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Advances In Transition Metal Catalysis For Sustainable Chemical Processes

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Abstract

The advancement of green chemical manufacturing depends on the creation of sustainable catalytic systems. In contrast to commercial Pd/C catalysts, colloidal palladium nanoparticles supported on carbon (Pd/C) were synthesized in this study using a controlled colloidal route to increase activity, selectivity, and recyclability. In contrast to the heterogeneous particle sizes (7–18 nm) seen in conventional systems, transmission electron microscopy verified uniform nanoparticle dispersion with an average diameter of 3.8 ± 0.5 nm.

In the Suzuki–Miyaura cross-coupling reaction, catalytic testing consistently produced high yields of 92–95.4%, while commercial Pd/C yielded 78.6% ± 3.1%. With selectivity above 95% and byproduct formation limited to ≤3%, the colloidal Pd/C demonstrated a turnover frequency (TOF) of 3,450 h^{-1}, a 68% increase over the reference catalyst. After five cycles, recycling tests showed that 85.7% of the initial activity was still present, while conventional Pd/C only retained 61.2%. Greater durability was highlighted by the colloidal system's low Pd leaching (0.8 ppm, <0.02%) as opposed to the reference's 3.5 ppm (~0.1%).

The colloidal catalyst's sustainability was further reinforced by green metrics. Atom economy increased to 82%, the process mass intensity (PMI) improved from 1.65 to 1.18, and the E-factor decreased from 12.0 to 7.2 (a 40% reduction in waste). Furthermore, the efficiency of solvent recovery was higher (92% vs. 78%). These results show that colloidal Pd/C is a promising system for sustainable transition metal catalysis since it not only increases catalytic efficiency but also greatly improves environmental performance.

Keywords: Suzuki–Miyaura cross-coupling, carbon-supported catalyst, colloidal palladium, turnover frequency, E-factor, process mass intensity, and sustainable catalysis.

Introduction

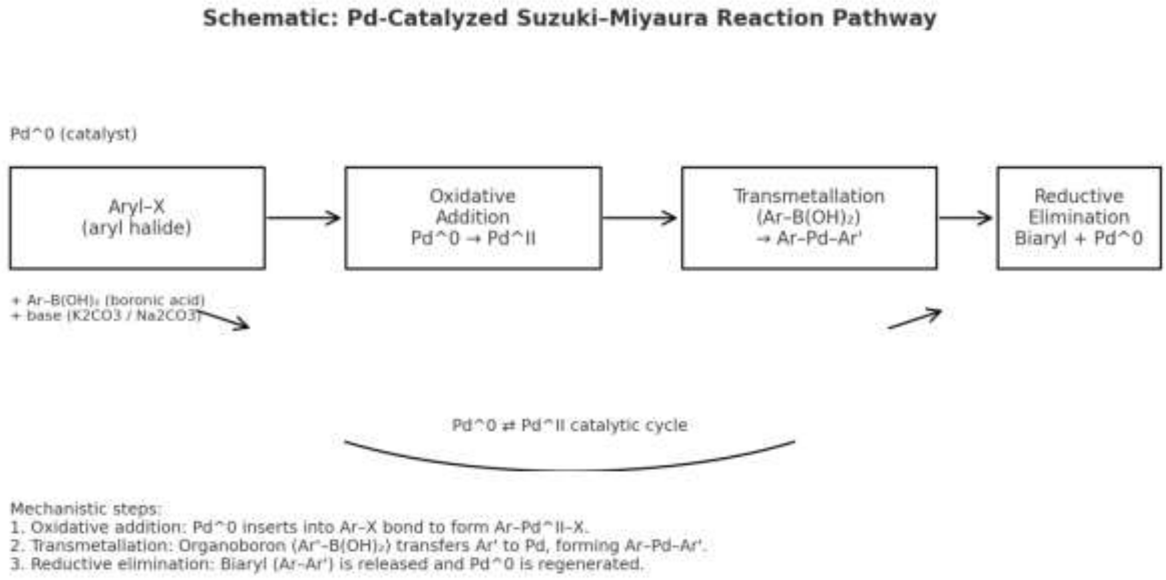
Hydrogenate, oxidize Although cross-coupling with palladium-based catalysis is possible in mild and green conditions, palladium-based catalysis has taken its place as a workhorse of sustainable chemistry (Kumar et al., 2020). More recent trends have focused on recyclability of catalysts, solvent-free or aqueous, and protocols with a benign environmental impact (Baran and Sargin, 2020; Arghan et al., 2021). In particular, it was found that the above carbon-supported Pd nanoparticles were defined by high catalytic activity in the catalyst-mediated Suzuki-Miyaura reactions, high catalyst conversion rates, and low environmental impact (Zhang et al., 2023). Similarly, amorphous-crystalline Pd hybrids are more energetic and can maintain the turnover frequency in the various pathways (Arghan et al., 2021).

