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Regioselective Direct C2 Arylation of Indole, Benzothiophene and Benzofuran: Utilization of Reusable Pd NPs and NHC-Pd@MNPs Catalyst for C–H Activation Reaction

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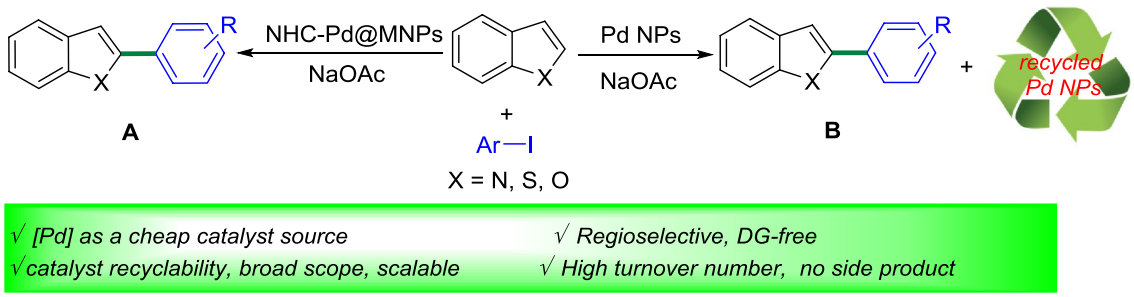
Abstract
A regioselective C2 arylation of indoles, benzothiophene and benzofuran without directing group has been accomplished using economically cheap Pd NPs and NHC-Pd@MNPs catalyst. The reusable catalyst is efficiently employed to access C2 arylated heterocycles in good to excellent yield. The reusability of the catalyst is studied up to five cycles and a gram-scale synthesis has been achieved. The reaction mechanism is well supported by control experiments and literature precedents.

Grapic Abstract



Keywords Heterogeneous catalysis · C2 arylation · Pd NPs · NHC-Pd@MNPs · Catalyst recyclability

1 Introduction

The direct arylation of heteroaryl compounds via C–H bond activation has emerged as a powerful synthetic strategy, as it reduces additional steps associated with pre-functionalized coupling partners (For selected review, see [1–11]). Nevertheless, indole, benzothiophene and benzofuran are the heterocyclic core commonly found in natural products and pharmaceuticals (For selected review, see [12, 13]) for various biological activities (Fig. 1) (For selected examples, see [14–17]).

Therefore, the formation of aryl-heteroaryl bonds has been focus of intensive research for developing numerous methodologies and depicts several known synthetic strategies used for constructing the heterocyclic molecules (Scheme 1a). For instance, coupling of aryl metal with heteroaryl or arene with heteroaryl metal reagent [18–25] (Route I); cross-coupling of heteroaryl and arene (Route II) [26–29] coupling of aryl halide with heteroaryl metal reagent or aryl metal with heteroaryl halide (For reviews, see [30–33]) (Route III) and coupling of aryl halide with heteroaryl or arene with heteroaryl halide (Route IV) [34–43].

