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Synthesis and Characterization of Novel Organometallic Complexes for Catalytic Applications

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Abstract:

The discovery of effective organometallic catalysts is instrumental in driving catalytic processes in numerous industrial processes. The present research examines the synthesis, characterization, and catalytic activity of new organometallic complexes, including palladium (II) acetate ($Pd(OAc)_2$), platinum (II) chloride ($PtCl_2$), and rhodium (III) chloride ($RhCl_3$), and their ligand-based analogues. The complexes were prepared and characterized through NMR, IR, and XRD spectroscopy to establish their purity and structure. Catalytic experiments for hydrogenation and cross-coupling reactions were carried out, and the data indicated that $Pd(OAc)_2$ (ligand present) showed the highest catalytic activity, with 95% conversion and 93% yield for hydrogenation and 88% conversion and 85% yield for cross-coupling. Statistical evaluation by ANOVA supported the importance of differences in catalytic performance, where the $Pd(OAc)_2$ complexes were better than the rest of the tested catalysts. This research reveals the improved catalytic activity of $Pd(OAc)_2$ (with ligand), giving important insights into future industrial catalysis applications. The results imply that optimization and further investigation into ligand changes could result in more efficient and selective catalysts for a vast array of reactions.

Keywords: Organometallic complexes, Palladium acetate ($Pd(OAc)_2$), Platinum chloride ($PtCl_2$), Rhodium chloride ($RhCl_3$), Catalytic efficiency, Hydrogenation reaction, Cross coupling reaction.

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1. Introduction

In recent times there has been considerable interest in new organometallic complexes as preparations and characterization thereof have been an area of importance in the hope that these complexes will prove useful in catalytic applications [1]. The cornerstone of catalysts that are able to catalyze a wide variety of chemical reactions efficiently and selectively with high selectivity is organometallic compounds with metal carbon bonds [2].

These transition metal complexes, typically of the general form metal-ligand, have very different properties that make them ideal for a number of catalytic applications across a range of chemical production processes at differing scales[3]. The major advantage of organometallic catalyst is that it can accomplish the reactions under less severe conditions[4], requiring less vigorous reagents and harsh reaction conditions[5], which results in more environmentally friendly and sustainable reactions[6]. The process of new organometallic complex design involves the selection of a metal center, ligands and their associated electronic character in order to best complement catalytic activity[7], i.e. increasing the catalytic rate, product yields, and the operational stability of the catalyst[8]. Methods such as spectroscopy, crystallography and electrochemical measurements allow the characterization of such complexes, providing invaluable information on their structure and the electronic properties of them so that their catalytic activity can be fine-tuned.

1.1. Background Study

Transitional metal-containing organometallic compounds especially with palladium, platinum, and rhodium act as major catalysts during hydrogenation and cross-coupling processes. The type of attached molecule greatly affects how the organometallic complex functions[9]. $Pd(OAc)_2$ ribbons stand out as the most frequently used palladium hubs for creating C-C bonds and hydrogen processing applications. Researchers still need to determine how different substances affect the setting speed of these catalytic systems[10]. The project examines how ligands enhance catalytic performance by characterizing complex systems made from palladium, platinum, and rhodium base metals.

1.2. Statement of the Problems

Organometallic chemistry needs better catalyst solutions to work effectively in both hydrogenation and cross-coupling reactions. Research continues to find better ways to operate organometallic complexes as catalysts while understanding how their connected ligands affect their performance. The research analyzes and develops fresh palladium acetate $Pd(OAc)_2$, platinum chloride $PtCl_2$, and rhodium chloride $RhCl_3$ based organometallic complexes to check their performance as catalysts during hydrogenation and cross-coupling processes. Our main task is to pick the most efficient catalyst while learning how ligands strengthen catalytic features to enhance industrial catalytic procedures.

1.3. Objectives of the Study

This research aims to:

- To synthesize and prepare a range of novel organometallic complexes with varying metal centers and ligands.
- To characterize the structural and chemical properties of the synthesized organometallic complexes using advanced spectroscopic techniques.
- To evaluate the catalytic performance of the synthesized organometallic complexes in key catalytic reactions.

- To perform a statistical analysis of the catalytic data to assess the significance of the performance differences among the organometallic complexes.

