

A SIMPLE AND EFFICIENT CATALYST [PdCl₂(CH₃CN)₂] FOR SUZUKI-MIYAURA CROSS-COUPPLING REACTION UNDER MILD REACTION CONDITIONS

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ABSTRACT

Highly efficient and general [PdCl₂(CH₃CN)₂] catalysed Suzuki-Miyaura coupling (SMC) reaction between bromoarenes and arylboronic acid is described. The reaction proceeds rapidly at low catalytic loading (0.01 mol%) in a water/*i*-PrOH mixture at 60 °C. Instead of using a bulky ligand system, we have used an easily available and cost-effective palladium reagent with mild reaction conditions. This protocol features the easy approachability of the catalyst, broad substrate scope and simple operation.

Keywords: Palladium, Catalysis, Suzuki-Miyaura, Water, Isopropanol, Bromoarenes, Boronic Acid.

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INTRODUCTION

The creation of carbon-carbon bonds facilitated by coordination complexes using transition metals is a potent method in organic synthesis, with significant applications in the production of carbon-based compounds, including medicines, natural products, and polymers.¹⁻⁶ The SMC reactions represent the most adaptable and extensively employed technique for the formation of discerning sp²-sp² carbon-carbon bonds.⁷⁻¹⁴ Since its detection in 1979, the SMC, which entails the coupling of a carbon boron substance with a carbon-based halide or pseudo-halide in the presence of a Pd or Ni catalytic agent and a base, has probably developed into one of the most employed methodologies for constructing carbon-carbon bonds. The reaction proceeds through an oxidative addition, transmetalation, and reductive elimination catalytic cycle that is enhanced by the incorporation of electron-rich and sterically bulky ligand systems that facilitate the initial and final stages.¹⁵⁻¹⁹ The distinct advantages of this palladium-catalysed SMC reaction encompass: (a) mild reaction environments; (b) the accessibility of carbon-based boron substances, which exhibit inertness to water, correlated solvents, and oxygen, and are usually stable at high temperature; (c) compatibility with diverse functional groups; and (d) low toxicity of both initial compounds and by-products. These characteristics have enabled researchers to employ it in diverse applications, ranging from the production of expected products to the creation of polymeric resources. In September 2010, Akira Suzuki, alongside Richard F. Heck and Ei-Ichi Negishi, was granted the Nobel Prize in Chemistry for their contributions to coupling reactions in organic compounds catalysed by palladium metals, reflecting its significance across multiple disciplines. Since that time, exemplary research addressing particular facets of this reaction has been published.²⁰⁻²¹ Typically, electron-rich ligands such as phosphines and *N*-heterocyclic carbenes (NHC) are necessary to stabilise the active Pd species in palladium-catalysed SMC processes. Nonetheless, numerous ligands are air-sensitive, costly, poisonous, and non-recoverable, imposing considerable constraints on their synthetic utilities. Consequently, the creation of a water-soluble, phosphine-free Pd catalyst is a subject of significant interest.²²⁻²⁴ Taking into account economic, safety, and environmental concerns, the utilisation of aqueous solvent for Pd-catalysed coupling processes, including Ullmann, Suzuki, Hiyama, and Stille couplings in atmospheric conditions, was initially investigated by Li and colleagues.²⁵ The investigation of SMC reactions in aqueous or aqueous organic solvent mixtures has garnered significant consideration since that

