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Nanocellulose Supported PdNPs as *in situ* Formed Nano Catalyst for the Suzuki Coupling Reaction in Aqueous Media: A Green Approach and Waste to Wealth

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ABSTRACT

An effective and highly sustainable method has been developed for the Suzuki coupling reaction of aryl halides and phenylboronic acid using *in situ* generated nacocellulose (NC) supported palladium nanoparticles (Pd@NC NPs) as heterogeneous catalysts in water. The formation of PdNPs was observed in the absence of additional reagents, during the course of the Suzuki reaction from NC and Pd(OAc)$_2$. *In situ* formed Pd@NC NPs have been systematically characterized using TEM, EDX, XRD and FT-IR techniques. The *in situ* generated catalyst was separated and studied for its successful reusability up to 11 cycles. The application of aqueous media, use of *in situ* formed catalyst, mild reaction conditions, absence of volatile organic solvents, broad substrate scope, high product isolated yields are the significant developments of this protocol. The nanocellulose used for this protocol has been generated form the waste cotton cloth and hence it is an example for the waste to wealth and sustainable approach.

1. Introduction

The today's chemical science is making efforts to provide a healthy environment to the mankind *via* green approaches. On this concern, researchers are developing chemical products *via* eco-friendly approaches [1–9]. Green chemistry has been promoting significant awareness in the scientific community, particularly in organic synthesis due to its financial and environmental advantages. From the industrial point-of-view cost-effective and trouble-free process always desirable. Nano-Pd-catalysis is a major multidisciplinary area in cross-coupling reactions. Among the coupling reactions Suzuki coupling reaction is the widely employed strategy for the preparation of biaryls and these are very essential in pharmaceuticals and other industries [10,11].

In general researchers follow two different routes in the preparation of biaryls; one through homogenous catalysis and the other using heterogeneous catalytic systems. A considerable number of homogenous palladium (Pd) catalytic systems were reported for the easy synthesis of biaryls [11–17]. However, it is very difficult to separate the catalyst and ligands from the final product in these systems. On the other hand, to address this problem, researches are focused on heterogeneous system, which fecilitates the easy separation and recovery of the Pd-catalysts [18,19]. Though, the catalytic activity is slightly reduced when the recycled catalyst had employed, but, giving priority to benignity, heterogeneous catalytic systems significantly dominates homogenous catalytic systems.

In organic transformations, metal nanoparticles (NPs)-based heterogeneous catalysts display outstanding performance, because, metal NPs consists large surface area, which results in a very huge number of catalytically active sites [20]. Recently, several solid-supported Pd-catalysts have been accounted *via* mild and/or environmentally benign reaction conditions. These supported catalysts were prepared by immobilizing Pd(II) on supported ligands (like phosphine [21], amines [22], *N*-heterocyclic carbene [23], cyclodextrin [24] and metformin [14]) or ligand-free Pd(0) nanoparticles on various solid supports (such as Fe$_2$O$_3$ [25], silica [26], polyionic polymers [27], cellulose [28], metal alloys [29] and fullerene [30]) and bio-reductants [31–33]. These immobilizations or captivity of Pd(0) NPs by various supports elates water-suspend ability, biodegradability and the metal ion uptake capacity for Pd-catalyzed cross-coupling reactions.

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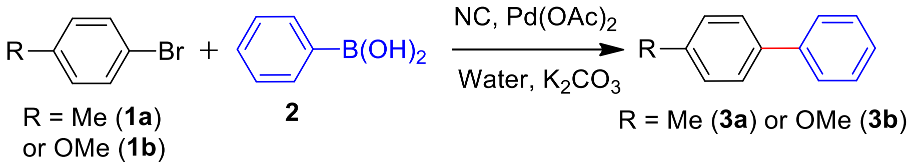
Table 1
Comparison of NC and cellulose in aqueous $Pd(OAc)_2$-catalyzed Suzuki reaction.[a]



Entry	R	Support	Temp (°C)	Time (h)	Isolated yield (%)
1	4-CH_3	NC	rt	5	99
2	4-CH_3	Cellulose	rt	6	70
3	4-OCH_3	NC	rt	5	98
4	4-OCH_3	Cellulose	rt	6	74

[a] Reaction conditions: aryl bromide (0.4 mmol), phenylboronic acid (1.2 eq.), support (1 wt%), $Pd(OAc)_2$ (0.1 mol%), K_2CO_3 (2 eq.) and water (3 mL).