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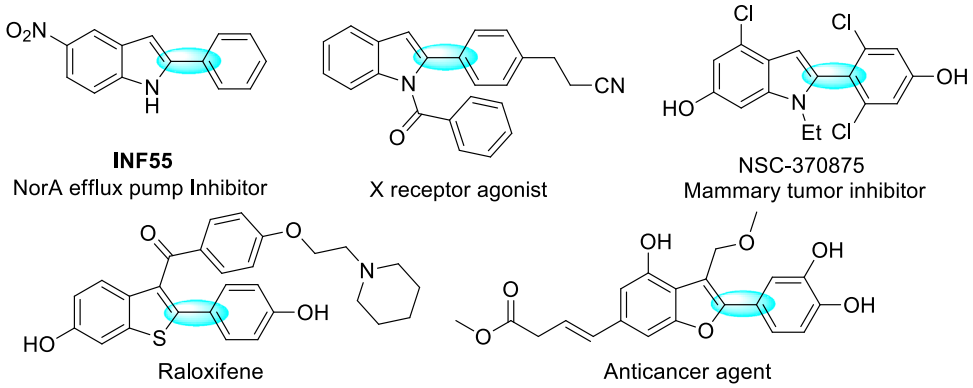
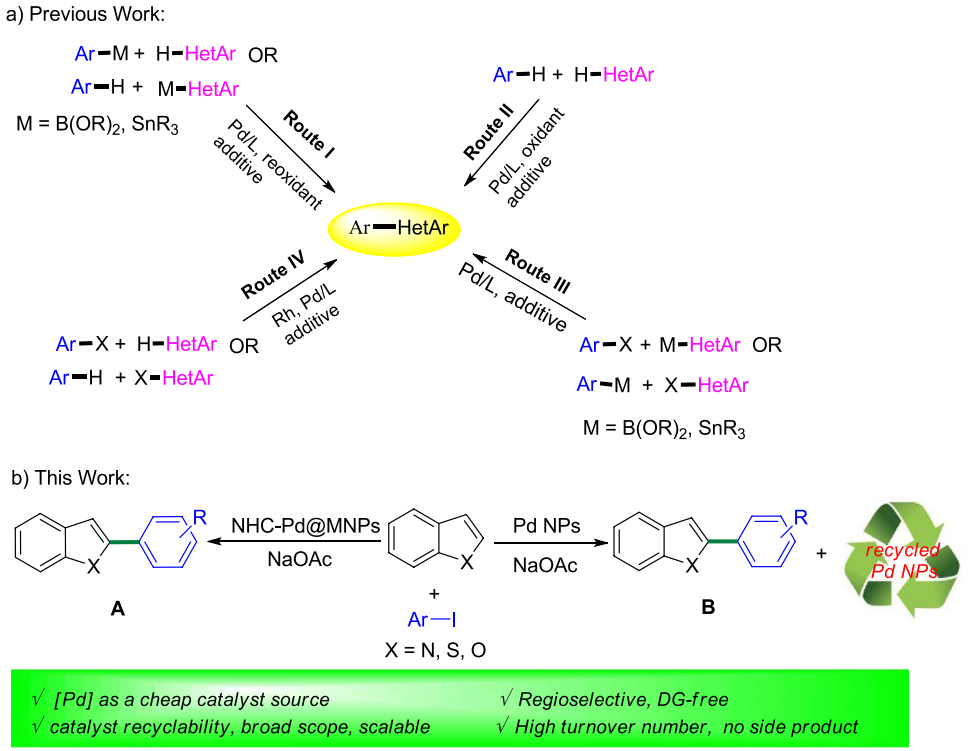


Fig. 1 Selected examples of natural products and biologically active molecules



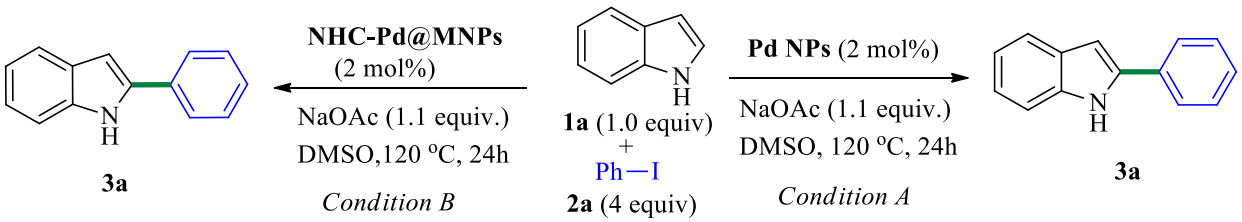
Scheme 1 Evolution of aryl-heteroaryl coupling reaction

