



Design and Synthesis of Supramolecular Polymer Network Equipped with Pd-porphyrin: An Efficient and Recoverable Heterogeneous Catalyst for C–C Coupling Reactions

Sheida Hamid¹ · Arash Mouradzadegun¹

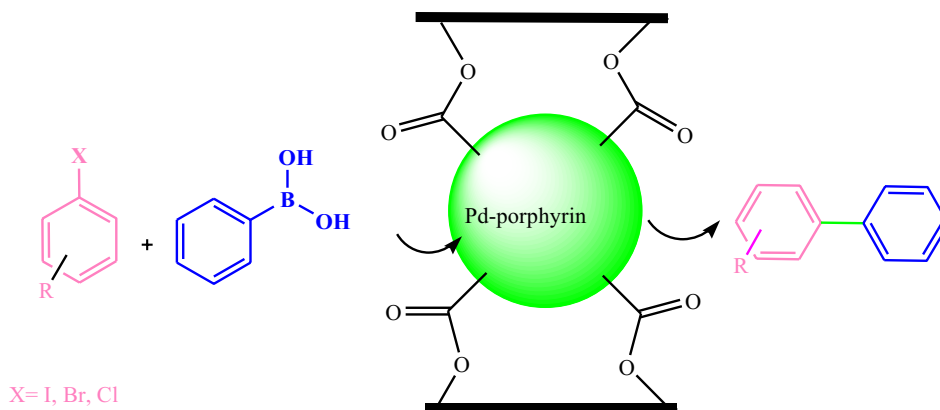
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Abstract

The present study aimed to explain a modern and attractive protocol for synthesizing hypercross-linked conjugated supramolecular polymer network contains palladium(II) porphyrin based on calix[4]resorcinarene as an efficient heterogeneous catalyst. Thus, characterizations were conducted by using spectroscopic methods including powder X-ray diffraction, energy dispersive spectroscopy, scanning electron microscopy, FT-IR, and UV–Vis spectroscopy. In addition, catalytic activity of Pd-porphyrin@polymer was evaluated for C–C coupling reactions. The catalyst demonstrated an excellent activity, which is highly potential for forming new bond under mild conditions.

Graphic Abstract

The synthesis of a novel, green and recoverable heterogeneous catalyst by grafting Pd-porphyrin on the surface of polymeric calix[4]resorcinarene for the synthesis of C–C coupling reactions.



Keywords Supramolecular polymer network · Pd-porphyrin · Suzuki reaction

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1 Introduction

Recently, supramolecular polymers, as a combination of the advantages of supramolecular chemistry and polymeric materials, have attracted a lot of attention [1]. Supramolecular polymers display broad application foregrounds such as stimuli-responsiveness, self-healing, recycling, and degradability due to their robust and adjustable nature [2, 3]. In

addition, network polymerization of the monomer produces supramolecular polymer networks which can prepare the materials with macroscopic robustness [4]. Porphyrins and metalloporphyrins are widely found in nature and play a significant role in the basic mechanisms of vital organisms [5–9]. These systems have been evaluated due to the catalytic use of organic synthesis, electrical light applications, and therapeutic properties. Although they are considered as homogeneous catalysts and their role can be highlighted by selectivity [10, 11], the major problem is related to their recovery. In this regard, immobilized porphyrin was suggested to solve this problem. Liu et al. reported metalloporphyrin supported on SBA-15 as an efficient catalyst for solvent-free Heck reaction [12]. In another study, metalloporphyrin on Dowex 50WX8 and Amberlite IR-120 were reported as recyclable catalysts in the reactions related to C–C coupling [13, 14]. However, the above-mentioned protocols had some drawbacks such as high temperature, low active sites on supporting catalyst, and very long reaction times. Nowadays, supramolecular polymers are considered as the applicable support materials for synthesizing heterogeneous catalysts [15, 16]. Thus, expanding the functionalization of this polymer is considered as an important issue for many scholars related to heterogeneous catalysis. Based on the results of the former studies on c-methylcalix[4]resorcinarene which can be performed by suggesting some different moieties at the upper and lower rim [17], the synergetic impact of functional groups and three-dimensional cavity of scaffolding leads to the creation of novel and attractive properties. In this regard, synthesis of functionalized supramolecular structures through the functional groups which are anchored covalently onto c-methylcalix[4]resorcinarene and its polymers have attracted a lot of attention [18–33]. On the other hand, synthesis of biaryl structural unit can be considered as one of the most fascinating reactions since there are a major number of developed materials encompassing natural, as well as some other synthetic products encompassing biaryl structural units which involved some attractive biological and pharmaceutical features. The cross-coupling reactions which are catalyzed by palladium have changed the chemical industry which is responsible for synthesizing a vast number of supramolecular and natural products, medicine, and agrochemicals [34]. This article explains the synthesis of new functionalized polymeric calix[4]resorcinarene contains covalently anchored metalloporphyrin and its efficiency is evaluated as a heterogeneous catalyst in C–C coupling reactions using the one-pot reaction.