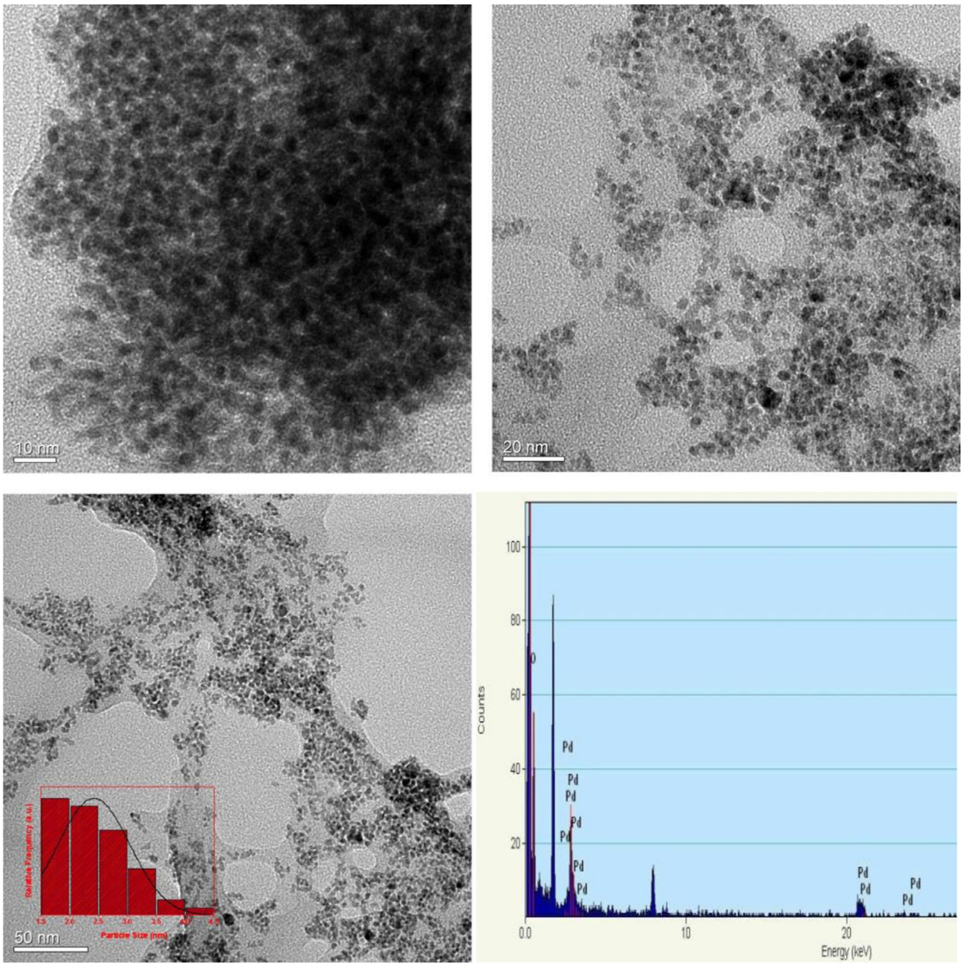
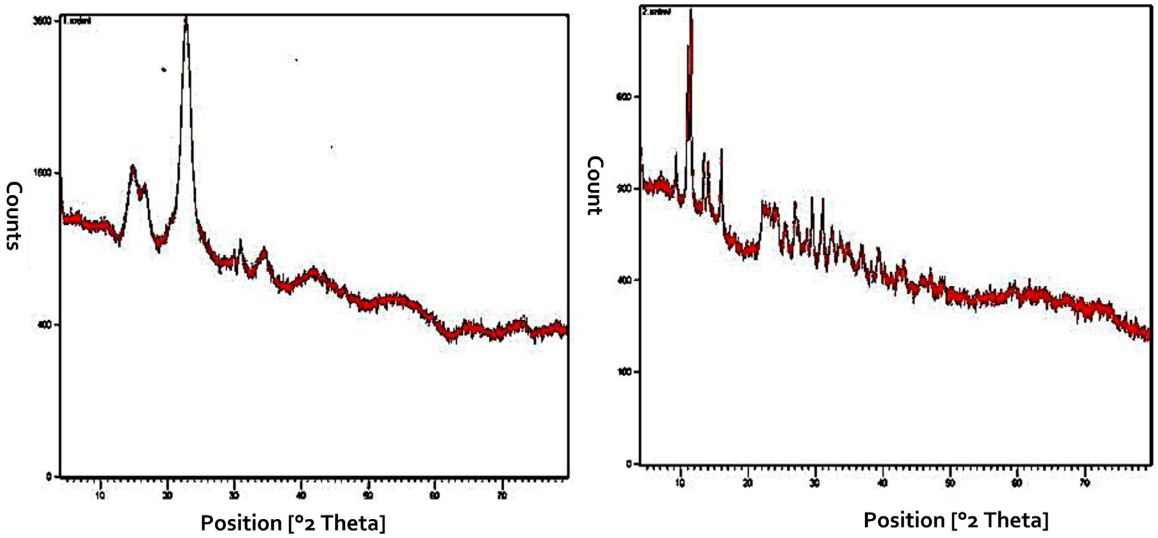