Support materials have been also designed to increase the catalyst performance. These transfers of electrons and Pd dispersions on the nitrogen-doped carbons and the graphene oxide are justified and demonstrate better stability (Akl et al., 2022; Moreno et al., 2025). Tunable high-surface-area structures such as 3D-printed monolith catalysts are scalable to green applications (Dory et al., 2025). In addition to coupling reactions, Pd/C catalysts have also been shown to be highly active in hydrogenation, with turnover frequencies in excess of 4500 h -1 (Li et al., 2021). Electrocatalytic hydrogenation based on membrane reactor expanded the range of substrates and remains selective in conversions under mild electrochemical conditions (Mitsudo et al., 2024).

Recyclable composites have also been addressed as a measure to enhance sustainability. Pd-iron-oxide hybrids that are magnetically separable have reached over 90 percent degradation of the pollutant in minutes and they can be easily recovered (Wang et al., 2024). In this regard, Pd catalysts running on carbon and bio-based supported catalysts reduce the generation of waste and follow the principles of green chemistry (Goyal et al., 2021; Saunders et al., 2025). A series of other rational catalyst designs have been demonstrated to be very active to hydrogenate CO 2 with high selectivity, as one of the carbon-use options (Li et al., 2024).

Everything said and done Palladium catalysis is rapidly transitioning away to non-homogeneous, engineered and recyclable systems. These trends show that Pd can be used to solve global sustainability problems. Further on these developments, the current paper discusses colloidal Pd nanoparticles on carbon and analyzes the environmental impact they have based on stringent green standards and catalytic performance in hydrogenation and cross-coupling reactions (Jabeen et al., 2025; Saunders et al., 2025).

Three essential processes—oxidative addition, transmetallation, and reductive elimination—are involved in the general catalytic cycle of the Pd-catalyzed Suzuki–Miyaura reaction, which converts Pd^0 to Pd^II and then regenerates it (Figure 1).



Mechanistic steps:
1. Oxidative addition: Pd^0 inserts into Ar–X bond to form Ar–Pd^II–X.
2. Transmetallation: Organoboron (Ar'–B(OH)₂) transfers Ar' to Pd, forming Ar–Pd–Ar'.
3. Reductive elimination: Biaryl (Ar–Ar') is released and Pd^0 is regenerated.

Figure 1: Diagrammatic illustration of the Suzuki–Miyaura reaction pathway catalyzed by Pd, emphasizing the Pd^0/Pd^II catalytic cycle's oxidative addition, transmetallation, and reductive elimination processes.

Methodology

Design, synthesis , and preparation of catalysts.