time. The capacity to solubilise bases in water for the activation of aryl boronic acids accelerates the process significantly in an aqueous environment. Zhang *et al.* observed that the palladium-catalysed SMC reaction of bromoarenes with arylboronic acids proceeded well in $\text{H}_2\text{O}-\text{CH}_3\text{COCH}_3$ or $\text{PEG}/\text{H}_2\text{O}$ (PEG = polyethylene glycol) without the presence of a ligand.²⁶ Sajiki and colleagues have delineated a Pd/C-catalysed ligand-free system for the SMC reaction conducted in a water-ethanol combination at ambient temperature.²⁷ Del Zotto *et al.* formulated a rapid method for the SMC reaction in aquatic 1,2 dihydroxy ethane monomethyl ether, being the sole instance of a extremely competent and ligand-free SMC reaction conducted in air at ambient temperature.²⁸ Liu and co-workers have developed the competent SMC reaction of bromoarenes with arylboronic acids in the presence of PdCl_2 (0.5 mol % loading), K_2CO_3 and aqueous DMF at room temperature in air with 5 to 120 minutes of reaction time²⁹. Karvembu and group have published the SMC reaction of bromoarenes with arylboronic acids (1.1 equiv.) in the presence of $[\text{PdCl}_2(\text{CH}_3\text{CN})_2]$ (2 mol % loading), Na_2CO_3 (1.1 equiv.) and aqueous medium at 45 to 60 °C temperature in air with 0.5 to 9 hours of reaction time.³⁰ We report herein the efficient SMC reaction of bromoarenes with arylboronic acids (1.2 equiv.) in the presence of $[\text{PdCl}_2(\text{CH}_3\text{CN})_2]$ (0.01 mol %), KOH (2 equiv.) and aqueous alcohol medium at 60 °C temperature in air with 15 to 30 minutes of reaction time.

EXPERIMENTAL

Materials and Methods

Analytically grade reagents were used. The palladium catalyst was purchased from Sigma-Aldrich, and all the bromoarenes and phenylboronic acid were obtained from CDH and used without extra purification. ^1H and ^{13}C NMR spectroscopic studies were performed using 300 MHz Bruker Topspin NMR spectrometers; *d*-chloroform was used as a solvent. The chemical shifts δ are reported in ppm and are referenced internally with respect to the residual solvent (^1H NMR) and solvent (^{13}C NMR) resonances.

General Procedure for Suzuki Coupling Reactions

All aryl bromides, phenylboronic acids, and KOH were used as received. A mixture of *iso*-propyl alcohol (analytical grade) and distilled water was used as the solvent. A round-bottom flask was charged with aryl bromide (1.0 mmol), phenylboronic acid (1.2 mmol), KOH (2.0 mmol), *i*-PrOH (4.0 mL), H_2O (4.0 mL) and catalysts with continuous stirring in a 60 °C oil bath. After different time intervals, the maximum conversion of haloarenes to the coupling product was monitored by ^1H NMR spectroscopy. The reaction mixture was taken out with dichloromethane, washed with water and dried over anhydrous Na_2SO_4 , and the product was further decontaminated by column chromatography (hexane/ethyl acetate) on silica gel.

4-phenylacetophenone

The general procedure was used for the product formation using *p*-bromoacetophenone (1 mmol, 200 mg) and phenylboronic acid (1.2 mmol, 147 mg) as reactants. White solid (92 %, 181 mg). ^1H NMR (300 MHz, CDCl_3 , ppm): δ 2.61 (s, 3H, COCH_3), 7.37-7.47 (m, 3H, *Ar*), 7.59-7.67 (m, 4H, *Ar*), 8.00 (d, $^3J_{\text{HH}} = 6.6$ Hz, 2H, *Ar*). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 26.75 (COCH_3), 127.37, 128.30, 129.05, 136.01, 140.01, 145.90 (C_{Ph}), 197.82 (COCH_3).

4-phenylbenzaldehyde

The general procedure was used for the product formation using 4-bromobenzaldehyde (1 mmol, 300 mg) and phenylboronic acid (1.2 mmol, 237 mg) as reactants. Yellow solid (94 %, 555 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.34-7.38 (m, 3H, *Ar*), 7.57 (m, 2H, *Ar*), 7.67 (d, $^3J_{\text{HH}} = 8.1$ Hz, 2H, *Ar*), 7.86 (d, $^3J_{\text{HH}} = 6.6$ Hz, 2H, *Ar*), 10.00 (s, 1H, CHO). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 127.38, 127.68, 128.51, 129.05, 130.28, 135.25, 139.71, 147.16 (C_{Ph}), 191.89 (CHO).