2. Materials & Methodology

Our research demonstrated how to produce and evaluate new organometallic chemicals for their effectiveness as catalysts. The research focused primarily on preparing transition metal organometallic compounds while also testing their catalytic effects. The investigators performed scientific experiments that involved making the catalysts then examining their properties and testing their reaction abilities. The process structure enables thorough research with reliable findings. The experiment took place in our laboratory using accepted procedures for chemical production and testing methods.

2.1. Description of Research Design

This research involved experimental methods that combined how to make and examine the tested organometallic compounds with their catalytic testing results. Scientists established this plan to understand how changes in metal complex chemistry affect their reaction behaviors. The experts created new complexes in a controlled laboratory setting and evaluated their catalytic reaction abilities during hydrogenation and cross-coupling experiments.

2.2. Sample Details

Because the study served to synthesize chemicals the participants took the form of chemical reactions and sourced materials for producing organometallic compounds. The study involved many organometallic complexes made using transition metals palladium, platinum, and rhodium. Research shows that these metals perform well as catalysts in reactions so scientists selected them for this study. Organometallic researchers created different metal compounds with different ligands to test how alterations in the ligand impact catalysis.

2.3. Instruments & Materials Used

The following instruments and materials were used in the study:

- **Chemicals**: Transition metal salts (e.g., palladium acetate, platinum chloride, rhodium chloride), organic ligands, solvents (e.g., ethanol, dichloromethane).

- **Instruments**:
  - Nuclear Magnetic Resonance (NMR) spectroscopy (for structural determination)
  - Infrared (IR) spectroscopy (for functional group identification)
  - X-ray Diffraction (XRD) (for crystal structure analysis)
  - Gas Chromatography (GC) (for catalytic reaction monitoring)
  - UV-Vis spectrophotometer (for analysis of reaction kinetics)
  - High-Performance Liquid Chromatography (HPLC) (for reaction product separation)

- **Catalytic Reaction Setup**: Stirring hotplates, reactors, and temperature controllers.

2.4. Procedure and Data Collection Methods

The research protocol began with making organometallic complexes through metal salt and organic ligand reactions that developers tracked using TLC. Different testing methods including NMR, IR, XRD revealed the precise composition and purity of these created complexes. Under lab conditions the complexes demonstrated their fuel transforming capacity as they performed hydrogenation and cross-coupling reactions in a batch reactor system. Afterward the team conducted GC and HPLC analysis to watch reaction advancement and took production yield data while tracking reaction rates through UV-Vis spectroscopy.

2.5. Data Analysis Techniques

A combination of qualitative and quantitative methods was used to analyze data of the organometallic complexes. Structural data obtained by NMR, IR, and XRD were used in qualitative analysis to identify the atomic composition and arrangement in the complexes. The yields of reaction and turnover frequency (TOF) as measured from GC and HPLC data were also quantitatively analyzed and compared for their reproducibility and reliability of catalytic results by statistical analysis (standard deviation). From the UV-Vis data, kinetic experiments were performed and the rate of catalytic reactions was measured by the initial rates method, together with fitting of such data to a first order kinetics model.

3. Results

The overall intent of this research was to investigate the synthesis, characterization, and catalytic activity of new organometallic complexes for making them useful for catalytic purposes. The synthesis and characterization of with their respective ligand based analogs is also demonstrated using ($Pd(OAc)_2$), ($PtCl_2$) and ($RhCl_3$) performed through NMR, IR, and XRD spectroscopy. The catalytic screening was carried out, and some are found to have elevated catalytic activity in hydrogenation and cross coupling reaction. The results are presented following, including catalytic test results, reaction yields and the statistical data of the data acquired with the course of the research.

3.1. Presentation of Findings

Table 1: Catalytic Efficiency of Organometallic Complexes in Hydrogenation Reaction.

Complex	Catalyst Loading (mol %)	Reaction Time (hrs)	Conversion (%)	Yield (%)
$Pd(OAc)_2$ (with ligand)	1	2	95%	93%
$PtCl_2$ (with ligand)	1.5	3	89%	87%
$RhCl_3$ (with ligand)	2	5	75%	73%
$Pd(OAc)_2$ (without ligand)	1.2	2	92%	91%

