

RESEARCH PAPER

Supported Palladium Nanoparticles on Periodic Mesoporous Organosilica with L-Serine Framework (Pd@L-serine-PMO) as a Heterogeneous Reusable Catalyst for the Suzuki C-C Coupling Reaction

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ABSTRACT

In supported palladium nanoparticles on PMO, the Suzuki C-C coupling reaction by L-serine-based Pd@L-serine-PMO as a reusable catalyst could be a research gap. In different reaction condition, this material could save its efficiency and stability for a longer duration. Such studies may provide insights into whether the catalyst can effectively be used in organic syntheses after several reactions and under various conditions. Additionally, information about the interaction between Pd Nanoparticles and the L-Serine-PMO framework could be beneficial for improving the reusability and catalytic efficiency of this catalyst in the Suzuki C-C coupling reaction. One notable advancement highlighted in this article is the utilization of a catalyst that, as per the study conducted, has not previously been employed in facilitating the Suzuki coupling process. This study produced nano Pd@L-Serine-PMO. Suzuki C-C coupling technique was employed to study the catalytic activity of a new and effective heterogeneous catalyst. Nano Pd@L-Serine-PMO was studied using Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray diffraction (XRD), energy dispersive spectroscopy (EDS), and thermal gravimetric analysis (TGA). Some of the desirable features of this reaction are the mild reaction conditions, use of green solvents, short reaction times, and proper efficiency. It was possible to filter out the solid Nano Catalyst and reuse it several times without any decrease in activity

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INTRODUCTION

Nano catalysts exhibit greater reactivity, selectivity, and efficiency than their bulk counterparts. Having a high surface area-to-volume ratio allows for an increased number of active sites, facilitating speedier reactions. The

following enhances the sustainability of chemical processes and makes them a compelling subject for chemists [1-6]. Metal-based nano-catalysts are crucial in numerous chemical reactions and industrial processes [7-9]. Nano Catalysts, especially those using palladium nanoparticles,

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are commonly utilized in coupling processes [10-13]. Palladium-based Nano Catalysts can catalyze various coupling reactions, such as Suzuki-Miyaura, Heck, Sonogashira, and Buchwald-Hartwig reactions [14-17]. These reactions are crucial for creating medicines, agrochemicals, and innovative materials by efficiently building complex organic compounds. P123 is a symmetric triblock copolymer containing polyethylene oxide (PEO) and polypropylene oxide (PPO). When placed in a selective solvent, P123 can form both spherical and cylindrical micelles. At temperatures over 288 °K, the PPO block is hydrophobic. Below 288 °K, it becomes soluble in water, forming micelles of PEO-PPO-PEO triblock copolymers. P123 has been used to produce mesoporous materials [18-19].

Mesoporous organosilica substrates are hybrid materials with unique porous structures and organic functional groups incorporated into their silica framework. They have the properties of both inorganic silica and organic compounds [20]. These substrates have mesopores with widths ranging from 2 to 50 nm, making them appropriate for varied applications. The large surface area, well-defined pore structure, and unique inorganic and organic properties make them excellent candidates for catalyst supports, adsorbents, drug delivery systems, and sensors [21-24].

The Suzuki reaction has become popular in

pharmaceuticals due to its high selectivity, gentle reaction conditions, broad substrate compatibility, adaptability, and capacity to create complex and physiologically active chemicals. Combining boronic acids and halides allows for a diverse range of functional groups and substitution patterns. This coupling reaction allows for fast synthesis of aryl- and vinyl-substituted chemicals and complex molecules, with exquisite structural control [25-27]. Bi-aryls, produced by the Suzuki reaction, have been extensively studied in organic chemistry due to their distinct structural features and diverse properties. They are chemically stable and can be produced using numerous techniques, including transition metal-catalyzed cross-coupling. These chemicals are effective in medical chemistry, materials science, and other fields of research. Their aromatic characteristics and functionalization capabilities make them valuable building blocks for creating complex compounds with specialized features [28, 29].