4-methoxybiphenyl

The general procedure was used for the product formation using 4-bromoanisole (1 mmol, 250 mg) and phenylboronic acid (1.2 mmol, 195 mg) as reactants. White solid (90 %, 221 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 3.81 (s, 3H, OCH_3), 6.95 (dd, $^3J_{\text{HH}} = 2.4$ Hz, $J_{\text{HH}} = 4.5$ Hz, 2H, *Ar*), 7.26-7.30 (m, 1H, *Ar*), 7.36-7.41 (m, 2H, *Ar*), 7.48-7.54 (m, 4H, *Ar*). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 55.45 (OCH_3), 114.35, 126.78, 126.86, 128.27, 128.84, 133.93, 140.97, 159.31 (C_{Ph}).

Biphenyl

The general procedure was used for the product formation using 4-bromobenzene (1 mmol, 300 μ L) and phenylboronic acid (1.2 mmol, 417 mg) as reactants. White solid (92 %, 405 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.37-7.42 (m, 2H, *Ar*), 7.49 (dt, $^3J_{\text{HH}} = 6.0$ Hz, 1.8 Hz, *Ar*), 7.63-7.69 (m, 4H, *Ar*). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 127.30, 127.38, 128.89, 141.37 (C_{Ph}).

4-methylbiphenyl

The general procedure was used for the product formation using *p*-bromotoluene (1 mmol, 200 mg) and phenylboronic acid (1.2 mmol, 171 mg) as reactants. White solid (95 %, 187 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 2.39 (s, 3H, CH_3), 7.31 (d, $^3J_{\text{HH}} = 2.4$ Hz, 1H, *Ar*), 7.42 (s, 2H, *Ar*), 7.49 (d, $^3J_{\text{HH}} = 7.8$ Hz, 2H, *Ar*), 7.57 (d, $^3J_{\text{HH}} = 6$ Hz, 2H, *Ar*). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 29.87, 127.13, 128.85, 129.62 (C_{Ph}).

4-nitrobiphenyl

The general procedure was used for the product formation using 1-bromo-4-nitrobenzene (1 mmol, 250 mg) and phenylboronic acid (1.2 mmol, 181 mg) as reactants. Yellow solid (91 %, 224 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.40-7.50 (m, 3H, *Ar*), 7.58-7.62 (m, 2H, *Ar*), 7.71 (dt, $^3J_{\text{HH}} = 6.9$ Hz, 2.2 Hz, 2H, *Ar*), 8.27 (dt, $^3J_{\text{HH}} = 9.0$ Hz, 2.2 Hz, 2H, *Ar*). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 124.21, 127.50, 128.91, 129.04, 129.27, 138.89, 147.74 (C_{Ph}).

4-tert-butylbiphenyl

The general procedure was used for the product formation using 1-bromo-4-tert-butylbenzene (1 mmol, 200 μ L) and phenylboronic acid (1.2 mmol, 169 mg) as reactants. White solid (92 %, 223 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 1.35 (s, 9H, CH_3), 7.30-7.38 (m, 1H, *Ar*), 7.41-7.47 (m, 4H, *Ar*), 7.51-7.59 (m, 1H, *Ar*). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 31.53 ($\text{C}(\text{CH}_3)$), 125.85, 126.93, 127.12, 127.17, 127.32, 127.39, 128.84, 128.90 (C_{Ph}).

3-methoxybiphenyl

The general procedure was used for the product formation using 3-bromoanisole (1 mmol, 200 μ L) and phenylboronic acid (1.2 mmol, 231 mg) as reactants. White solid (88 %, 256 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 3.8 (s, 3H, OCH_3), 6.88 (dd, $^3J_{\text{HH}} = 8.4$ Hz, 2.4 Hz, 1H, *Ar*), 7.86-7.90 (m, 2H, *Ar*), 7.33-7.36 (m, 2H, *Ar*), 7.42 (t, $^3J_{\text{HH}} = 6.9$ Hz, 2H, *Ar*), 7.57 (d, $^3J_{\text{HH}} = 7.1$ Hz, 2H, *Ar*). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 55.43 (OCH_3), 112.81, 113.05, 119.82, 127.33, 127.54, 128.86, 129.87, 141.25, 142.91, 160.08 (C_{Ph}).