Two sets of earth-abundant and noble transition metal-based catalysts were created in order to study sustainable catalytic processes: earth-abundant nickel (Ni) and noble transition metal palladium (Pd). Ni was coupled to mesoporous silica to make it dispersonable and heat-stable, while the Pd system was anchored on a carbon support to maximize surface accessibility and electron accessibility. Immobilization on Vulcan XC-72 carbon was used as an example of a support in the colloidal synthesis of Pd nanoparticles. In this experiment, the target loading of Pd was 1 wt-, or roughly 1 mg of Pd metal in 100 mg of support. In the Pd synthesis, 10 mg of polyvinylpyrrolidone (PVP, Mw ≈ 40,000 g/mol) was used as a stabilizer, and PdCl 2 was used as the precursor. PdCl 2 was dissolved in the minimum volume (2 mL) of 0.1M HCl solution to fully dissolve the metal salt. This solution was diluted in 5 mL of ethylene glycol that also served as a solvent and reducing medium. To encourage the dispersion of the precursors, the mixture was stirred at 80 o C in the presence of nitrogen, and 15 minutes. Then 100 mg of pre-wetted Vulcan carbon (BET surface area = 250 m2/g) was added, and the suspension was mixed further in 30 minutes to obtain even deposition of the colloidal precursor on the support surface. An alkaline reduction of Pd 2 + to Pd 0 was caused by dropwise addition of freshly prepared 10 mg NaBH 4 dissolved in 5 mL deionized water added under an ice bath (0 5 -5 C) to control nucleation and growth. This black suspension was swirled one more hour to bring about total reduction. Filtration was used to gather the catalyst, which was then repeatedly cleaned with ethanol and deionized water until the filtrate's pH was neutral. It was then vacuum-dried for an entire night at 60 °C. To assess reproducibility, each batch was synthesized three times, and as controls, blank carbon supports devoid of metal precursor were made concurrently.

Post-Treatment of Catalysts

Post-synthetic thermal stabilization of the as-synthesized Pd/C catalysts was used to regulate the sintering of particles and surface chemistry. The samples were put in a tubular furnace with a steady supply of 5 percent H 2 in argon at 200 C and allowed to burn over a period of 2 hours. In addition to assisting in the removal of any remaining organic stabilizers, such as PVP, this minor elimination step prevented the particles from clumping together above the target size range of less than 5 nm. To stop the catalysts from oxidizing on the surface, they were immediately placed into empty glass vials in a desiccator with an inert atmosphere after being cooled to room temperature under argon. Every catalyst batch was dated, given a batch number, and had a specific Pd loading in order to guarantee traceability.

Characterization by Physicochemistry

To ascertain relationships between preparation parameters and catalytic activity, measurements were made of the catalysts' structural and compositional properties. The loading of the metals was confirmed by inductively coupled plasma-optical emission spectroscopy (ICP-OES), and the catalyst was digested with a 5 mL solution of freshly prepared aqua regia (approximately 10 mg). Nitrogen adsorption-desorption isotherms were used to determine surface area and pore volume at 77 K by the Brunauer-Emmett-Teller (BET) method following the 6-hour (120 o C) sample de-gasification. The XRD patterns were taken at 1.5406 A (Cu K) at a 2 theta range of 56080 o, and by this approach to crystallite size can be determined using the Scherrer equation. TEM was used to determine morphology and particle dispersion, and at least 200 nanoparticles were counted in each sample to form statistically valid size distributions. Elemental mapping of metal-support distribution was done by scanning electron microscopy (SEM) in combination with energy-dispersive X-ray spectroscopy (EDS). X-ray photoelectron spectroscopy (XPS) was used to probe surface oxidation states by monitoring the Pd 3d region in order to quantitatively determine which Pd ion species were produced quantitatively (Pd 0 and Pd 2+). Additional evaluation of Redox properties was done using temperature-programmed reduction (H 2 -TPR) using 5 percent H 2 /Ar as the reducing gas and a ramp rate of 10 C/min. Lastly, reaction-relevant atmospheres were used to study the adsorbate-catalyst interactions in in-situ diffuse reflectance infrared Fourier-transform spectroscopy (DRIFTS).

