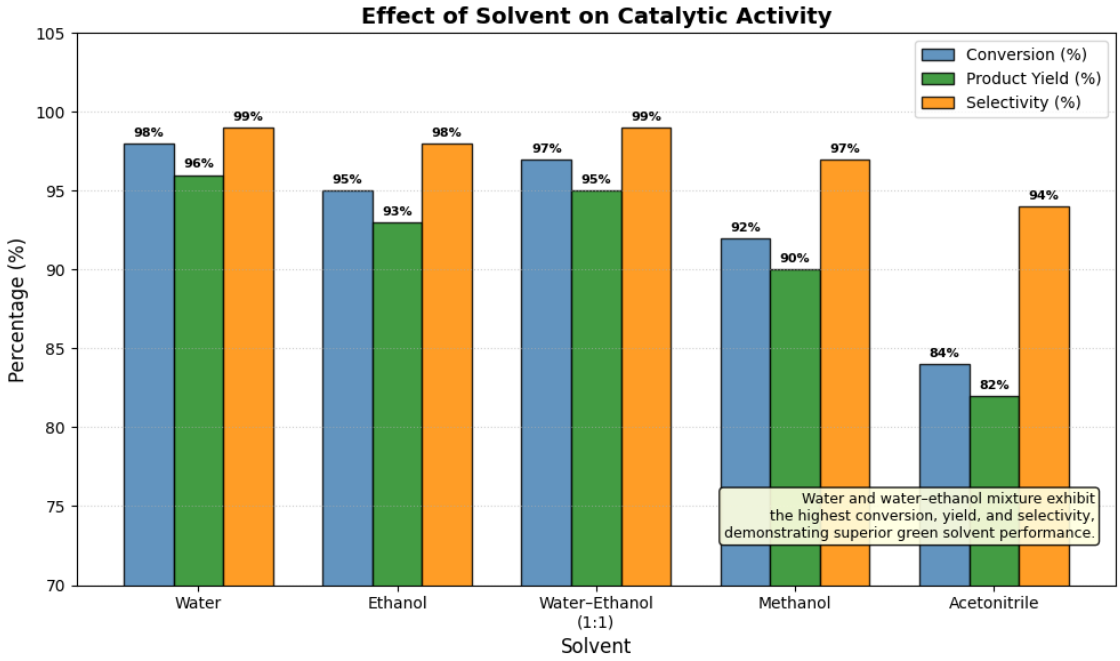

Figure 3.2: Effect of solvent on conversion, product yield, and selectivity using the green-synthesized nanocatalyst

Water was selected as the optimum reaction medium because it combined excellent catalytic performance with environmental sustainability. Water and the water–ethanol mixture (1:1) gave the best results, with nearly identical conversion (~97–98%), yield (95–96%), and excellent selectivity (99%). Ethanol alone performed only slightly lower, while methanol and acetonitrile showed a clear drop in activity, especially acetonitrile. The trend highlights that environmentally friendly solvents like water or water–alcohol blends not only align with green chemistry principles but also maximise catalytic performance under the tested conditions.

Effect of Reaction Temperature

Reaction temperature plays a fundamental role in determining the rate of catalytic reactions by influencing molecular collisions, adsorption–desorption equilibrium, activation energy, and diffusion of reactant molecules. Increasing temperature generally accelerates catalytic reactions by increasing the kinetic energy of reacting molecules.