Despite of the significant advantages, these reported methods are still suffering from several issues of employing stoichiometric metallic reagents, expensive catalyst (e.g. Pd, Rh, and Ru etc.) and ligands. Importantly, all of these reactions are performed in homogenous manner, which suffered from the recyclability of catalyst and metallic contamination. Our extensive literature search shows that the development of transition metal-catalysed C–H activation, particularly C2-selective arylation of indole, benzothiophene and benzofuranis is developed preferably with homogeneous catalysis ([44–46, For electrophilic metalation–migration see 47, For directing groups assisted reaction see 48–53]. However, despite of several reports (For C2 arylation of indole by Pd NPs see [54–61]), the heterogeneous catalyst for such a transformation are comparitively less explored. Therefore,the new paradigm for the development of heterogeneous catalyst to address the C2 functionalization at heterocycles would be highly desirable from the perspective of synthesis. In this context, we are particularly interested into developing heterogeneous support (For examples, see [62–67]) in a greener set-up (For C2 arylation with under green condition, see [68–72]) as possible contemporary

method to address C–H arylation of heterocycles. Along this line, we successfully discloseregioselective C–H arylation of (N–H free) indole, benzothiophene and benzofuran with iodoarenes using economically cheap palladium nanoparticles (Pd NPs) and N-heterocyclic carbene-palladium magnetic nanoparticles (NHC-Pd@MNPs) catalyst [73–78]. It is worth mentioning that, the phytochemicals present in black pepper extract plays a dual role for reducing the Pd^{II} to Pd^{0} and also act as stabilizing agents for Pd NPs (See the SI for schematic representation). The wide range of C2 substituted indoles, benzothiophene and benzofuran can be transformed effectively to the corresponding coupling products without installing additional auxiliary directing group onto substrates. Importantly, the concept on the recyclability of nano-catalyst is demonstrated in this work.

2 Experimental Section

2.1 Experimental Procedure for the Synthesis of 2-Phenyl-1H-indole (3a)



Condition A The DMSO (3 mL) were added in to the oven dried 25 mL R.B.F containing compound **1a** (58.57 mg, 0.5 mmol, 1 equiv), followed by addition of NaOAc (45.11 mg, 1.1 equiv), Pd NPs (2.39 mg, 2 mol% Pd content: 44.48 w/w), iodobenzene **2a** (408.02 mg, 2 mmol, 4 equiv). The reaction mixture was stirred at 120 °C for 24 h, after complete conversion of starting material (indicated by TLC), the reaction was quenched with water and the organic layer was extracted with EtOAc (25 × 3) the combined organic layer was dried over anhydrous Na_2SO_4 the solvent was evaporated by rotary evaporator and crude compound was purified by column chromatography (eluent: 2% EA/Hexane) to get the compound 2-phenyl-1H-indole (**3a**, 88.01 mg, 92%).

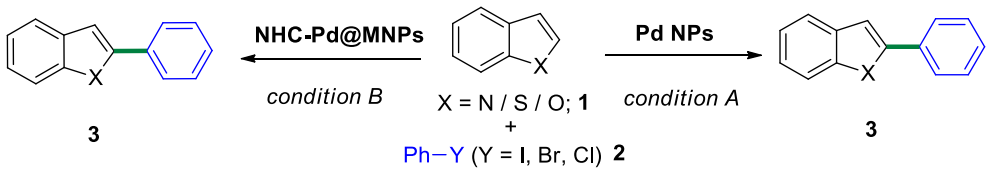
Condition B The similar experimental procedure was followed for condition B using NHC-Pd@MNPs (17.62 mg, 2 mol%, Pd content: 6.04 w/w) catalyst to get the compound 2-phenyl-1H-indole (**3a**, 85.14 mg, 89%).