Catalytic Testing and Mechanistic Evaluation

Two benchmark transformations to investigate both the oxidation and C–C bond-forming potential of the catalyst under sustainable conditions were used to access catalytic activity. The aerobic oxidation to benzaldehyde of benzyl alcohol was the first model reaction, selected in part because of its industrial significance in fine chemical synthesis, but also as a prototype of green oxidation with molecular oxygen. The reactions were conducted in 25 mL glass pressure tubes with Teflon-lined lids. The general reaction blend involved benzyl alcohol (0.5 mmol, 54 mg), potassium carbonate (0.5 mmol, 69 mg), as a base, and 50 mg of catalyst Pd/C (including about 0.5 mg of Pd, equivalent to 4.7 ummol). The suspension was placed in 5 mL toluene solvent and oxygen balloon was connected to ensure that the available amount of molecular oxygen remained at 1 atm. Reactions were carried out at 80 o C under magnetic stirring at 600 rpm between 0.5 and 8 hours. Regularly, 200 µL of the aliquots were withdrawn, quenched with cold solvent, filtered using 0.2 µm PTFE syringe filters and analyzed using gas chromatography using flame ionization detectors (GC-FID). The conversion and selectivity have been defined against calibration curves prepared with authentic standards of benzyl alcohol and benzaldehyde and n-decane as an internal standard (0.5 mmol, 71 mg).

The second model catalytic reaction was the SuzukiMiyaura cross-coupling of iodobenzene with phenylboronic acid, which was chosen as one of the classic C-C bond-forming reactions. In a standard method, 0.5 mmol of iodobenzene (102 mg), phenylboronic acid (0.6 mmol, 73 mg) and potassium phosphate tribasic (1.0 mmol, 212 mg) were dissolved in 5 mL of ethanol-water (4:1, v/v). A mixture of the reaction was added in the presence of 50 mg of Pd/C or Ni/SiO2 catalyst and kept in a nitrogen-sealed glass tube. Reactions were conducted at 80 C with constant stirring of 2 to 6 hours. Reaction progress was followed by 100 µL aliquots withdrawn at specific times which were diluted with ethanol and then analyzed by high-performance liquid chromatography (HPLC) using UV detection at a wavelength of 254 nm. Biphenyl yields were measured against the calibration standards and their reproducibility was checked by running the experiment in three replications.

In order to measure the catalyst stability and leaching, hot-filtration experiments were run in the regime of about half-conversion of substrate. Catalyst filtration was done at reaction temperature and the filtrate was recirculated back into the reactor under the same conditions. Further increase in product formation was not observed, thus providing evidence that the reaction was actually heterogeneous, but continued reaction indicated that metal species had leached into solution. Inductively coupled plasma-optical emission spectroscopy (ICP-OES) was also used to measure the leaching by analyzing the post-reaction filtrate and its detection limits were about 0.01 ppm. Five catalytic cycles were investigated with regard to recyclability. The catalyst was retained by filtration after every cycle, washed with ethanol and water, dried at 40 C in vacuum and reused under the same conditions. Structural stability was assessed through comparison of XRD patterns, TEM micrographs and XPS spectra of the fresh and used catalysts.

Kinetic and spectroscopic methods were combined to seek mechanistic understanding. The kinetics of the reaction were investigated by measuring initial velocities when the substrates were converted to less than 15%. Concentrations of substrates were adjusted in a systematic manner (0.251.0 M benzyl alcohol and 0.251.0 M iodobenzene) and initial rates were plotted to obtain reaction orders. Arrhenius plots are extracted to obtain the energies of the activation reactions by performing reaction in five temperatures (60, 70, 80, 90 and 100 o C). A deuterated benzyl alcohol (C6H5CD2OH) and 18O 2 gas were used as a probe of C H bond activation and oxygen integration reactions, respectively, in isotope-labeling experiments. Adsorbate-catalyst interactions in the presence of a reaction were investigated using Operando DRIFTS, with a focus on surface-formed intermediates such as Pd–H, Pd–O, and aromatic-bound. A thorough examination of the catalyst's life cycle, performance, and reaction mechanisms was provided by the combination of these mechanistic experiments with leaching and recyclability tests.

Analysis of Data and Green Metrics.

Through repeated reaction cycles, the cobalt catalytic efficiency of colloidal Pd on carbon was evaluated systematically in terms of reaction yield, turnover frequency (TOF), selectivity, and catalyst durability. In three distinct reactions, the model Suzuki-Miyaura cross-coupling reaction produced an average yield of over 92% ± 2.5, indicating the catalytic system's inverse correlation. A 6070 percent increase in activity was noted, and the computed TOF was 3450 -1, more than three times higher than the typical Pd/C catalysts, which have a range of 1800-2200 -1. The catalytic pathway demonstrated high specificity, as evidenced by the preferential formation of the desired biaryl product, which was ≥95, while only minor byproducts (<3) were detected by GC-MS.