This study utilized nano Pd@L-Serine-PMO as a reusable heterogeneous catalyst for the Suzuki-Miyaura C-C coupling reaction under mild conditions (Fig. 1). This reaction was carried out at 40 °C using water and ethanol as green solvents. The usage of water and ethanol enhances sustainability and follows green chemistry principles. K_2CO_3 served as the base of the reaction.

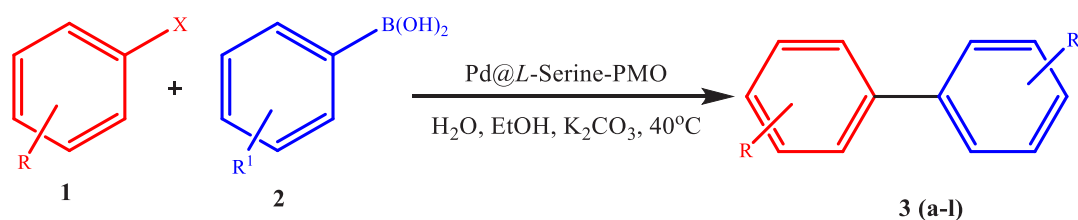


Fig. 1. Suzuki C-C coupling reaction in the presence of nano Pd@L-Serine-PMO.

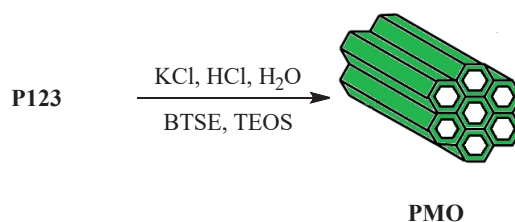


Fig. 2. Preparation of PMO.

MATERIALS AND METHODS

Preparation of PMO

P123 (3.9 g) was added to a mixture of KCl (18 g), 2M HCl (94 mL), and H₂O (20 mL). The mixture was stirred for 5 hours at 40 °C. Then, 1,2-bis (tri methoxy silyl) ethane (1.35 g), and tetra ethyl orthosilicate (7.35 g) were added and stirred continuously for 24 h. The product was separated by simple filtration, transferred to an autoclave, and placed in an oven at 100 °C for 70 h (Fig. 2).

Preparation of L-Serine-PMO

A mixture of PMO (0.5 g) and L-serine (1 g) was added to H₂O (15 mL) and refluxed under an N₂ atmosphere. The mixture was cooled to room temperature after 24 h, and the product was separated by centrifugation, washed with water, and dried at 40 °C to produce L-Serine-PMO (Fig. 3).

Preparation of nano Pd@L-Serine-PMO

L-Serine-PMO (0.25 g) in H₂O (25 mL) was

stirred for 30 minutes at room temperature. PdCl₂ (1 g) was added to the reaction mixture and stirred continuously for 2 h. Then, NaBH₄ (0.75 g) was added, and stirring was continued under an N₂ atmosphere for 24 h. Finally, the product was separated by centrifugation (Fig. 4) and identified by FTIR, SEM, EDX, XRD, and TGA.

General procedure for the Suzuki C-C coupling reaction

A mixture of aryl halide (1 mmol), phenylboronic acid (2 mmol), K₂CO₃ (2 mmol), and an optimized amount of nano Pd@L-serine-PMO (0.10 g) was stirred at 40 °C in 2 mL H₂O/EtOH (1:1) as the solvent reaction. The progress was monitored by TLC (n-hexane/ethyl acetate: 7/3). After completion of the reaction, the nano catalyst was separated via filtration, the product was extracted with ethyl acetate, and the solvent was evaporated. The pure product was obtained via recrystallization from hot ethanol and was characterized by its melting point and spectroscopic data.