2-phenylbenzonitrile

The general procedure was used for the product formation using 2-bromobenzonitrile (1 mmol, 150 mg) and phenylboronic acid (1.2 mmol, 120 mg) as reactants. White solid (85 %, 125 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.69 (d, $^3J_{\text{HH}} = 8.0$ Hz, 1H, *Ar*), 7.47-7.57 (m, 3H, *Ar*), 7.34-7.44 (m, 4H, *Ar*), 7.25-7.31 (m, 1H, *Ar*). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): 111.22, 118.66, 127.09, 127.22, 127.52, 128.69, 130.03, 132.78, 133.68, 138.12, 145.42 (C_{Ph}).

2-phenylbenzaldehyde

The general procedure was used for the product formation using 2-bromobenzaldehyde (1 mmol, 300 μ L) and phenylboronic acid (1.2 mmol, 805 mg) as reactants. White solid (86 %, 223 mg). ^1H NMR (CDCl_3 , 300 MHz, ppm): δ 7.31-7.34 (m, 2H, *Ar*), 7.38-7.46 (m, 5H, *Ar*), 7.58 (t, $^3J_{\text{HH}} = 7.5$ Hz, 1H, *Ar*), 7.99 (d, $^3J_{\text{HH}} = 7.8$ Hz, *Ar*), 9.94 (s, 1H, CHO). $^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, CDCl_3): δ 127.61, 127.83, 128.18, 128.49, 130.16, 130.83, 133.62, 133.76, 137.79, 146.01, 192.45 (C_{Ph}).

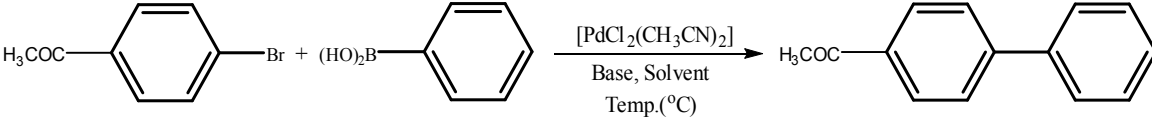
RESULTS AND DISCUSSIONS

Optimization of Reaction Condition

To get compelling results, the reaction conditions, with solvent, base, catalyst loading, and time duration, were optimised for the SMC reaction. *p*-bromoacetophenone and phenylboronic acid were designated as model substrates with $[\text{PdCl}_2(\text{CH}_3\text{CN})_2]$ catalyst for the SMC reaction, and the progress was checked by ^1H NMR (Table-1). Initially, the commercially available base (KOH) and different solvents (*i.e.* toluene,

acetonitrile, THF, water, isopropanol) with 0.1 mol % catalyst loading were analysed for 30 min of reaction time at 60 °C temperature and found that the isopropanol and water were the good solvents compared to others (entries 1-5 in Table-1). After that, we changed the solvent polarity by mixing a 1:1 ratio of isopropanol and water and found the excellent results with 99 % conversion of reactant to product within 20 min of reaction time (entry 6 in Table-1). After the solvent study, parameters like catalyst loading, base and reaction temperature were analysed. Under similar conditions but with K₂CO₃ base only, 68 % conversion was found (entry 7 in Table-1). As shown in entry 8 in Table-1, the reaction was performed at R.T.; the reaction rate is slow, with 51 % conversion only. Further, when the reaction was performed with low catalyst loading (0.01 mol %) and 20 min reaction time, 99 % conversion was observed (entry 9 in Table-1). Under similar conditions, further reduction of catalyst amount (0.001 mol %) was not fully convert the reactant to product, with 76 % conversion (entry 10 in Table-1). Consequently, from the above results, it was concluded that 0.01 mol % of [PdCl₂(CH₃CN)₂], KOH base, *i*-PrOH and water solvents (1:1 ratio) and 20 min reaction time at 60 °C temperature were suitable conditions for the SMC reaction.