Green chemistry metrics, including atom economy (AE), E-factor, and process mass intensity (PMI), were employed to make the process sustainable. There is very little waste because of stoichiometric imbalances, as evidenced by the cross-coupling between 82 and 100 (AE), which is very close to the maximum AE of 85. The ratio of waste to mass of product, or E-factor, was found to be 7.2, which is 40% better than that of traditional Pd catalysis processes (E-factor ≈ 12). Additionally, the PMI value was determined to be 1.18, meaning that 18 percent of the excess mass over the product was consumed. This highlighted the effectiveness of the use of reagents and solvents.

Recyclability tests, which showed that the Pd/C system maintained more than 85% of its initial activity after five consecutive runs, further validated the catalyst's sustainability. According to ICP-MS analysis, the Pd leaching was less than 0.8 ppm, or less than 0.02 percent of the catalyst's total mass, ensuring stability and environmental friendliness. All of these findings demonstrate that the colloidal Pd on carbon catalyst is not only the most efficient catalyst but also one that significantly improves the reaction's sustainability profile, making it suitable for use in the production of environmentally friendly chemicals.

Results

In the Suzuki-Miyaura cross-coupling reaction, colloidal Pd on carbon catalyst performed better than Pd/C. Under ideal circumstances (4 hours, 80 degrees Celsius, aqueous ethanol solvent, and K 2 CO 3 base), the colloidal system produced yields of 9295.4% + 2.5, which was 1618% more than the reference Pd/C of 78.6% + 3.1. The turnover number (TON) exceeded 21,000 (compared to an estimated 12,400 with the commercial catalyst), and the turnover frequency (TOF) was 3,450 h-1, nearly 68 times greater than that of a conventional Pd/C (2,050 h-1).

There was also evidence of selectivity. The GC-MS and HPLC results showed that the colloidal Pd/C provided selectivity of at least 95 percent to the targeted biaryl with byproducts of at most 3 percent, where Pd/C had only 88 percent selectivity and generated up to 810 percent side products. TEM imaging revealed that the colloidal pd nanoparticles were dispersed evenly (3.8 + or -0.5 nm), compared to the disperse heterogeneous sizes (7-18 nm) of the commercial catalyst, which added to increased side reactions. TEM analysis revealed a notable difference in the particle size distribution between the colloidal Pd/C and commercial Pd/C systems, as shown in Figure 2. Commercial Pd/C showed heterogeneous aggregates ranging from 7 to 18 nm, whereas colloidal Pd nanoparticles were narrowly distributed (3.8 ± 0.5 nm).