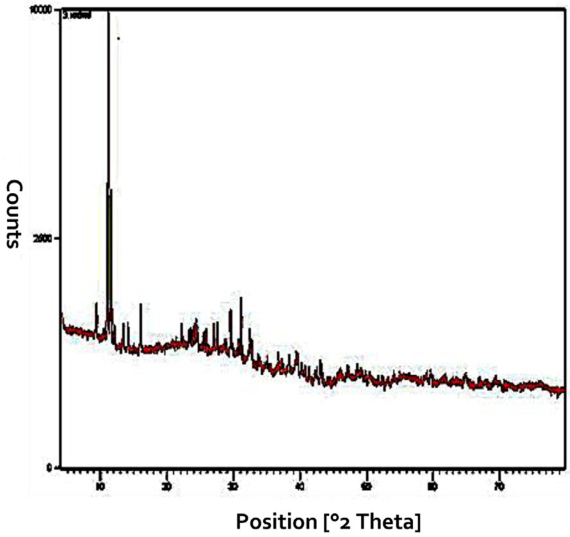
Fig. 1. TEM images and EDX of Pd@NC NPs.

The high health concern, safety and environmental regulations of today, promotes usage of safer solvents towards the reduction of environmental risks [34]. The application of aqueous media in organic transformations has been focused at extreme exploration. In addition, many organic chemists have prepared significant attempts to replace the organic solvents in organic synthesis with aqueous media owing to their safe, eco-friendly and non-toxic properties [35–39]. Saikia et al. used 'water extract of rice straw ash' and 'water extract of banana' in $Pd(OAc)_2$-catalyzed Suzuki coupling reaction under base/ligand/promoter/organic co-solvents-free conditions at room temperature (rt) [40,41]. Recently, Venkateswarlu et al. reported aqueous extract of pomegranate ash mediated $Pd(OAc)_2$ or Pd-KIT-6-catalyzed Suzuki cross-coupling reaction [42]. The development of novel/new catalytic systems for Suzuki coupling reaction in aqueous media is highly desirable due to the environmental, economical and health concerns.

Cellulose is the best option to be used as a support in chemical transformations, because it is a sustainable, cost-effective, and most abundant natural biopolymer [43,44]. Xiang et al. introduced a highly recyclable dip-catalyst produced from PdNPs-embedded on bacterial cellulose and plant fibers for Suzuki reaction [45]. Baruah et al. synthesized cellulose supported PdNPs using hearth wood extract of *Artocarpus lakoocha* and used as sustainable heterogeneous catalyst for microwave assisted Suzuki coupling in wa-



Nanocellulose		**Pd(OAc)₂**



Nanocellulose + Pd(OAc)₂

Fig. 2. Powder XRD spectra.