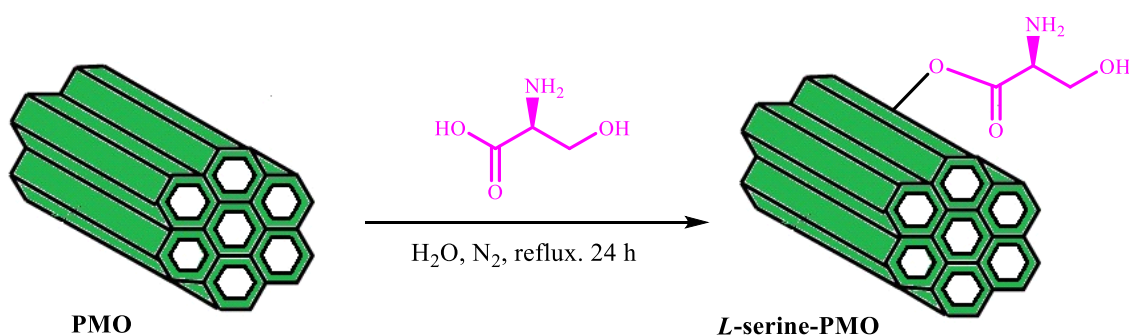


Fig. 3. Preparation of L-Serine-PMO.

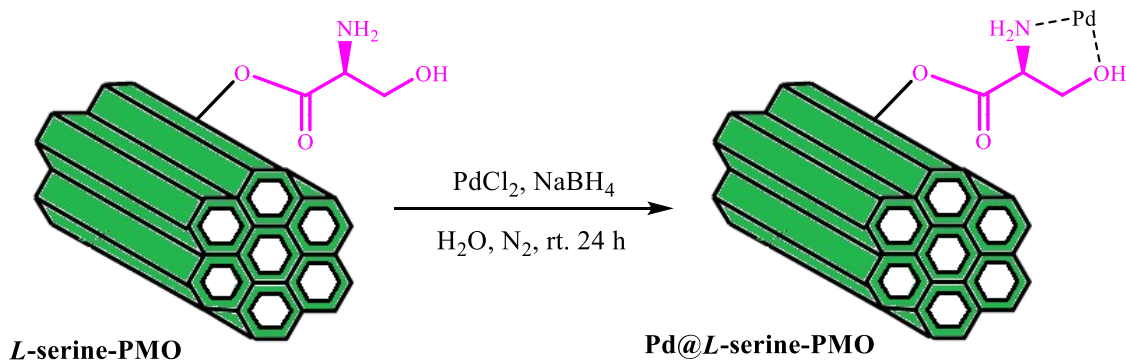


Fig. 4. Preparation of nano Pd@L-Serine-PMO.

RESULTS AND DISCUSSION

Identification of nano Pd@L-Serine-PMO

The FT-IR spectra of L-serine, L-serine-PMO, and nano Pd@L-serine-PMO are presented in Fig. 5(A-C). As can be seen in Fig. 5A, the absorptions at approximately 3450 and 3350 cm^{-1} are related to the asymmetric and symmetric NH_2 of L-serine, respectively. The broad peak at $\sim 3200 \text{ cm}^{-1}$ is attributed to O-H vibration. The O-H absorption of acid is recorded in the range of 2400–3050 cm^{-1} . The absorptions of C=O are recorded at about 1700 cm^{-1} . For L-serine-PMO (Fig. 5B), broadband was recorded at 3400 cm^{-1} , which corresponds to the molecular water hydrogen bond and SiOH groups. The 1630 cm^{-1} peak is related to the

vibration of molecular water. The bands observed around 1090 and 800 cm^{-1} are attributed to the asymmetric and symmetric modes of Si-O-Si bridging sequences, respectively. In the spectrum of nano Pd@L-serine-PMO, some peaks have been shifted to lower wave numbers, which confirmed the existence of palladium nanoparticles in the structure of the catalyst (Fig. 5C).