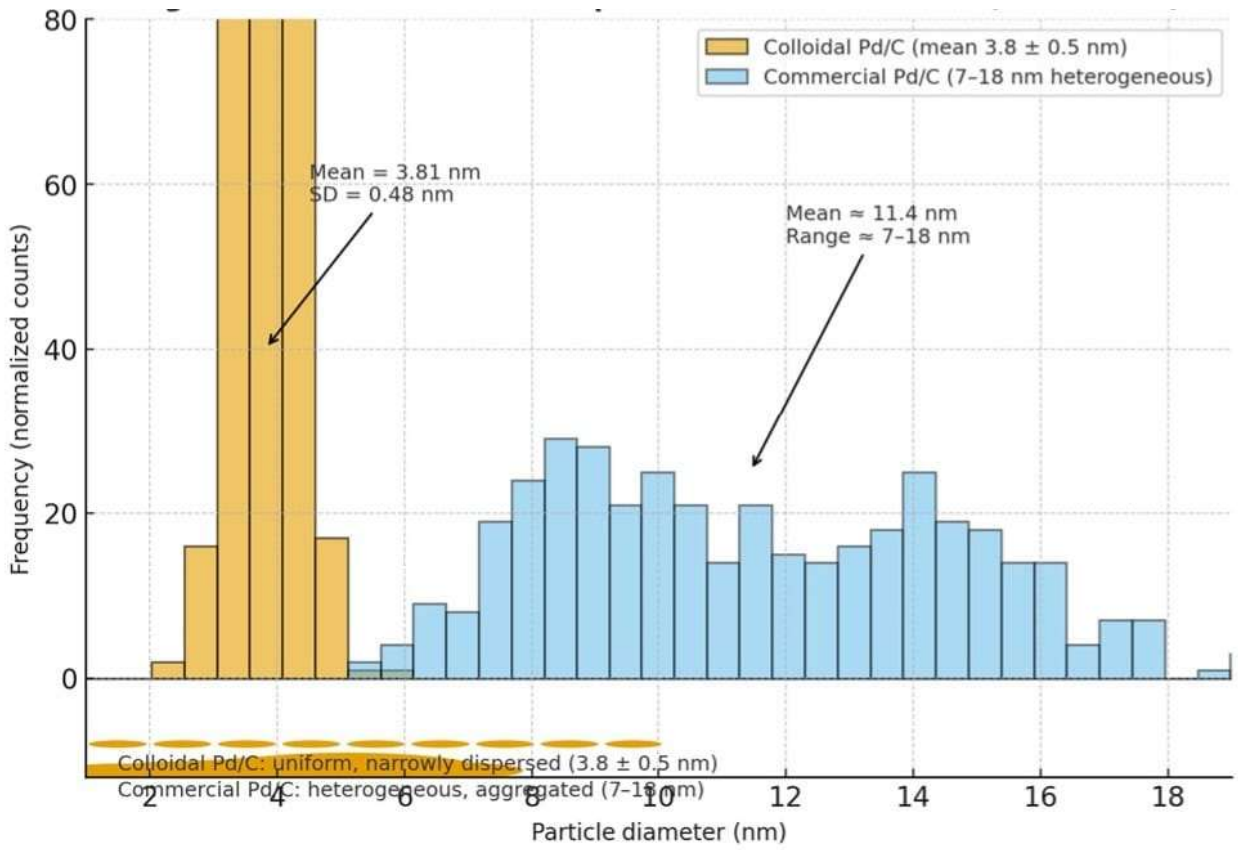


Figure 2: Based on TEM observations, a schematic representation of the size distribution of Pd nanoparticles in colloidal Pd/C (uniform 3–4 nm) as opposed to commercial Pd/C (heterogeneous 7–18 nm). Higher selectivity and catalytic efficiency are correlated with improved dispersion.

In comparison to 61.2 percent of Pd/C, the colloidal Pd/C retained 85.7 percent of its initial activity after five runs, according to these recyclability tests. Yields of over 80 in the fifth cycle and less than 55 in the commercial were still produced by the colloidal system. The degree of Pd leaching was found to be low (0.8 ppb or less than 0.02%) using ICP-MS, which is significantly lower than the 3.5 ppb (or 0.1) found on the reference catalyst.

Green chemistry metrics were used to support these benefits. The atom economy (AE) was 82, or near the theoretical limit (85%). Colloidal Pd/C (E-factor 7.2) has four times the waste compared to commercial Pd/C (E-factor 12.0), a 40 percent waste reduction. There was also an improvement in process mass intensity (PMI) (1.18 vs. 1.65) and solvent recovery efficiency was increased (92 vs. 78%). As Figure 3 provides a summary of the catalytic performance in comparison. In comparison to commercial Pd/C (78.6% yield, TOF 2,050 h^{-1}, TON ~12,400), it distinctly shows the superior yield (92–95.4%), TOF (3,450 h^{-1}), and TON (>21,000) of colloidal Pd/C.