3 Results and Discussion

3.1 Table 1

We started our exploration in accessing the regioselective C–H arylation of 1-methyl-*1H*-indole (**1a**) with iodobenzene (**2a**). We selected Pd NPs [73] (Pd content: 44.48 w/w, analysed by ICP-OES) as a catalyst and K_2CO_3 as a base (Table 1, condition A). At room temperature, no reaction was observed even at longer reaction time (entry 1). Increasing the reaction temperature to 70 °C in 18 h led to formation the product **3** in 35% yield, which is purified by column chromatography and its identity was further confirmed by ^{1}H NMR spectroscopy with singlet signal appeared at 7.20 ppm, indicative of C2-arylation over C3 [73, 75] (entry 2). Bases such as sodium carbonate, cesium carbonate, sodium acetate and potassium acetate furnished low to moderate yield (entries 3–6), while sodium hydroxide and potassium hydroxide were not effective (See the SI for more details). Keeping sodium acetate as an optimum base, we have screened numerous solvents and found the significant improvement in the reaction efficiency with DMSO (entries 9 and 10). Other high boiling solvents such as DMF, DMA and toluene are ineffective in this methodology (See the SI for more details). Encouragingly, such high efficiency and C2 selectivity can still be maintained when the catalyst loading was further reduced to 2 mol % and desired product obtained in 92% yield, albeit in longer reaction time at 120 °C (entry 11). The blank reaction indicated that the coupling reaction failed to proceed in an absence of catalyst (entry 12). Other halide sources such as bromobenzene or chlorobenzene is not suitable in this Pd NPs mediated reaction (See the SI for more details). Whereas this C2-arylation method could be applied to other precursors such as, benzothiophene (**1b**) and benzofuran (**1c**) under the similar reaction condition in 86 and 87% yield, respectively (entries 13 and 14).

Table 1 Optimization of reaction condition



S.No	1 (X)	Solvent	Base	T (°C)/t (h)	Yield % (3)[a]	
					Condition A	Condition B
1	=NH (1a)	THF	K_2CO_3	25/36	Trace	Trace
2	=NH (1a)	THF	K_2CO_3	70/18	35	23
3	=NH (1a)	THF	Na_2CO_3	70/18	33	31
4	=NH (1a)	THF	Cs_2CO_3	70/18	29	32
5	=NH (1a)	THF	NaOAc	70/18	57	55
6	=NH (1a)	THF	KOAc	70/18	61	62
7	=NH (1a)	CH_3CN	NaOAc	70/18	47	30
8	=NH (1a)	EtOH	NaOAc	70/18	38	31
9	=NH (1a)	DMSO	NaOAc	70/18	71	68
10	=NH (1a)	DMSO	NaOAc	120/18	80	77
11	=NH (1a)	DMSO	NaOAc	120/24	92	89
12[b]	=NH (1a)	DMSO	NaOAc	120/24	0	0
13	=S (1b)	DMSO	NaOAc	120/24	86	74
14	=O (1c)	DMSO	NaOAc	120/24	87	80

Reaction condition: condition A: 1 (0.5 mmol), 2a (4.0 equiv), Pd NPs (0–4.0 mol%, 44.48 w/w), NaOAc (0–1.1 equiv) solvent, 25–120 °C, 12-32 h. Condition B: 1a (0.5 mmol), 2a (4.0 equiv), NHC-Pd@MNPs (0–4 mol%, 6.04 w/w), NaOAc (0–1.1 equiv), solvent, 12–36 h

[a]Yields after purification from silica gel column chromatography (average of two run)

[b]No catalyst used

3.2 Table 2

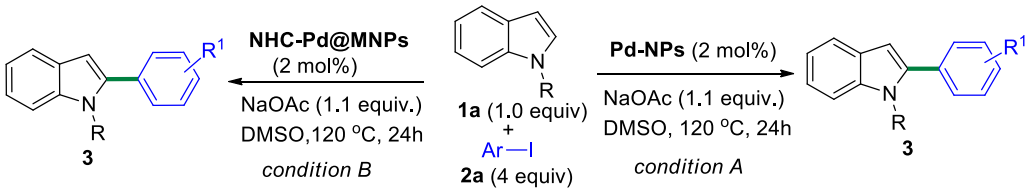
We also examined the viability of this protocol with other heterogeneous catalyst, NHC-Pd@MNPs [78] (Pd content: 6.04 w/w, analyzed by ICP-AES) for similar reaction (Table 1 condition B), which gave a comparable reactivity to the condition A. Thus, optimized reaction condition for (NH-free) indole, benzothiophene and benzofuran is: Pd NPs (2.0 mol%, Pd content: 44.48 w/w, for condition A) and NHC-Pd@MNPs (2.0 mol%, Pd content: 6.04 w/w, for condition B) as a catalyst, iodobenzene (4.0 equiv) as a halide precursor, NaOAc (1.1 equiv) as a base, DMSO as solvent at 120 °C for 24 h.