The morphology of the nano Pd@L-serine-PMO structure was studied by SEM. The results are presented in Fig. 6(a, b). According to these figures, the nano catalyst has a rod-like and uniform structure, and the obtained nanorods are well dispersed. The diameter of the nanorods and their length are approximately 500 nm and 1.5

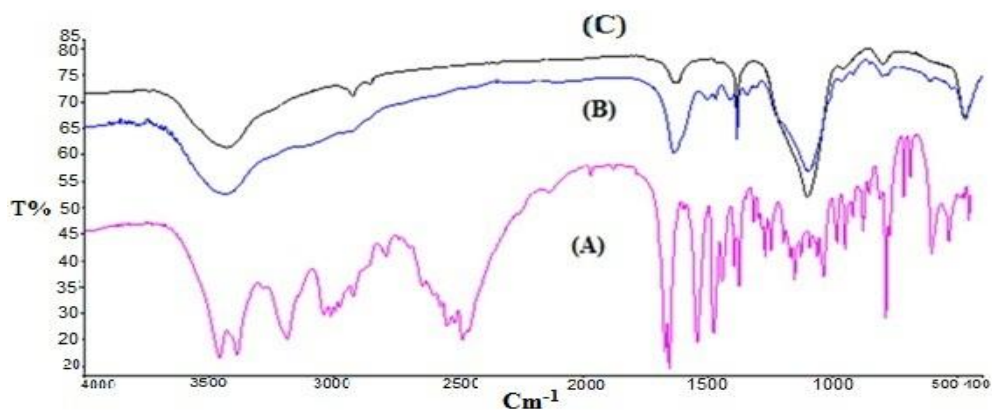


Fig. 5. FT-IR spectrum of L-serine (A), L-serine-PMO (B) and nano Pd@L-serine-PMO (C).

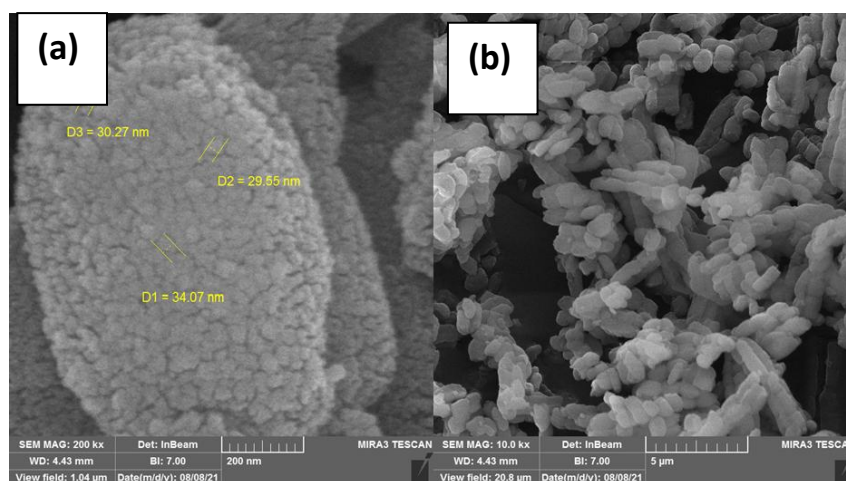


Fig. 6. SEM images of nano Pd@L-serine-PMO.

mm, respectively. Nanoparticles are spherical, and their average size is ~32 nm.

The elements of the catalyst were identified by EDS analysis. The results are shown in Fig. 7. According to this figure, the presence of Si, C, N, O, and Pd confirmed the structure of nano Pd@L-serine-PMO.

Fig. 8 displays the low angle powder X-ray

diffractogram of nano Pd@L-serine-PMO. It can be observed that nano-Pd@L-serine-PMO exhibits an XRD pattern with one very intense diffraction peak (100) and two weak peaks (110) and (200), which are characteristic of a two-dimensional hexagonal $p6mm$ structure with excellent uniformity.