Table-1: Reaction Condition Optimisation for SMC Reaction

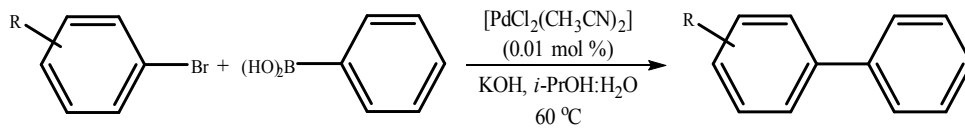
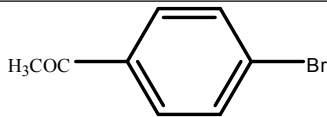
						
Entry No.	Catalyst (mol %)	Base	Solvent	Time(min)	Temp. (°C)	Conversion (%)
1.	0.1	KOH	Toluene	30	60	25
2.	0.1	KOH	CH ₃ CN	30	60	26
3.	0.1	KOH	THF	30	60	42
4.	0.1	KOH	H ₂ O	30	60	74
5.	0.1	KOH	<i>i</i> -PrOH	30	60	85
6.	0.1	KOH	<i>i</i> -PrOH: H ₂ O	20	60	99
7.	0.1	K ₂ CO ₃	<i>i</i> -PrOH: H ₂ O	30	60	68
8.	0.1	KOH	<i>i</i> -PrOH: H ₂ O	30	R.T.	51
9.	0.01	KOH	<i>i</i> -PrOH: H ₂ O	20	60	99
10.	0.001	KOH	<i>i</i> -PrOH: H ₂ O	20	60	76

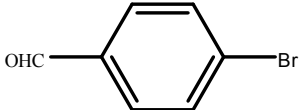
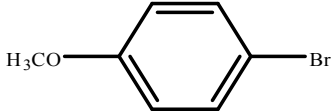
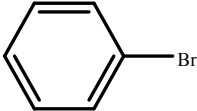
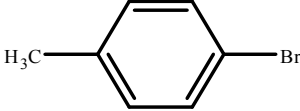
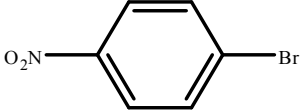
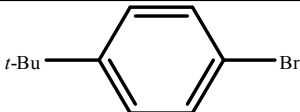
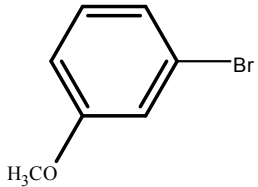
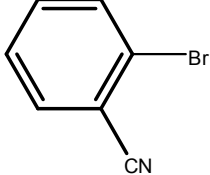
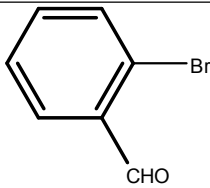
Reaction conditions: *p*-bromoacetophenone (1 mmol), phenylboronic acid (1.2 mmol), KOH (2 mmol), solvent (4 mL), R.T. (room temperature). Conversion was checked by proton NMR spectroscopy.

Substrates Scope

With the optimised reaction conditions in hand, we explored the scope of this reaction with various bromoaryl substrates (Table-2). Table-2 displays the conversion of aryl halides to biaryl derivatives in high yields. Several bromoarene substrates with substitution at different positions of the benzene ring bearing EDG (Electron donating group) and EWG (Electron withdrawing group) gave the corresponding product with good yield.