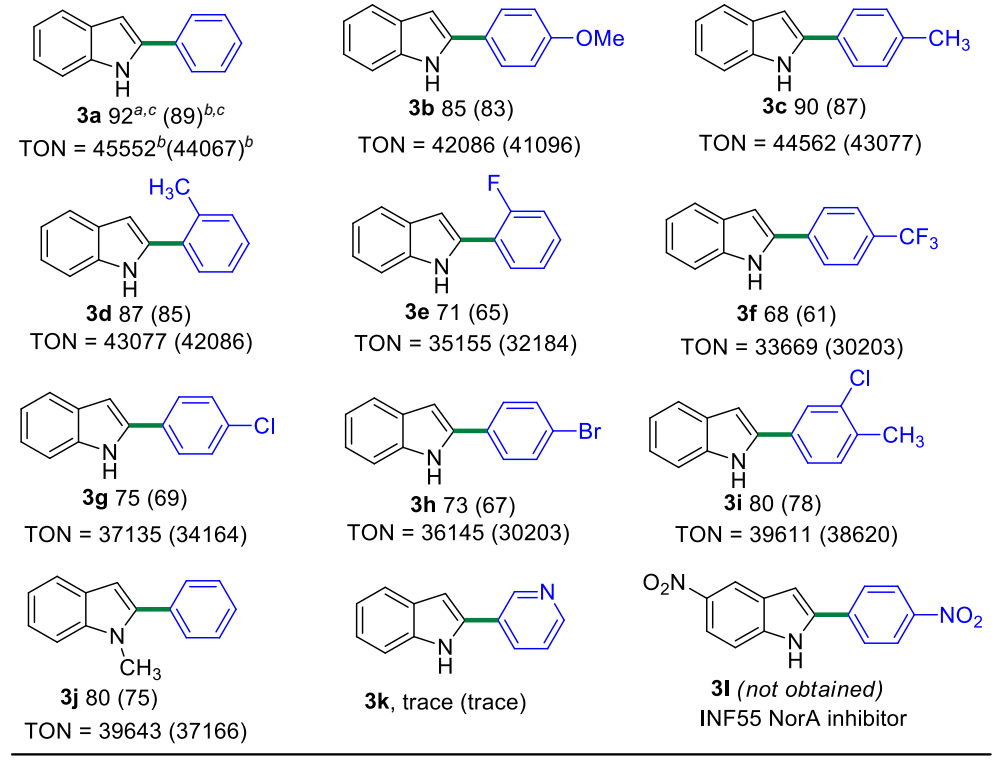
Having gained preliminary insights in to the novel reaction and identified the optimized reaction conditions, we first explored the scope and generality of this process against different aryl iodide **2** with (N–H free) indole (see Table 2 for condition A and B). The aryl iodides bearing electron donating 4-methoxy, 4-methyl and 2-methyl substituents furnishes C-2 arylated products (**3b**–**3d**) in high yield of 85–90% (condition A) and 83–87% (condition B). While, electron withdrawing substituents such as, 2-fluoro (**3e**), 4-trifluoromethyl (**3f**), 4-chloro (**3g**), 4-bromo (**3h**), 3-chloro 4-methyl (**3i**) reacted smoothly, affording C2 arylation product in satisfactory yield of 68–80% (condition A) and 61–78% (condition B). In addition, *N*-methylindole also reacted smoothly that alkyl-substitution does not affect yield and regioselectivity of this synthetic methodolgy. The heteroaryl halide such as 3-iodopyridine are not reactive under both condition, which would be used as precursor to synthesize *INF55 NorA efflux pump inhibitor3l.*