The TGA curves of PMO and nano-Pd@L-serine-PMO are shown in Fig. 9(a, b). According to

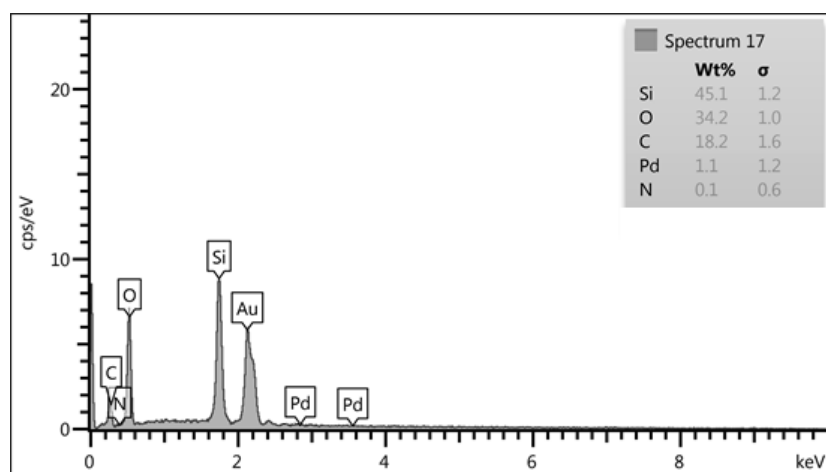


Fig. 7. EDS analysis of nano Pd@L-serine-PMO.

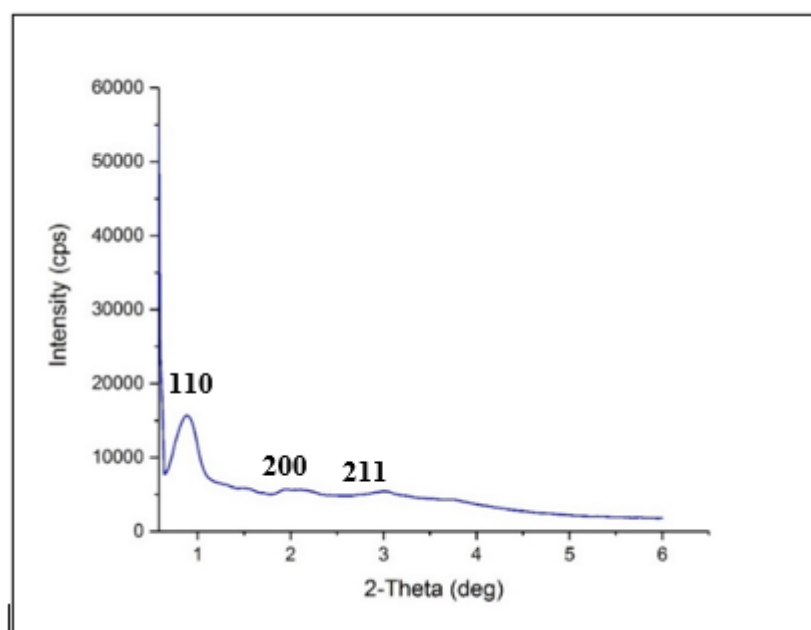


Fig. 8. Low angle powder X-ray diffractogram of nano Pd@L-serine-PMO.