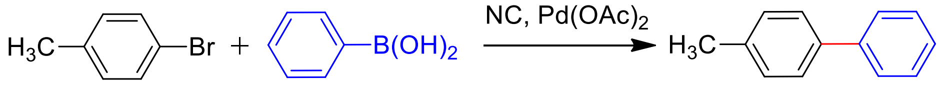
ter [46]. Applications of NC supported metal NPs have gained considerable attention in the past decade [47] for the reason that NC has a high surface area [48], water suspend ability, thermal stability, reductive surface functional groups and superior mechanical strength [49]. Further, NC shows high stability in water is an important property that provides good stability to catalysts when it is used as support. Opening a greener approach for metal NP synthesis, NC with hydroxy and sulfate ester groups display the ability to reducing metal species, without the use of any external reducing agents. The high crystalline order of NC may contribute in catalytic actions. Thus, we aimed in developing a new green protocol using NC as biocompatible support for Pd-catalyzed Suzuki reaction and found that the *in situ* formation of nanocatalyst which can be separated and reused for several times. The results are discussed in the following section.

2. Results and Discussion

NC was prepared from the waste cotton cloth according to the procedure reported by Wang et al. [50]. NC has been investigated for the $Pd(OAc)_2$-catalyzed Suzuki reaction and the results are listed in Table 1. A test reaction between 4-bromotoluene (**1a**) (0.4 mmol) and phenylboronic acid (**2**) (1.2 eq.) was conducted using NC (1 wt%), $Pd(OAc)_2$ (0.1 mol%) and K_2CO_3 (2 eq.) in 3 mL water. The formation of 99% of biaryl, **3a** was observed at rt in 5 h (Table 1, entry 1). The same reaction was also conducted using cellulose and found only the formation of 70% of **3a** in 6h (Table 1, entry 2). Similarly, the reaction of 4-bromoanisole (**1b**) with **2** using NC and cellulose under the above conditions shows the formation of coupling product, **3b** in 98% and 74% in 5 h and 6 h (Table 1, entries 3,4). These results indicates that the NC as best support than cellulose because of the small size and better adsorption and adhesive properties of NC.

The NC-based Suzuki reaction has further been screened towards the optimization of the reaction conditions using **1a** (0.4 mmol), **2** (0.48 mmol, 1.2 eq.) in 3 mL of water (Table 2). By using the bases such as Cs_2CO_3, Na_2CO_3, Et_3N, KF, NaOH and KOH the yields of **3a** was observed in 50–90% at rt in 5–8 h (Table 2, entries 1–6), indicates that the K_2CO_3 is the best choice (Table 1, entry 1) among the studied bases under present conditions. The model reaction is then screened at 60 °C, 70 °C, 80 °C and 90 °C and found the formation of 99% of yields in 3 h, 1.5 h, 1 h and 1h (Table 2, en-

Table 2
Screening for experimental conditions.[a]



1a + **2** → **3a**

Entry	Base (2 eq.)	Temp (°C)	Time (h)	$Pd(OAc)_2$ (mol%)	Isolated yield (%)
1	Cs_2CO_3	rt	7	0.1	79
2	Na_2CO_3	rt	6	0.1	90
3	Et_3N	rt	8	0.1	65
4	KF	rt	7	0.1	50
5	NaOH	rt	6	0.1	75
6	KOH	rt	5	0.1	77
7	K_2CO_3	60	3	0.1	99
8	K_2CO_3	70	1.5	0.1	99
9	K_2CO_3	80	1	0.1	99
10	K_2CO_3	90	1	0.1	99
11	K_2CO_3	80	6	0.05	55
12	K_2CO_3	80	1	0.15	99

[a] Reaction conditions: **1a** (0.4 mmol), **2** (1.2 eq.), NC (1 wt%), base (2 eq.) and water (3 mL).