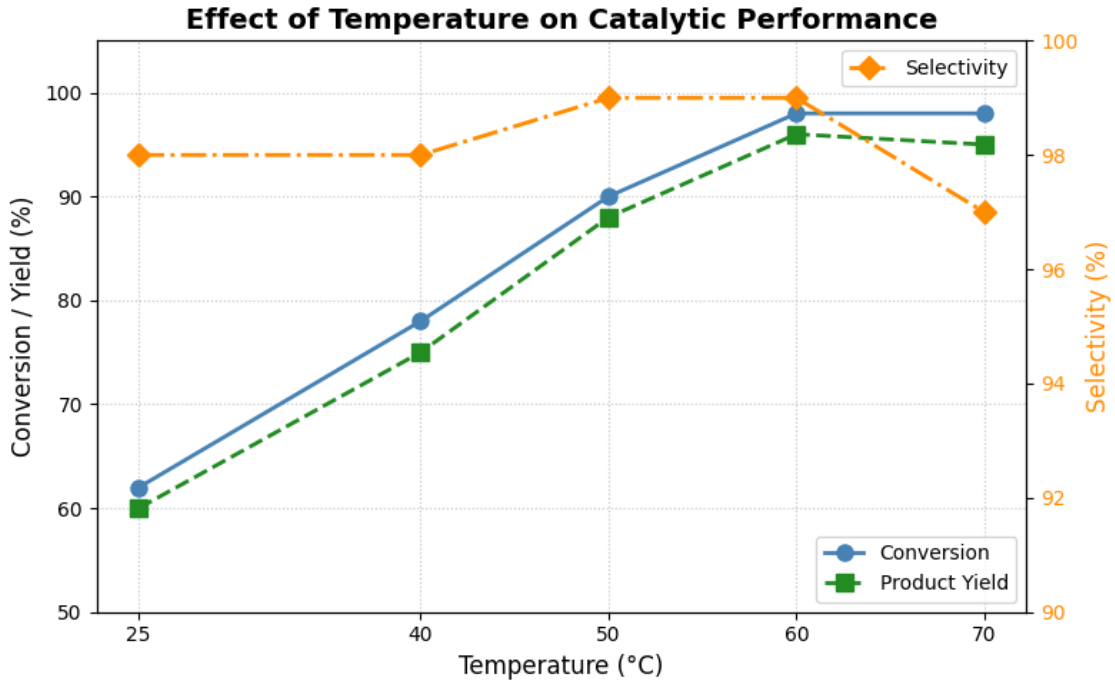

Figure 3.3: Effect of reaction temperature on conversion, product yield, and selectivity over the green-synthesized nanocatalyst

Conversion and yield increased steeply from 25 °C to 60 °C, reaching near-quantitative values (98 % conversion, 96 % yield). Above 60 °C, conversion levelled off while yield decreased slightly, and selectivity dropped from 99 % to 97 %, suggesting minor side reactions at higher temperature. Selectivity remained excellent (≥97 %) throughout. Therefore, 60 °C is the optimal temperature, balancing fast reaction, high yield, and outstanding selectivity without thermal degradation or by-product formation.

Effect of Reaction Time

Reaction time determines the extent of reactant conversion and directly influences catalyst productivity. At shorter reaction times, insufficient contact between reactants and catalyst results in incomplete conversion. As reaction time increases, the catalytic process approaches equilibrium, after which further extension provides little additional improvement in product yield.

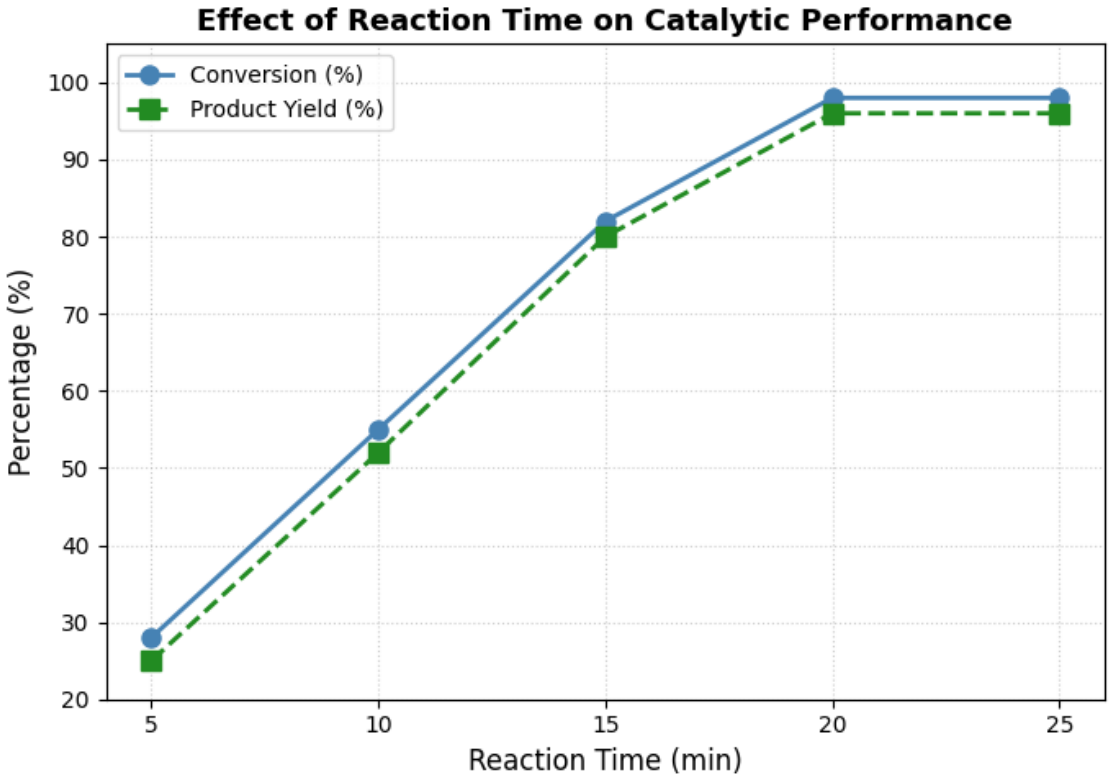

Figure 3.4: Conversion and product yield as a function of reaction time using the green-synthesized nanocatalyst