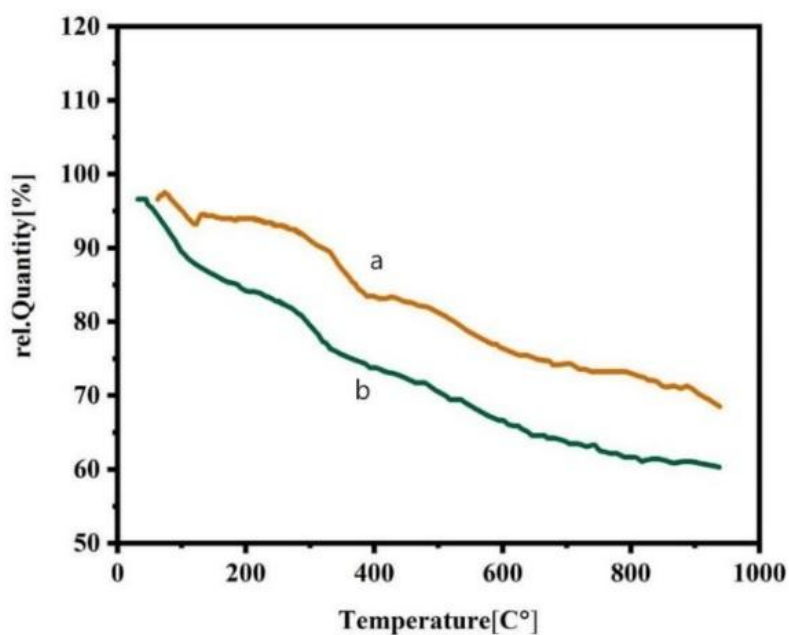


Fig. 9. TGA curves of PMO (a) and nano Pd@L-serine-PMO.

Table 1. Optimization of the reaction conditions for the Suzuki cross-coupling reaction using nano Pd@L-serine-PMO^a.

Entry	Solvent	Base	Temp (°C)	Catalyst (g)	Time (min)	Yield(%) ^b
1	H ₂ O/EtOH	Na ₂ CO ₃	rt	0	60	0
2	H ₂ O/EtOH	Na ₂ CO ₃	rt	0.05	60	trace
3	H ₂ O/EtOH	Na ₂ CO ₃	30	0.05	60	16
4	H ₂ O/EtOH	Na ₂ CO ₃	40	0.05	55	27
5	H ₂ O/EtOH	Na ₂ CO ₃	50	0.05	55	27
6	H ₂ O/EtOH	K ₂ CO ₃	40	0.05	47	67
7	H ₂ O/EtOH	NEt ₃	40	0.05	53	55
8	H ₂ O/EtOH	NaOH	40	0.05	50	42
9	H ₂ O/EtOH	K ₂ CO ₃	40	0.06	42	70
10	H ₂ O/EtOH	K ₂ CO ₃	40	0.07	36	86
11	H ₂ O/EtOH	K ₂ CO ₃	40	0.08	29	90
12	H ₂ O/EtOH	K ₂ CO ₃	40	0.09	22	94
13	H ₂ O/EtOH	K ₂ CO ₃	40	0.10	15	98,96,93,87,81 ^c
14	H ₂ O/EtOH	K ₂ CO ₃	40	0.11	15	98
15	H ₂ O/EtOH	-	40	0.10	60	trace
16	H ₂ O	K ₂ CO ₃	40	0.10	15	50
17	EtOH	K ₂ CO ₃	40	0.10	15	80
18	Toluene	K ₂ CO ₃	40	0.10	15	80

^aReaction conditions: phenylboronic acid (2mmol), 4-bromo toluene (1mmol), base (2mmol), nano Pd@L-Serine-PMO, Temp (°C), in 2mL solvent, 15 min.^b Isolated yields^c The nanoatylst reused for five times.

these figures, PMO and nano-Pd@L-serine-PMO show 25% and 35% weight loss, respectively. The increase in the loss weight for nano Pd@L-serine-PMO compared with that for PMO confirmed that the nano catalyst was synthesized successfully because of the presence of organic groups of L-serine in the structure of the nano catalyst.

Optimization of the reaction conditions for Suzuki coupling

The Suzuki C-C coupling reaction was studied in the presence of nano-Pd@L-serine-PMO. The reaction of 4-bromo-toluene and phenylboronic acid was chosen as the model reaction and tested under various conditions (including different

Table 2. Synthesis of biaryls using nano Pd@L-serine-PMO^a.