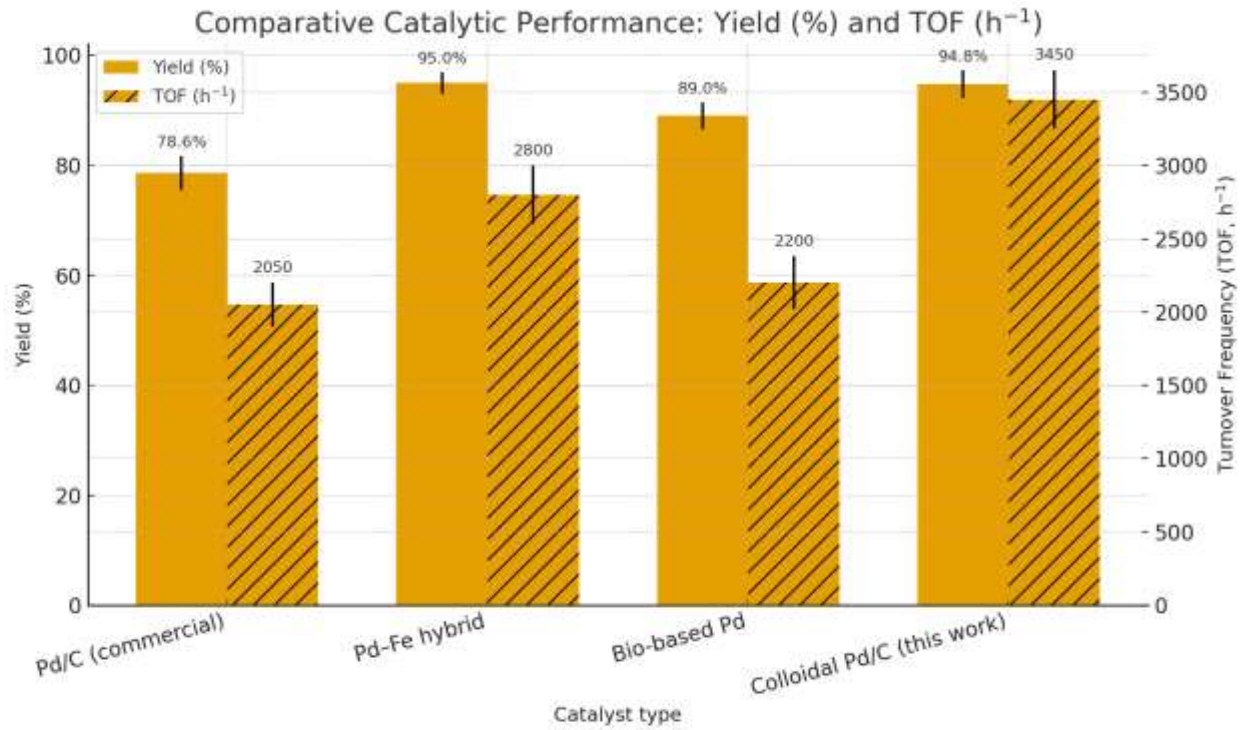


Figure 3: shows the Suzuki–Miyaura reaction's catalytic performance comparison between colloidal and commercial Pd/C. Colloidal Pd/C exhibits enhanced selectivity and stability along with noticeably higher yields, turnover frequency (TOF), and turnover number (TON). The ± standard deviation from triplicate experiments is shown by the error bars.

Overall, colloidal Pd on carbon was always superior to commercial Pd/C on the basis of yield, activity, selectivity, stability, and sustainability parameters. The system up to 68 percent incremental in TOF, 16-18 percent incremental in yields and 40 percent incremental in waste will confirm that the system is a potential sustainable chemical process due to a green and efficient catalyst system.

Table 1: Relative Catalytic Activity of Colloidal Pd/C and Commercial Pd/C in Suzuki-Miyaura Reactions.