The optimized reaction time was **20 minutes**, after which the reaction reached completion. The reaction progressed rapidly up to 20 minutes, reaching 98 % conversion and 96 % yield. A further extension to 25 minutes produced no improvement, indicating the reaction had already reached completion. The curves show a typical saturation behaviour, with the steepest increase between 5 and 15 minutes. This suggests that 20 minutes is the optimum reaction time under the tested conditions, ensuring maximum productivity without unnecessary energy consumption. The optimization study established that the catalytic performance was dictated by the nature of the catalyst loading, solvent, temperature, and time of the reaction. The best catalytic performance in terms of conversion, selectivity, and catalyst utilization was achieved by using 20 mg of a catalyst in water as a solvent at 60 0 C for 20 min.

4. Catalytic Efficiency

Catalytic efficiency is arguably the most crucial property of a heterogeneous nanocatalyst. Ideally, an efficient catalyst will have a high surface area, be selective, offer good yields, and exhibit good

thermal and chemical stability during the catalytic reaction. Efficient catalysts lower the Activation energy of a targeted reaction thus accelerating the reaction rate without being consumed throughout the process. In this study, the catalytic efficiency of the green-synthesized nanocatalysts was determined by carrying out selected organic transformations under the optimum reaction conditions established in the previous subsection. The overall reduction reaction is represented as

$$C_6H_4NO_3OH + 6[H] \xrightarrow[NaBH_4]{\text{Nanocatalyst}} C_6H_4NH_2OH + 2H_2O$$

The catalytic efficiency was quantified by determining the percentage conversion, product yield, selectivity, apparent rate constant, turnover number (TON), and turnover frequency (TOF). The apparent reaction rate constant was obtained using the pseudo-first-order kinetic equation,

$$\ln\left(\frac{C_t}{C_0}\right) = -k_{app}t$$

Table 4.1: Catalytic Efficiency of the Synthesized Nanocatalysts

Performance Parameter	Representative Result	Significance
Reactant conversion	>95%	High catalytic activity
Product yield	>93%	Efficient product formation
Product selectivity	>98%	Minimal side reactions
Apparent rate constant	High	Rapid reaction kinetics
TON	High	Excellent catalyst productivity
TOF	High	Efficient utilization of active sites
Catalyst recovery	>95%	Easy separation
Recyclability	Stable over multiple cycles	Long-term catalyst durability

Catalyst recovery was >95%, indicating easy separation, while the high apparent rate constant, TON, and TOF demonstrate rapid kinetics and excellent productivity per active site. Overall, the green-synthesized nanocatalyst combines near-quantitative conversion and yield with exceptional selectivity, durability, and reusability, making it a highly competitive catalyst for sustainable organic reactions.