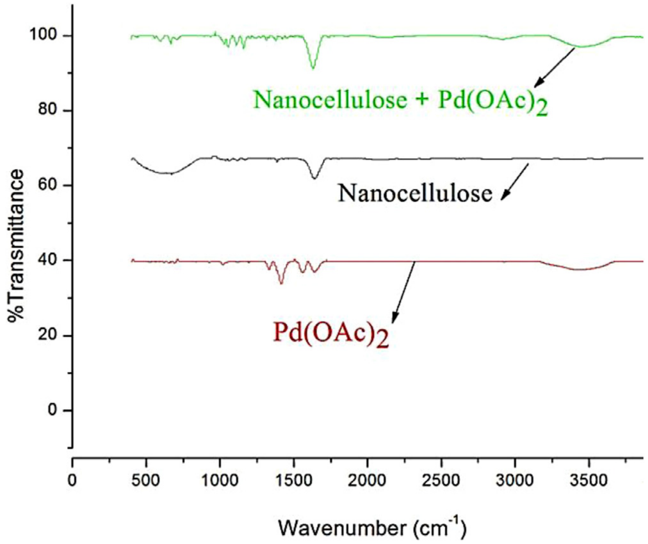


Fig. 3. FT-IR Spectra.

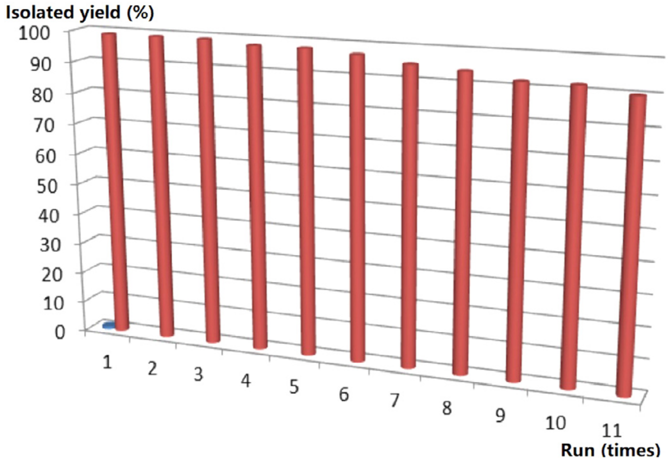


Fig. 4. Recyclability study (Series 1 = no of times; Series 2 = percentage of product).

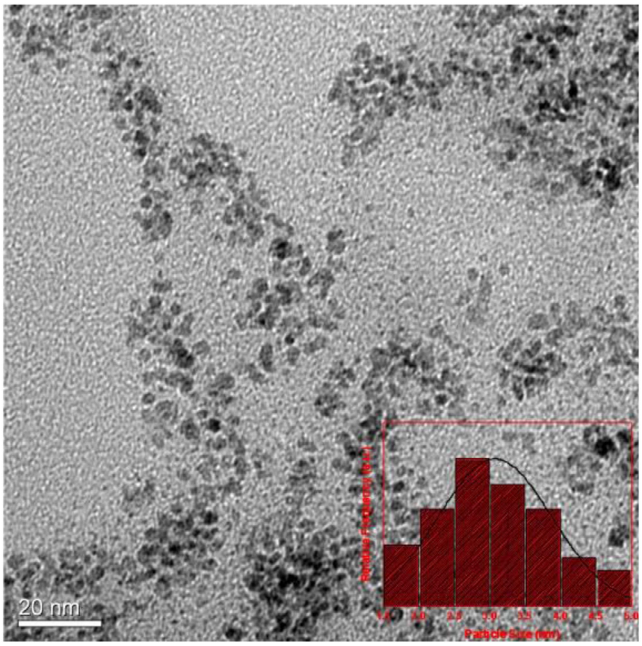
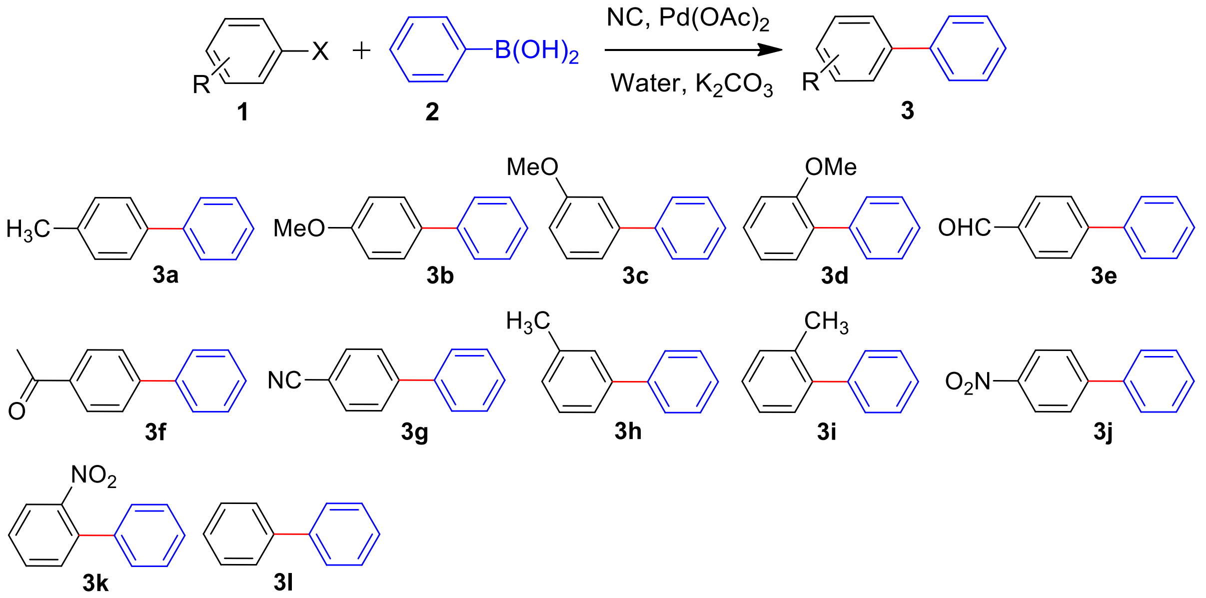