Table-2: Bromoarenes Substrates for SMC Reaction

			
Entry	Aryl bromide	Time (min)	Conversion (%)
1.		20	99

2.		15	99
3.		30	98
4.		15	98
5.		25	99
6.		20	99
7.		15	98
8.		30	94
9.		20	97
10.		15	99

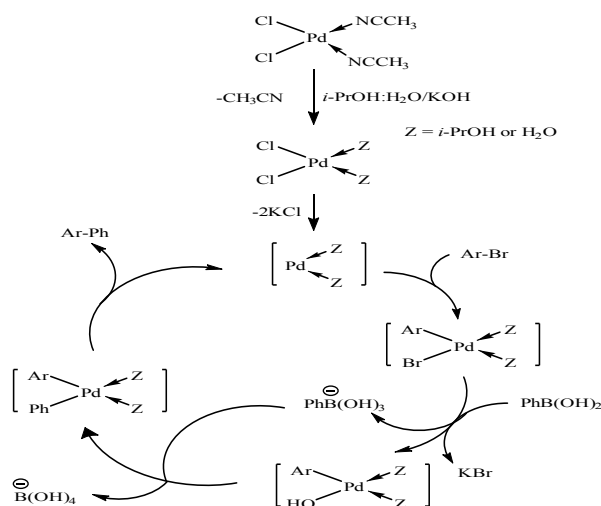
Conditions: haloarenes (1 mmol), phenylboronic acid (1.2 mmol), KOH (2 mmol), *i*-PrOH (2 mL), H₂O (2 mL), Catalyst (0.01 mol %), Temp. 60 °C. Conversion was checked by ¹H NMR spectroscopy.

The electron-withdrawing groups on aryl bromide increased the reaction rate, which led to an excellent yield. Only 15 minutes were required for the whole conversion of *p*-bromobenzaldehyde and *p*-nitrobenzaldehyde to biphenyl products, which are electron-deficient and more reactive in this kind of reaction. For those haloarenes with EDGs, longer reaction times were needed. 4-bromoanisole and 3-bromoanisole afforded a quantitative yield 98 and 94%, respectively, in 30 minutes of reaction time, and the other electron-donating substrate, 4-bromotoluene, afforded the biphenyl product in 25 minutes of reaction time. Longer reaction time for electron-donating substrates may be due to the less reactive C-Br bond compared to electron-withdrawing substrates. Bulky groups attached to the aryl halide did not affect the coupling product yield. 4-tert-butylbromobenzene gave the corresponding product in 98% yield within 15 minutes of reaction time. Even though the presence of ortho groups at the aryl halide also did not affect the desired product yield. 2-bromobenzonitrile and 2-bromobenzaldehyde afforded the biaryl

product with 97 and 99 % yield, respectively, within 20 and 15 minutes of reaction time, respectively. As expected, a wide range of functional groups such as nitro, cyano, aldehyde and keto were tested for the SMC reaction, and no reaction was found on those functional groups.

Expected Mechanism

Based on the literature review³⁰, we have proposed the mechanism for the $[\text{PdCl}_2(\text{CH}_3\text{CN})_2]$ catalysed SMC reaction in a mixed solvent (aqueous and alcohol) medium (Scheme-1). In the initial step, the catalyst is converted to a solvent-soluble Pd (II) complex, which is coordinated by solvent molecules instead of the acetonitrile molecule. The Pd (II) complex is reduced by KOH base and generates an activated catalyst, which is a palladium (0) complex.



Scheme-1: Proposed Mechanism for Suzuki-Miyaura Coupling Reaction for the $[\text{PdCl}_2(\text{CH}_3\text{CN})_2]$ Catalytic System

The activated palladium (0) catalyst gives the oxidative addition reaction with an aryl halide. After that, phenyl boronic acid is activated by KOH base, which enhances the polarisation of bonds and encourages the transmetalation. In the last step, the palladium (II) complex eliminates the cross-coupling product (Ar-Ph) and regenerates the activated palladium (0) catalyst complex to start a new catalytic cycle.

CONCLUSION

In summary, we have used a commercially available, man-made ligand-free palladium-catalysed $[\text{PdCl}_2(\text{CH}_3\text{CN})_2]$ for the SMC reaction of bromoarenes with arylboronic acid in an aqueous alcohol mixture medium. Significantly, a various functional group such as EDG and EWG could be tolerated, which makes this an efficient synthetic protocol for cross-coupling reactions. These outcomes are very useful for the easy production of various synthetic organic compounds without using harsh reaction conditions.

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CONFLICTS OF INTEREST

The authors have no conflicts of interest to declare.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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