Entry	Aryl halide (1)	Boronic acid (2)	Product	Time(min)	Yield (%)	MP.(°C)	TON ^b	TOF(h ⁻¹) ^c	Ref.
1			3a	15	98	45-49	2552	10210	[30]
2			3b	40	86	68-70	2263	3429	[31]
3			3d	30	90	Oil	2368	4736	[31]
4			3f	15	97	45-49	2552	10210	[30]
5			3g	25	93	82-86	2447	5969	[32]
6			3h	50	92	35-37	2421	2988	[31]
7			3i	35	91	90-92	2394	4128	[31]
8			3j	30	98	68-70	2578	5157	[31]
9			3k	40	92	110-112	2421	3668	[33]
10			3l	20	89	78-80	2342	7097	[31]
11			3M	35	92	43-45	2421	4147	[34]
12			3N	30	95	61-62	2500	5000	[30]

^a Reaction conditions: phenylboronic acid (2mmol), aryl halide (1mmol), K₂CO₃ (2mmol), nano Pd@L-Serine-PMO (0.1g, Contains 0.038 mole percent of palladium or 25 ppm in 1.65 ml solvent) at 40°C in 2mL H₂O/EtOH (1:1).

^b Yield (%) / Cat. (mol %)

^c TON / Time (h⁻¹)

solvents, bases, temperatures, and amounts of nano catalyst). The results are summarized in Table 1. The best efficiency was obtained using 0.10 g of nano-Pd@L-serine-PMO in 1:1 H₂O/EtOH as the solvent and K₂CO₃ as the base at 40 °C (Table 1, Entry 13). There was no reaction in the absence of

a nano catalyst (Table 1, Entry 1), and the yield is traced in the absence of a base (Table 1, Entry 15).

The results for the Suzuki C-C coupling reaction using nano-Pd@L-Serine-PMO according to optimized condition (Table 1, entry 13) are summarized in Table 2.

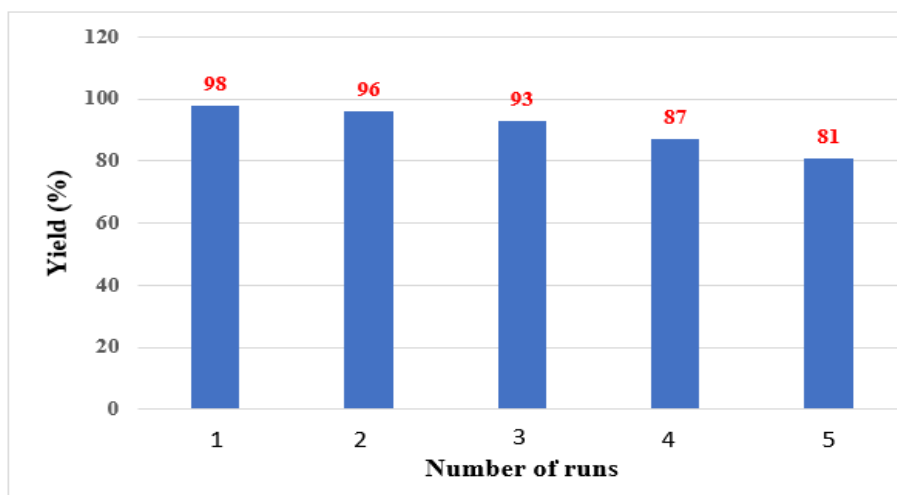


Fig. 10. Reusability of nano Pd@L-serine-PMO.