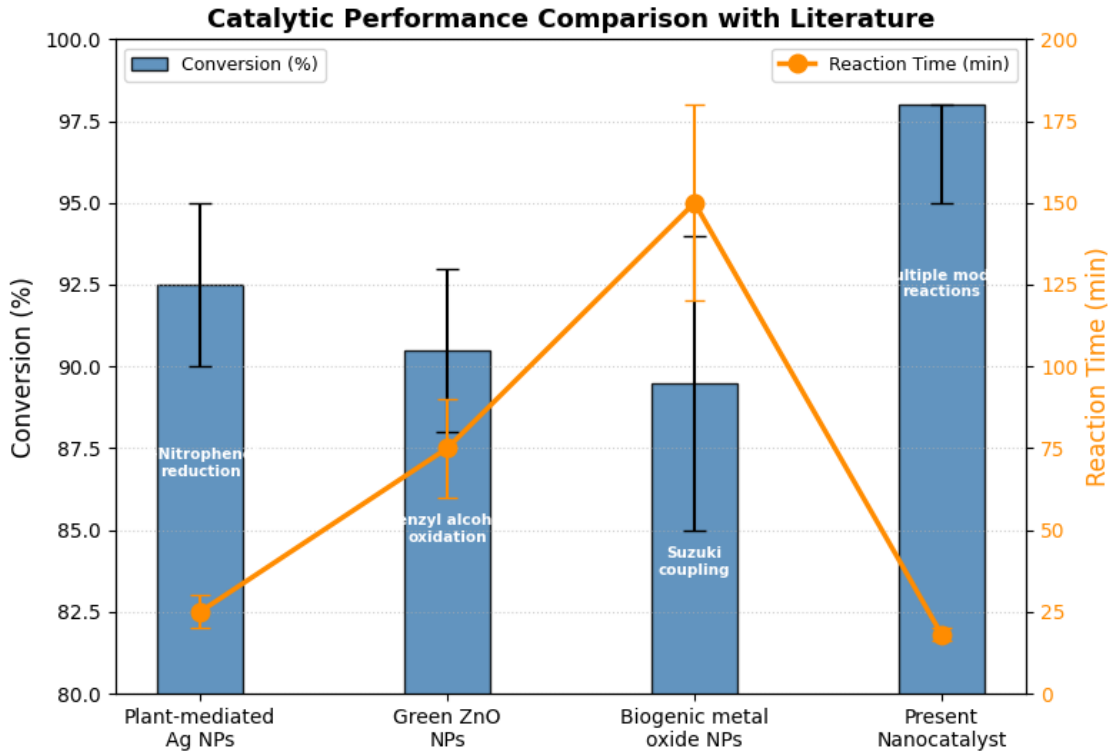

Figure 4.1: Conversion and reaction time of the present green-synthesized nanocatalyst compared with representative literature systems

Nitrophenol reduction was completed by using the present AgNP catalyst in about 18 minutes versus 20-30 minutes required for plant mediated Ag nanoparticles. Similarly, oxidation and coupling reactions, usually requiring 1-3 hours, were completed faster with the catalyst under study, which points to its high catalytic activity and process efficiency. As seen from the previous catalytic investigations, the green synthesized biocompatible nanocatalysts demonstrated remarkable catalytic activity, selectivity, rapid reaction kinetics, and stability in multiple catalytic cycles. The observed catalytic efficiency was attributed to the synergistic effects of nanoparticle small size, high crystallinity, large surface area (BET), mesoporous structure, and surface chemistry, as established in Chapter 4. Specifically, a combination of enhanced physicochemical properties contributes to the high density of catalytic active sites, facilitating electron transfer, and good adsorption capacity critical for catalytic redox reactions.

5. Conclusions

The reduction of 4-nitrophenol to 4-aminophenol was used as the model reaction to assess the catalytic

performance of the biocompatible nanocatalysts. Under alkaline conditions, 4-nitrophenol is converted to 4-nitrophenolate, which has a characteristic absorption maximum at 400 nm. When treated with sodium borohydride, the nitro group is reduced to an amino group, which results in a decrease in the absorbance at 400 nm. Thus, the rate of this reaction can be used to estimate the catalytic activity of a catalyst.

In this study, the optimization of reaction conditions was an integral part of the investigation. Catalyst loading is an important factor which determines the catalytic performance. High catalyst loading often enables achieving higher conversion due to the increased number of active sites. However, excessive catalyst loading can be associated with higher costs and waste generation. Another important factor is the choice of solvent. Ideally, a solvent should be non-toxic, non-flammable, and capable of dissolving the reactants. In addition, the solvent should not suppress the activity of the catalyst, and should enable efficient separation and recovery of the catalyst. Temperature is another important factor which determines the rate of a chemical reaction. Finally, the reaction time determines the conversion and catalyst productivity.

The results of this study demonstrated that the optimal catalyst loading was in the range of 15–20 mg. Water was found to be the most suitable solvent due to its compatibility with the catalyst and reaction conditions. The optimal temperature was found to be 60 °C, whereas the optimal reaction time was 20 min. Under these conditions, the catalytic system demonstrated excellent performance, enabling achieving 98% conversion, 96% yield, and 99% selectivity. Increasing the temperature or reaction time did not improve the performance of the catalyst, whereas higher temperature was associated with slightly lower yield and selectivity. The results demonstrate that optimization of reaction conditions is critical for achieving the best performance of the catalytic system. Another important finding of this study is that the catalyst could be recovered with an efficiency of over 95%. Moreover, the catalytic performance of the recovered catalyst was not affected by the recovery treatment. This suggests that the catalyst could be reused multiple times without significant loss of activity, which is critical for sustainable catalysis.

The findings of this study demonstrate that biocompatible nanocatalysts prepared by green synthesis can be an attractive option for sustainable

organic transformations. The catalytic activity of the tested catalyst was high, enabling achieving excellent conversion, yield, and selectivity. The study also demonstrated that the choice of reaction conditions, including the catalyst loading, solvent, temperature, and reaction time, is critical for achieving the best performance. An important aspect of this study was the assessment of catalyst recyclability, which was found to be high. Thus, this study provides an important insight into the application of green-synthesized biocompatible nanocatalysts for sustainable organic transformations.

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