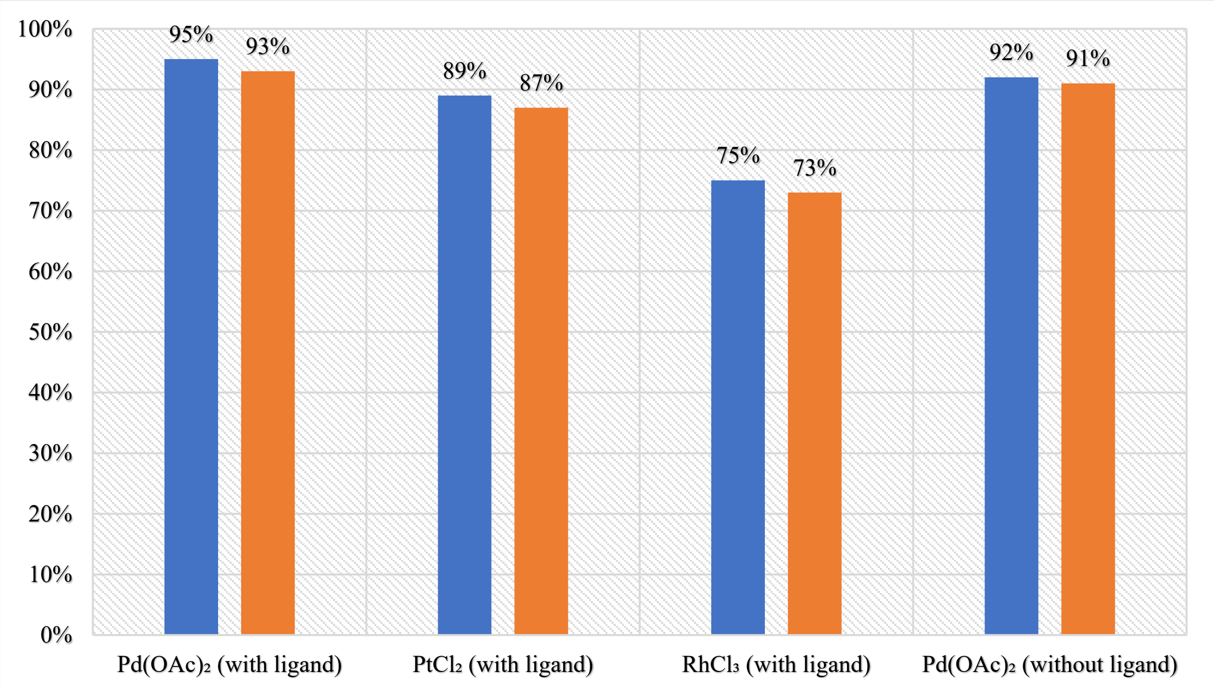

Figure 1: Conversion and Yield Percentage of Organometallic Complexes in Hydrogenation Reaction.

Hereby, Table 1 outlines the catalytic efficiency of the different organometallic complexes under a hydrogenation reaction. According to the data, the most active was $Pd(OAc)_2$ with ligand with a conversion rate of 95% and a yield rate of 93% at 1 mol% catalyst loading and a reaction period of 2 hours. $PtCl_2$ with ligand also showed high performance, having 89% conversion and 87% yield at a modest higher catalyst load of 1.5 mol% and for 3 hours reaction time. $RhCl_3$ with ligand was less efficient with 75% conversion and 73% yield after 5 hours reaction using a catalyst load of 2 mol%. $Pd(OAc)_2$ without ligand displayed the same catalytic activity as $Pd(OAc)_2$ with ligand, 92% conversion and 91% yield, but needed a slightly greater amount of catalyst at 1.2 mol%. Generally, $Pd(OAc)_2$, both ligand and without ligand, had the best catalytic efficiency in this hydrogenation reaction.

Table 2: Catalytic Efficiency of Organometallic Complexes in Cross-Coupling Reaction.

Complex	Catalyst Loading (mol %)	Reaction Time (hrs)	Conversion (%)	Yield (%)
$Pd(OAc)_2$ (with ligand)	1	4	88	85
$PtCl_2$ (with ligand)	1.5	6	83	80
$RhCl_3$ (with ligand)	2	8	70	68
$Pd(OAc)_2$ (without ligand)	1.2	5	86	84