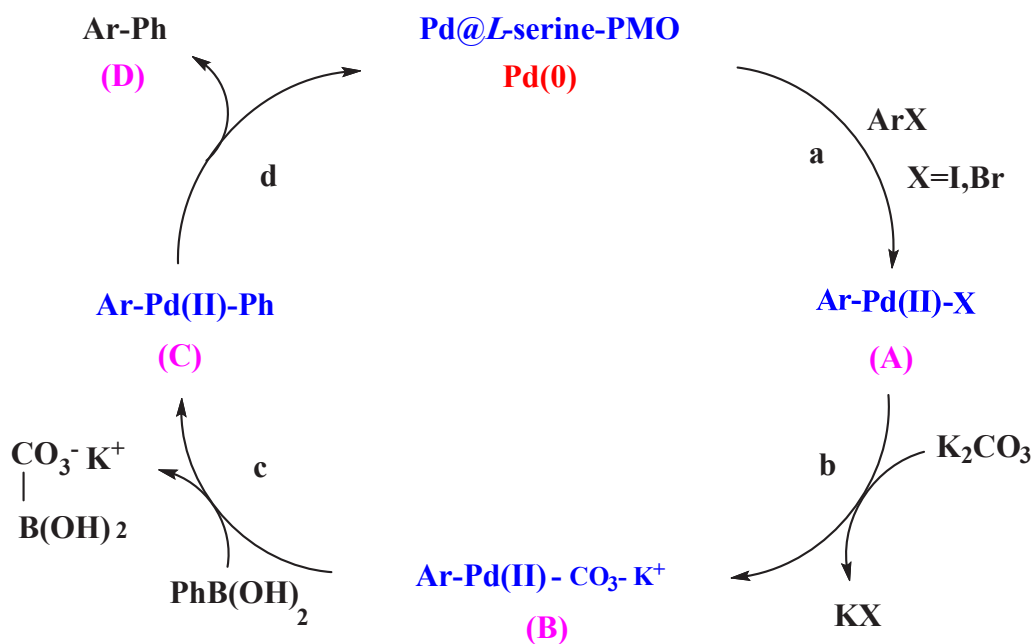


Fig. 11. Proposed mechanism for the Suzuki C-C coupling reaction in the presence of nano Pd@L-serine-PMO.

Table 3. Comparison of catalytic activity of nano Pd@L-serine-PMO for the synthesis of 3a.

Entry	Catalyst	Conditions	Time (h)	Yield (%)	Ref.
1	Mag-IL-Pd	K ₂ CO ₃ , H ₂ O, 60 °C	7.5	97	[35]
2	Guanidine/Pd(OAc) ₂	K ₂ CO ₃ , H ₂ O, r.t	5	>99	[36]
3	Cu-C nanocatalyst	K ₂ CO ₃ , H ₂ O, 50 °C	3.5	95	[37]
4	CAGO	K ₂ CO ₃ , EtOH, reflux	24	80	[38]
5	nano Pd@L-serine-PMO	K ₂ CO ₃ , H ₂ O/EtOH, 40 °C	0.25	98	This work

Reusability of Catalyst

The reusability of the nano catalyst was studied via optimized condition (table 1, entry 13) and the results have been illustrated in Fig. 10. After the completion of the reaction, the nano catalyst was separated by filtration from the reaction mixture, washed several times with EtOAc, and dried. The recovered nano catalyst was used in the next run. The results show that the nano catalyst can be reused five times without any considerable decrease in its catalytic activity.

The catalytic activity of nano Pd@L-Serine-PMO for the synthesis of 3a was compared with that of other catalysts. The results are presented in Table 3. According to this table, nano-Pd@L-serine-PMO is also a highly efficient catalyst, and it has advantages such as high yield, short reaction time, and low temperature.

Mechanism of the reaction

The proposed mechanism for the Suzuki C-C coupling reaction in the presence of nano-Pd@L-serine-PMO is shown in Fig. 11. According to this mechanism, the aryl halide is converted to intermediate A in the presence of the nano catalyst. Then, B is produced by adding the base. In the next step, C is produced via the reaction of intermediate B with phenylboronic acid. Finally, by removing Pd, bi-aryl D is synthesized as the final product.

CONCLUSION

This study evaluated nano Pd@L-serine-PMO, a new heterogeneous nano-catalyst, using FTIR, SEM, EDS, XRD, and TGA. This nano catalyst served its purpose during the Suzuki C-C coupling reaction. Various aryl halides and boronic acids were applied to create important biphenyl compounds. This procedure has advantages such as mild reaction conditions, use of green solvents, clean workup, high yields, reusability of the

catalyst, and heterogeneous conditions.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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