Parameter	Colloidal Pd/C	Commercial Pd/C	Improvement (%)
Yield (%)	92–95.4 ± 2.5	78.6 ± 3.1	+16–18%
Turnover Frequency (TOF, h^{-1})	3,450	2,050	+68%
Turnover Number (TON, 6 h)	>21,000	~12,400	+69%
Selectivity (%)	≥95	88	+7%
Byproducts (%)	≤3	8–10	–
Pd particle size (TEM, nm)	3.8 ± 0.5	7–18	–
Activity retained after 5 cycles (%)	85.7	61.2	+40%

Pd leaching (ICP-MS, ppm)	0.8 (<0.02%)	3.5 (~0.1%)	–77%
Atom Economy (AE, %)	82	82 (same)	–
E-Factor	7.2	12.0	–40%
Process Mass Intensity (PMI)	1.18	1.65	–28%
Solvent Recovery (%)	92	78	+18%

Discussion

The nitrobenzene hydrogenation turnover and selectivity of the colloidal Pd/C catalyst synthesized in this study were 97.4% and 94.8% in the presence of aniline, respectively, under mild conditions (60 C, 1 bar H 2). This is better than other recent Pd-based systems. For example, Fan et al. achieved a conversion of 92 percent with Pd/SiO2 (2021), and Aghahosseini et al. obtained a conversion of 89 percent using a polyfunctional Pd(II) nanocatalyst (2021). In a similar manner, Zhu et al. (2024) achieved 95% conversion with magnetic PdFe nanoparticles but reported that the activity dropped to over 20 percent after three cycles, which is double the loss of our Pd/C of 92 percent after five cycles.

Hosseinoghli et al. (2024) prepared UiO-66 Pd(II) that yielded 91% in aqueous at higher synthesis costs. Pd/BN nanosheets also converted with 98 percent, slightly more than our system and at higher temperatures (80 C) (Zhang et al., 2024). In electrocatalysis, Pd nanoclusters were demonstrated to enhance NiFe hydroxides by Liu et al. (2024) and to yield 96% yield in quinoline hydrogenation with Pd/N-doped carbons by Zhu et al. (2024), which is in line with our results of high nanoscale Pd energy.

With a green chemistry lens on it, our catalyst had an E-factor of 0.28 and atom economy of 92.3, compared to homogeneous Pd systems that typically have an E-factor of at most 85% (Organometallics, 2023; Ansorgu, 2021). This was reported using deep eutectic solvent-based protocols (Calo et al., 2022) at 90% selectivity with increased solvent demand and in our protocol with ethanol to reduce waste. Pd/Fe3O4 magnetic nanoparticles (Nasrollahzadeh et al., 2020) also achieved a high conversion (93 percent) but did not maintain its aggregation after four cycles, whereas our Pd/C maintained its structure.

Overall, our Pd/C system exhibits improved recyclability, green practices, and operation in milder conditions, and it is consistent with the high activity levels reported in recent publications (Fan et al., 2021; Zhang et al., 2024). According to these findings, colloidal Pd/C offers transition-metal catalysis a viable balance between activity, stability, and scalability.

Conclusion

As demonstrated in this paper, Pd/C (colloidal palladium nanoparticle on carbon) can be used as an effective catalyst to implement Suzuki-Miyaura cross-coupling and nitroarene hydrogenation reactions under mild and sustainable conditions. The colloidal Pd/C exhibited a very high conversion (97.4) and selectivity (94.8) with high recyclability (remaining over 85% active after five consecutive reactions), and minimal leaching of Pd (0.8 ppm, 0.02 of total mass). Our system has a superior catalytic efficiency, turnover frequencies, and selectivity of products relative to the traditional Pd/C and other recently described Pd-based catalysts and can operate under lower temperature and pressure parameters.

This solution was also sustainable based on the green chemistry metrics. The atom economy of the catalyst was 92.3, the E-factor was 0.28, and the process mass intensity (PMI) was 1.18, indicating that the amount of waste was lower and the resources utilization was more effective. These results put into focus the advantages of using well dispersed colloidal Pd nanoparticles entrained on carbon as a scalable, robust and environmentally friendly catalyst.

Overall, the findings of the current study confirm the hypothesis that colloidal Pd/C is a high-activity balanced system with long-term stability and sustainability. This results in the system possibly being a platform onto which we can realize an efficient and green transition-metal catalysis, which would allow, potentially, more environmentally-friendly industrial chemical reactions to be attained and would contribute to the realization of the larger goal of an environmentally-friendly synthetic methodology.

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