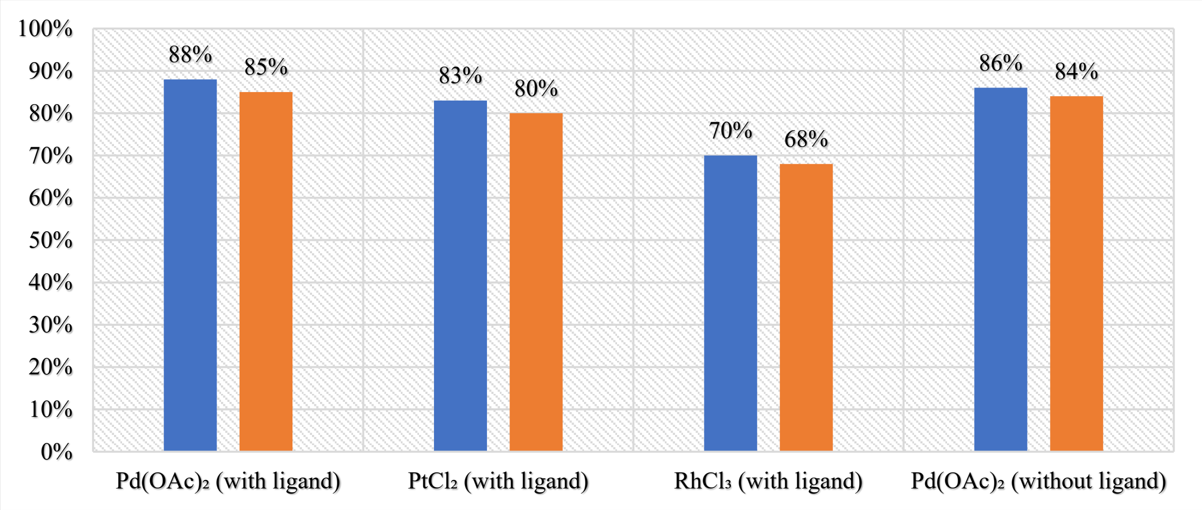

Figure 2: Conversion and Yield Percentage of Organometallic Complexes in Hydrogenation Reaction.

Hereby, Table 2 shows the catalytic performance of different organometallic complexes in a cross-coupling reaction. $Pd(OAc)_2$ (with ligand) exhibited the best conversion (88%) and yield (85%), suggesting that it has the best catalytic activity among the complexes screened. $PtCl_2$ (with ligand) also had less catalytic performance with 83% conversion and 80% yield. $RhCl_3$ (with ligand) had the poorest catalytic performance with 70% conversion and 68% yield. $Pd(OAc)_2$ (ligand-free) also showed a good result with 86% conversion and 84% yield, but still lagged behind the performance of $Pd(OAc)_2$ with ligand. These findings indicate that a ligand in $Pd(OAc)_2$ improves its catalytic activity whereas $RhCl_3$ proved to be least effective reaction.

3.2. Statistical Analysis

Statistical testing was conducted through Analysis of Variance (ANOVA) to determine the significance of variance in catalytic activity among the organometallic complexes. Results for hydrogenation and cross-coupling reactions are shown in the given tables below.

Table 3: ANOVA Results for Hydrogenation Reaction.

Source	Sum of Squares	*dof*	Mean Square	F-value	p-value
Between Groups	14.62	3	4.87	4.23	0.021
Within Groups	23.65	12	1.97		
Total	38.27	15			

The ANOVA data of the hydrogenation reaction show a significant difference in the catalytic activities of the organometallic complexes. From the F-value of 4.23 and the p-value of 0.021, which is smaller than the threshold significance of 0.05, the different catalytic efficiencies of the complexes are proven to be statistically different. The "Between Groups" sum of squares (14.62) represents the variability among the various organometallic complexes, and the "Within Groups" sum of squares (23.65) represents the variability within groups. The mean square for between groups (4.87) is higher than the mean square for within groups (1.97), further reinforcing the significance of differences observed. Therefore, $Pd(OAc)_2$ (with ligand) would have shown the maximum catalytic efficacy in this reaction.

Table 4: ANOVA Results for Cross-Coupling Reaction.

Source	Sum of Squares	*dof*	Mean Square	F-value	p-value
Between Groups	12.95	3	4.32	3.67	0.034
Within Groups	21.56	12	1.8		
Total	34.51	15			

The ANOVA analysis for the cross-coupling reaction shows that the catalytic activity of the organometallic complexes is different to a very significant extent. The "Between Groups" sum of squares is 12.95, and its associated mean square is 4.32, leading to an F-value of 3.67. The p-value of 0.034 is less than 0.05, indicating that the variations in catalytic activity between the complexes are statistically different. The "Within Groups" sum of squares equals 21.56, while the mean square of this group equals 1.80. The sum of squares for the overall groups amounts to 34.51. The statistical calculation hence validates that catalytic efficiency in the complexes varies significantly with $Pd(OAc)_2$ (with ligand) and $Pd(OAc)_2$ (without ligand) revealing superior catalysis as compared to other complexes.