3.3 Table 3

Next, the generality of Pd NPs mediated selective C-H activation of benzothiophenes were also evaluated by exploring different substrates (Table 3). For instance, the benzothiophene reacted with aryl iodides to furnish desired products **3m**–**3t** in 69–85% yield (under condition A) and 58–81% yield. (Under condition B). Similarly, reaction of benzofuran with iodobenzenes bearing different functionality handles achieved expected coupling product **3u**–**3y** in moderate to

Table 2 Scope with aryl halides





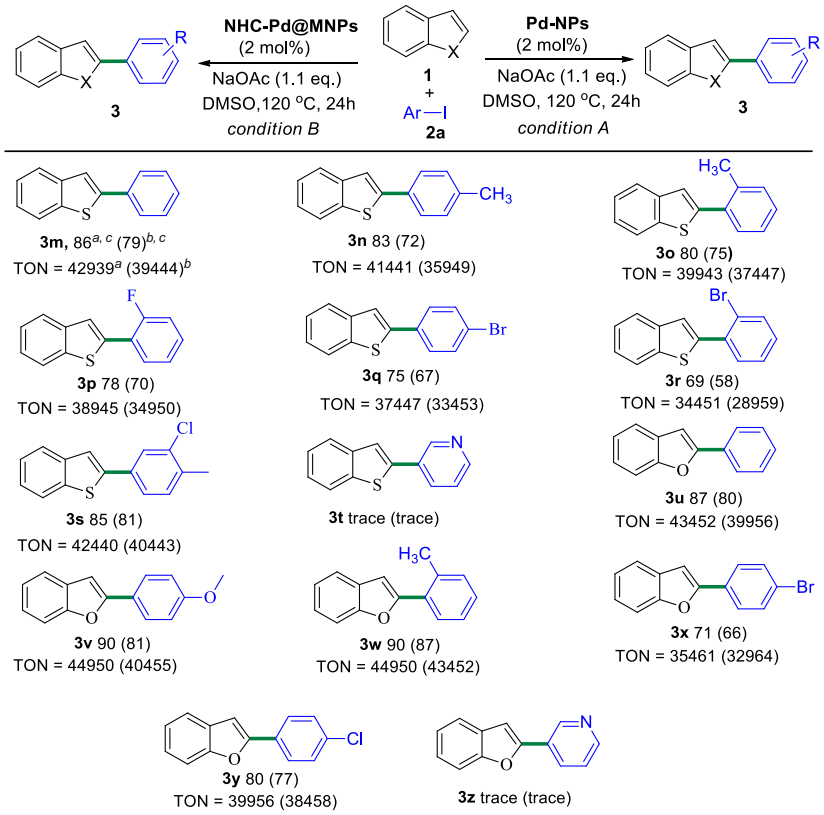
Reaction condition: condition A: 1a (0.5 mmol), 2a (4.0 equiv), Pd NPs (2.0 mol%, 44.48 w/w), NaOAc (1.1 equiv.) DMSO at 120 °C in 24 h. Condition B: 1a (0.5 mmol), 2a (4.0 equiv), NHC-Pd@MNPs (2.0 mol%, 6.04 w/w), NaOAc (1.1 equiv), DMSO at 120 °C in 24 h. [a]Yields/TON using condition A; [b]yields/TON using condition B; [c]yields after purification form silica gel column chromatography (average of two run)

good yields. The use of 3-iodopyridine as coupling partner resulted in no product formation (**3z**).