Fig. 5. TEM image of recycled Pd@NC NPs.

tries 7–10) and indicated that the reaction can be conducted conveniently at 80 °C. The catalyst loading was also screened using 0.05 mol% and 0.15 mol% of $Pd(OAc)_2$ and identified the formation of **3a** in 55% and 99% of yields in 6 h and 1 h at 80 °C (Table 2, entries 12,13) suggested the requirement of 0.1 mol% of catalyst (Table 2, entry 9) for the current protocol.

With the above optimized conditions, we have utilized the current NC-based Suzuki coupling reaction to a wide range of aryl halides (Table 3). The aryl bromides with both the electron releasing (methyl and methoxy) and electron withdrawing (formyl, acetyl and nitrile) groups shows high yields of biaryls (**3a–3g**) (90–99%) in 1–4 h (Table 3, entries 1–7). The Suzuki coupling reaction of aryl iodides has observed to be quick on comparison with aryl iodides and the reaction proceeds at 60 °C (Table 3, entries 8–17). The aryl iodides with different substituents such as methyl, methoxy, acetyl and nitro groups showed 90% - nearly quantitative yields in just 0.4–1 h (Table 3, entries 8–16). Unsubstituted aryl iodide such as iodobenzene also showed high yield (95%) of biphenyl (3l) in 0.4 h (Table 3, entry 17). The position and nature of substituent on aryl bromide or aryl iodide display negligible effect on the present Suzuki coupling.

Table 3
Substrate scope.[a]