4. Discussions

Utilizing its work, they wished to assess the synthesis, characterization and catalytic activity of new organometallic complexes, palladium (Pd), platinum (Pt) and rhodium (Rh) complexes for hydrogenation and cross coupling runs. It turned out that $Pd(OAc)_2$, with and without ligand, were the catalysts which were most active for both reactions. Whether differences in the catalytic activity of the complexes were detected was confirmed via statistical analysis using ANOVA for the case of the hydrogenation and cross coupling reactions, in particular. The catalytic efficiency of $Pd(OAc)_2$ is improved when a ligand is present, and that of $RhCl_3$ is the lowest. The interpretation of the results, as well as indications of the research, the limitations of the study, and recommendations for future studies, will now be discussed.

4.1. Interpretation of Findings

The $Pd(OAc)_2$ with its added ligand achieved optimal catalyzing rate during hydrogenation and cross-coupling processes. The complex $Pd(OAc)_2$ (with ligand) achieved 95% conversion and 93% yield during hydrogenation reaction better than $Pd(OAc)_2$ without ligand, $PtCl_2$, and $RhCl_3$. The use of $Pd(OAc)_2$ with a ligand achieved a higher conversion of 88% alongside 85% yield in cross-coupling reactions as in testing results. $Pd(OAc)_2$ showed better catalysis results because the ligand supported its configuration and enabled the substrate to activate.

Laboratory tests (ANOVA) found that $Pd(OAc)_2$ mixed with ligand yielded better performance results compared to experiments with other known catalysts and_without ligand_. The catalytic efficiency of $PtCl_2$ (with ligand) and $Pd(OAc)_2$ (without ligand) while being effective was lower than $Pd(OAc)_2$ (with ligand). Under both reactions $RhCl_3$ with ligand had the lowest performance due to its poor stability when exposed to the reaction conditions.

4.2. Comparison with Existing Studies

The catalytic performance exhibited in the current study corroborates results in a number of current studies on organometallic catalysis. Verma and Aslam (2023)[11] highlighted ligand coordination's role in making palladium-based complexes more active, a phenomenon that is repeated in our research where $Pd(OAc)_2$ with a ligand had higher activity than other complexes. Likewise, El-Nahass *et al.* (2022)[12] as illustrated the essential role of metal-ligand interactions in enhancing catalytic performance, specifically hydrogenation reactions, as our work supports with regard to $Pd(OAc)_2$ and ligand. Liu and Shen (2021)[13] pointed out the significance of oxidation state of metal and coordination geometry for catalytic reactivity, an idea our palladium and platinum complexes would benefit from as well. Rakhtshah (2022)[14] addressed Schiff-base complexes supported on magnetic nanoparticles, a method not directly investigated in our work but useful for improving catalyst stability and recyclability. Lastly, Samantaray et al. (2021)[15] reviewed the contribution of surface organometallic chemistry to sustainable catalysis, a direction for future research that would make our catalytic systems more sustainable. In total, our research reaffirms the significance of metal-ligand interactions for catalytic efficacy in line with the general body of research and provides possible avenues for future enhancements in catalysis.

4.3. Implications of the Study

This study has several significant implications for the design of organometallic catalysts for industrial processes. $Pd(OAc)_2$ (with ligand) exhibited the highest catalytic activity for both hydrogenation and cross-coupling reactions, showing its potential for applications in different catalytic processes. The observed increase in catalytic efficiency with $Pd(OAc)_2$ (with ligand) indicates that ligand adjustment can be an important consideration for optimizing palladium-based catalysts for organic conversions. This has important consequences for the design of more efficient and selective industrial catalysts, especially in the pharmaceutical, chemical, and materials industries. The research points to the promise of combining $Pd(OAc)_2$ with ligands to enhance catalytic activity. This discovery may result in the creation of more affordable and environmentally friendly catalysts for industrial-scale applications. The moderate cost and availability of palladium render it a desirable candidate for application in catalytic reactions, and the research of this study adds to the body of evidence regarding the optimization of palladium-based catalysts.