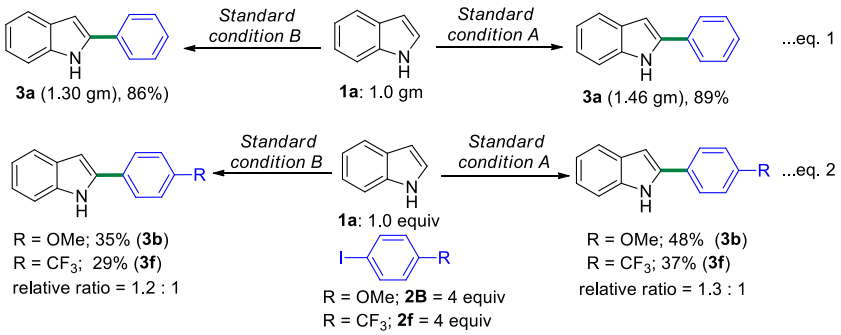
Notably, the catalyst recyclability [73–78] of Pd NPs was found to be more than five times with a significant decreasing in the reaction efficiency for each following reaction, We believed that the decrease in activity of Pd NPs might suffer from the possible particles aggregation caused poor solubility of catalyst in organic solution (See SI for FESEM images). It should be noted that, we are failed to recycle NHC-Pd@MNPs due to its high solubility in organic solution (Fig. 2).

Finally, to test the reaction potential for practical industrial applications, our protocol can be successfully scaled up using indole substrate without affecting the chemical yield and regioselectivity (Eq. 1). To get a better insight about the substituent effect on the rate of the reaction and product formation competitive experiments were performed with electronically biased iodoarenes (OMe and CF_3) with indole (Eq. 2). The competing substrates were taken into account by using equal stoichiometric amounts of substrates in 10 h.

Table 3 Scope with benzothiophene and benzofuran

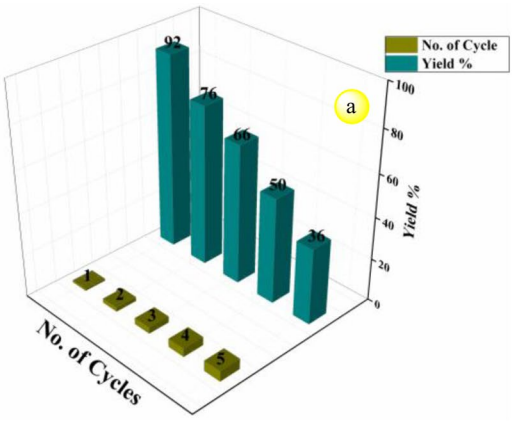


Reaction condition: condition A: 1 (0.5 mmol), 2a (4.0 equiv), Pd NPs (2.0 mol%, 44.48 w/w), NaOAc (1.1 equiv.) DMSO at 120 °C in 24 h. Condition B: 1a (0.5 mmol), 2a (4.0 equiv), NHC-Pd@MNPs (2.0 mol%, 6.04% w/w), NaOAc (1.1 equiv), DMSO at 120 °C in 24 h. [a]Yields/ TON using condition A; [b]yields/TON using condition B; [c]yields after purification form silica column chromatography (average of two run)



The desired products and un-reacted starting materials analysed by TLC revealing the complete conversion of the limiting substrates. It is then separated by flash chromatography and the isolated yields of products indicates that the substituent effect has no significant consequence over the nominal product selectivity, ruling out oxidative addition is the rate-determining step in this reaction [79, 80]. Furthermore, the existence of Pd (0) along with a traces of Pd (II) is clearly investigated in XPS analysis performed in our recent study [77]. It reveals that, phytochemicals inside black pepper extract serves as a ligand for reduction of Pd(II) to Pd(0) and thus, avoiding the need of external ligand [73].