Entry	X	R	Time (h)	Product	Isolated yield (%)	Mp (literature values) (°C)	TON[d]	TOF (h^{-1})[e]
1	Br	4-CH$_3$	1	**3a**	99	44–45 (42–44 [51])	990	990
2	Br	4-OCH$_3$	1	**3b**	99	80–81 (80–82 [51])	990	990
3	Br	3-OCH$_3$	2	**3c**	95	Oil (Oil [52])	950	475
4	Br	2-OCH$_3$	2	**3d**	92	Oil (Oil [53])	920	460
5	Br	4-CHO	3	**3e**	94	58–59 (56–58 [51])	940	313.3
6	Br	4-COCH$_3$	4	**3f**	90	116–117 (117–118 [54])	900	225
7	Br	4-CN	4	**3g**	90	84–85 (85–86 [51])	900	225
8[b]	I	4-CH$_3$	0.4	**3a**	99	44–45 (42–44 [51])	990	2475
9[b]	I	3-CH$_3$	0.4	**3h**	90	Oil (Oil [55])	900	2250
10[b]	I	2-CH$_3$	0.4	**3i**	90	Oil (Oil [52])	900	2250
11[b]	I	4-OCH$_3$	0.4	**3b**	99	80–81 (80–82 [51])	990	2475
12[b]	I	3-OCH$_3$	0.4	**3c**	95	Oil (Oil [53])	950	2375
13[b]	I	2-OCH$_3$	0.4	**3d**	90	Oil (Oil [54])	900	2250
14[b]	I	4-COCH$_3$	1	**3f**	92	116–117 (117–118 [55])	920	920
15[b]	I	4-NO$_2$	1	**3j**	90	110–111 (111–113 [51])	900	900
16[b]	I	2-NO$_2$	1	**3k**	90	37–38 (37–38 [56])	900	900
17[b]	I	H	0.4	**3l**	95	65–66 (67–68 [51])	950	2375
18[c]	Cl	4-CH$_3$	8	**3a**	99	44–45 (42–44 [51])	990	123.8
19[c]	Cl	4-OCH$_3$	8	**3d**	92	80–81 (80–82 [51])	920	115

[a] Reaction conditions: aryl halide (0.4 mmol), phenylboronic acid (1.2 eq.), NC (1 wt%), Pd(OAc)$_2$ (0.1 mol%), K$_2$CO$_3$ (2 eq.) and water (3 mL) at 80 °C.
[b] Reaction has been conducted at 60 °C.
[c] Reaction has been conducted at 100 °C
[d] TON = Turnover number; number of moles of product formed/number of moles of catalyst used.
[e] TOF = Turnover frequency; number of turnovers per hour.

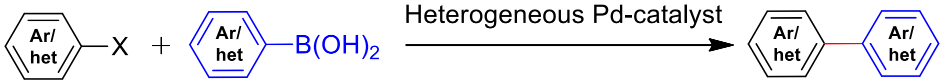
Further, the least reactive of aryl halides such as aryl chlorides showed excellent yields of coupling products under the present experimental conditions. In this connection, 4-chlorotoluene and 4-chloroanisole on reaction with phenylboronic acid at 100 °C formed 99% and 92% of biaryls, **3a** and **3b** (Table 3, entries 18,19). The good reactivity of aryl chlorides is an additional advantage of this method.

The effective reactivity of aryl halides with phenylboronic acid under the current reaction conditions is due to the *in situ* formation of PdNPs on NC (Pd@NC NPs) and these NPs are separated from the reaction mixture (see at supplementary material for the separation procedure) was characterized using TEM, EDX, powder XRD and FT-IR analysis.

The TEM images of Pd@NC NPs (Fig. 1) showed the formation PdNPs on the surface of NC and the average size of Pd was observed as 2.46 nm. The EDX (Fig. 1) confirms the only Pd that is available on the NC. The powder XRD studies (Figure 2) also reveals that the NC source adsorbed Pd, from the graph it is understood that both are in same form. Main diffraction peaks indicates at 39.9, 46.4, 67.5, which can be assigned to (1 1 1), (2 0 0), and (2 2 0) planes of the fcc Pd. The FT-IR spectra (Figure 3) also suggest that the availability of Pd on NC.

The separated Pd@NC NPs was also been studied for the study of reusability using **1a** and **2** as substrates (see at supplementary material for the reusability experiment) and the results are showed in Fig. 4. The *in situ* generated Pd@NC NPs showed a fair reusability up to 11 cycles and showed 89% of the product, **3a** in 11th cycle (Fig. 4). Generally, the low amounts of Pd catalysts are difficult in the separation, however, these Pd@NC NPs are found easy in separation and reuse. The fair reusability of Pd@NC NPs indicates high stability of PdNPs on NC support. Further the high stability of PdNPs on NC support was also confirmed by hot-filtration experiment (see supplementary material). The ICP-OES analysis of the aqueous portion after the separation of the catalyst was also showed no 'Pd' species and hence the PdNPs on the surface of NC are observed to be highly stable. The TEM analysis of recycled catalyst (Fig. 5) also indicated the negligible change in the size of Pd species on NC surface.