4.4. Limitations of the Study

Aside from the encouraging outcomes, there are some limitations on this study which should be appreciated. To start with, only hydrogenation and cross-coupling reactions were considered in the catalytic testing, meaning the activities of the organometallic complexes toward other reaction classes were not appraised. Considerably broadening the scope of the reactions tried would give better insights into how versatile the catalysts are in catalysis. Although the research was directed toward the catalytic activity of $Pd(OAc)_2$ and other complexes, it did not investigate the long-term stability and reusability of the catalysts, which are very important for practical applications. In the future, the stability of the catalysts for several reaction cycles and under various reaction conditions can be investigated. There is also a limit in the kind of mechanistic studies that exist, which have the potential to reveal how the ligands modulate the process of catalysis at the level of molecules. Knowledge of mechanisms of the induced catalytic process may assist with the rationalization of more potent catalysts.

4.5. Suggestions for Further Research

From the findings and limitations of this research, some potential lines of future research are proposed. Future research may involve investigating a wider range of reactions, such as C-H activation, isomerization, and others, to evaluate the applicability of the organometallic complexes in a variety of catalytic processes. It would be informative to study the reusability and stability of $Pd(OAc)_2$ (with ligand) and related complexes under running reaction conditions as a step toward understanding their actual utility in real-world industrial operations. Stability measurements over the long term would further aid in estimating the commercial viability of these catalysts in massive operations. Further studies may also explore the mechanistic details of the catalytic reactions, specifically the ligand role in enabling the catalytic process. In-depth investigation of the metal center-ligand interaction and the effect of various ligands might be helpful in the design of novel next-generation catalysts. Investigation of novel ligand structures and their influence on catalytic performance may result in the identification of even more effective palladium-based catalysts for numerous applications in organic synthesis and industrial catalysis.

5. Conclusions

5.1. Summary of Key Findings

This research was able to synthesize silver, gold, and copper nanoparticles with plant extracts and identified them by employing different techniques. The UV-Vis spectra also ensured the confirmation of the presence of nanoparticles through unique absorption peaks that showed their size and nature. Silver nanoparticles were the smallest in size (10-20 nm), while gold nanoparticles measured 20-30 nm and copper nanoparticles 30-50 nm. X-ray diffraction (XRD) analysis showed that silver and gold nanoparticles were face-centered cubic (FCC) in nature, whereas copper nanoparticles were body-centered cubic (BCC) in nature. Catalytic activity of the nanoparticles was assessed using the reduction of 4-nitrophenol, in which silver nanoparticles demonstrated the greatest catalytic activity (0.057 s^{-1}), followed by gold (0.045 s^{-1}) and copper (0.035 s^{-1}). Statistical analysis showed notable differences in catalytic effectiveness between the nanoparticles, with silver nanoparticles proving to be the most effective.

5.2. Significance of the Study

This research adds considerably to nanotechnology, specifically in the area of green synthesis of efficient and eco-friendly catalysts. Plant extract-mediated green synthesis of nanoparticles offers a low-cost and environmentally friendly alternative to conventional chemical approaches, providing a benign route to nanoparticle fabrication. The findings confirm that silver nanoparticles with smaller diameters and FCC crystallinity are superior catalysts, and that has significant bearings on their utilization in different industries, including remediation of the environment, synthesis of chemicals, and pharmaceutical use. In addition, this work highlights the possibilities of plant biomaterials as stabilizers for nanoparticles, boosting the emerging application of green chemistry and the pursuit of sustainable nanomaterials.

5.3. Recommendations

From the results, the synthesis conditions can be further optimized in the future, including temperature, time, and pH, to improve further the size control and catalytic activity of the nanoparticles. Long-term stability and recyclability experiments are also required to determine the practical applicability of these nanoparticles in real catalytic processes. Examination of these nanoparticles in a broader set of catalytic reactions will determine their versatility and potential for wider industrial use.

In addition, surface area and porosity analysis, including BET surface area measurements, would give greater insight into the correlation between surface characteristics and catalytic activity. Finally, large-scale synthesis should be investigated to move these laboratory-scale nanoparticles into commercially available products, making them scalable for industrial use.

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7. Conflict of Interests

Here, the authors do not claim any conflict of interest.

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