In fact, the traditional mechanistic investigation in C–H functionalization reactions catalyzed by homogeneous palladium catalysts [44–53] were not directly applicable in case of heterogeneous catalysis. Thus, detailed mechanistic understanding for heterogeneous reaction pathways is not completely known. Nevertheless, based on the control



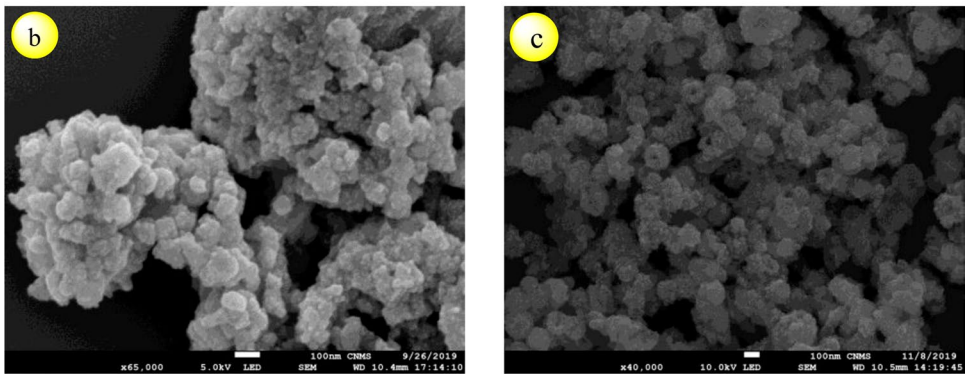
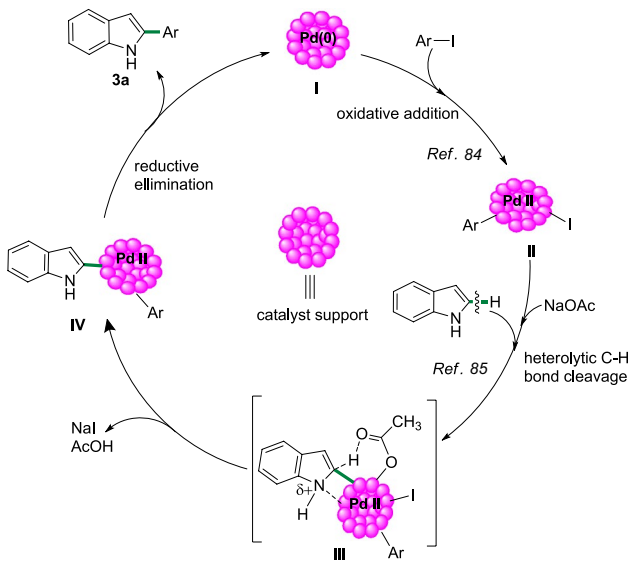


Fig. 2 **a** Recycling efficiency of Pd NPs in C–H arylation. FESEM Images: **b** Fresh Pd NPs, **c** Pd NPs after five cycles



Scheme 2 Plausible reaction mechanism

experiment and literature precedents [37, 54–61, 81–83] plausible mechanism for the regioselective C2 arylation is depicted in Scheme 2. First, oxidative addition of heterogeneous palladium catalyst across aryl iodide forms intermediate **II** [82]**.** The base mediated regioselective C(sp^2)–H bond cleavage/activation occurs heterolytically leading to the formation of **III** in which basic nitrogen of indole is further coordinated to the re-oxidised palladium surface [83]. The subsequent formation of intermediate **IV** followed by reductive elimination liberates the product **3a** with the regeneration of Pd(0) to complete catalytic cycle. The similar reaction pathwaysmay operates for the catalytic cycles in the case of NHC-Pd@MNPs catalyst.

4 Conclusion

In summary, without resorting to directing group instalment on the substrate, we have developed a new catalytic system for the regioselective C2 arylation of indoles, benzothiophene and benzofuran with aryl iodides. This transformation represents practicality of environmental friendly and economically cheap Pd NPs and NHC-Pd@MNPs as a reusable heterogeneous catalyst for regioselective C–H activation reaction. The catalytic method features the complimentry reaction pathways oparating for both the the catalysts highlights an important milestone toward the development of industrially relevant strategy. Ongoing work seeks to gain a detailed mechanistic understanding of the resemblance

offered by Pd NPs and NHC-Pd@MNPs catalysts. In addition, the possibilities to functionalize the C5 position of indoles, benzothiophene and benzofuran are currently under exploration in our laboratory.

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Compliance with Ethical Standards

Conflicts of interest There are no conflicts to declare.

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