Table 4
Comparison of some advanced heterogeneous Suzuki reactions with the present method.



Catalyst	Conditions	Reference	Remarks
Cu-Adenine@boehmite	Na_2CO_3, H_2O, 80 °C	[51]	Requirement of nitrogen-based ligand is critical. The catalyst was synthesized using multiple steps.
Pd-imi-CC@MCM-41/Fe_3O_4	Na_2CO_3, PEG-400, 80 °C	[52]	This method involves multi-step preparation of catalyst, use of PEG-400 as medium and necessity of large reaction times in case of aryl chlorides.
Pd-TBA@biochar	Na_2CO_3, PEG-400, 120 °C	[53]	Requires PEG-400, large reaction time for aryl chlorides and multi-step preparation of ligand based catalyst.
Pd-imi@MCM-41/Fe_3O_4	Na_2CO_3, PEG-400, 80 °C	[57]	Multi-step preparation of the catalyst, requirement of PEG-400 as medium and necessity of large reaction times with aryl chlorides are reported in this method.
Pd-isatin-boehmite	K_2CO_3, PEG-400, 80 °C	[58]	Complexicity in the preparation of the catalyst and necessity of PEG-400 as media are reported in this protocol.
Pd-adenine@boehmite	Na_2CO_3, water, 80 °C	[59]	The synthesis of Pd-complex-based catalyst needs multiple steps.
Pd-ABA-MCM-41	PEG, K_2CO_3, 60 °C	[60]	Requirement of PEG as reaction media and multi-step preparation of the catalyst are the key features of this protocol.
Pd@rice husk ash	EtOH, K_2CO_3, 100 °C	[61]	Requirement of ethanol as reaction media, large reaction times are the decisive in this protocol.
GO-NH_2-Pd^{+2}	EtOH, water K_2CO_3, 60 °C	[62]	Requirement of 4 eq. of base is critical and very low yields are reported with aryl chloride, even after large reaction time (24 h).
GO-Pd@Ag-AgBr	EtOH, water K_2CO_3, vis. light, 25 °C	[63]	Very low yield of coupling product using aryl chlorides was observed in this protocol.
GO-NHC-Pd^{+2}	EtOH, water K_2CO_3, 80 °C	[64]	Low yields of the coupling products was reported using aryl chlorides (in 24 h) using this protocol.
Zn free MOF-5-NPC-900-Pd	EtOH, water K_2CO_3, rt	[65]	Requirement of 3 eq. of base and multi-step preparation of catalyst are crucial in this protocol.
Pd@NC NPs	Water, K_2CO_3, 60–100 °C	Present method	*In situ* preparation of catalyst, use of water as reaction media and avoid of ligand are the advantages of this protocol.

A systematic comparison of some advantageous heterogeneous Suzuki coupling protocols with the present method has been provided in the Table 4. As can be seen the present protocol evidences the advantage as *in situ* formation of PdNPs on NC (Pd@NC NPs) as heterogeneous catalyst over the reported methods that are considered for the comparison. It also shows significance by avoiding ligand and utilizing water as reaction media. Since, the NC used in this protocol is produced from the waste cotton cloth, it is a very good example for the waste to wealth approach in chemical synthesis.

3. Conclusion

In conclusion, we have developed a sustainable and convenient Suzuki coupling reaction using *in situ* formed Pd@NC NPs [from NC and $Pd(OAc)_2$] as heterogeneous catalyst without the aid of external reducing agents and ligand in water. The *in situ* generated catalyst, Pd@NC NPs has been systematically characterized using TEM, EDX, powder XRD and IR techniques. The successful reusability (up to eleven tested cycles) is an added advantage of this finding. The application of *in situ* generated catalyst, use of water as reaction media, heterogeneous catalytic conditions, preparation of catalyst from the waste cotton cloth, high scope of reusability of the catalyst, easy separation of the catalyst by simple filtration and avoid of toxic ligands or volatile organic solvents are the striking and sustainable assets of this protocol.

Declaration of Competing Interest

Authors declare no known competing personal relation or financial interest that could appear to influence the work presented in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2021.121719.

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