2 Experimental Procedure

2.1 General

All the commercial chemicals which were available purchased from Aldrich and Merck companies and utilized without more purification. The products were determined by their physical constant compared with valid samples. Then, the reactions were monitored and the purity the products was determined by using TLC on silica gel PolyGram SILG/UV 254 plates. In addition, the IR spectra were recorded on a Perkin Elmer Spectrum Version 10.4.4. FT-IR spectrophotometer was done by implementing KBr pellets for the related samples and catalyst with the range of 4000–400 cm^{-1} . Further, a LEO 1455VP scanning electron microscope which operated at 1–30 kV was used for conducting the SEM analyses. Thermal stability of the functionalized catalyst was investigated by NETZSCH STA 409 PC/PG under a nitrogen atmosphere (rate of N_2 z 1 L h⁻¹). Melting points were measured on a Thermo Scientific IA9200 and were uncorrected. In the next procedure, ultraviolet–visible (UV–vis) spectra were measured from a Shimadzu UV-2600 UV–vis spectrophotometer. Further, ¹H and ¹³C NMR spectra were determined in CDCl_3 on a Bruker Advanced DPX 400 MHz spectrometer through utilizing TMS as internal standard. Finally, the heterogeneous catalysts were determined by X-ray powder diffraction (XRD) through utilizing a STOE diffractometer with Cu-K α radiation ($\lambda = 0.15418 \text{ nm}$) at 40 kV and 40 mA.

2.2 Synthetic Procedures

2.2.1 Preparing Meso-tetrakis[4-(methoxycarbonyl)phenyl]porphyrin(1)

Meso-tetrakis[4-(methoxycarbonyl)phenyl]porphyrin was made through modifying A. D. Adler-f. R. Longo method [35]. First, 2.33 mmol of freshly distilled pyrrole and 2.33 mmol of p-methoxycarbonylbenzaldehyde were added in 200 ml acetic acid in a typical reaction. Then, the mixture was refluxed for 4 h and cooled based on the room temperature. The mixture was added to 100 ml methanol in an ice bath after cooling. The purple crystals were filtrated and washed through using methanol and hot distilled water. In addition, column chromatography (Silica gel, ethyl acetate/n-hexane = 20:1 as an eluent) was used for purifying crude product, which resulted in creating a desired purple solid of meso-tetrakis[4-(methoxycarbonyl)phenyl]porphyrin(H_2TMCPP) (39%).

2.2.2 Synthesis of Pd-porphyrin(2)

PdCl_2 (0.135 g) was added into a solution of meso-tetrakis[4-(methoxycarbonyl)phenyl]porphyrin (H_2TMCPP) (0.3 g.) in 100 ml of DMF and refluxed for 12 h. Then, Pd-TMCP was purified by recrystallizing frequently and precipitating from DMF– H_2O solutions [36].

2.2.3 Synthesizing Calix[4]resorcinarene(3)

Calix[4]resorcinarene was made based on the already mentioned procedure [37]. This structure was confirmed by the ^1H NMR (Fig. 1).

Yield 60%; white powder, m.p.: $> 360^\circ\text{C}$; IR (KBr): $\nu = 3000\text{--}3500$ (OH) cm^{-1} ; ^1H NMR (400 MHz, DMSO-d_6):

$\delta = 1.29\text{--}1.30$ (d, 12H, CH_3), 4.43–4.49 (q, 4H, H-5), 6.15 (s, 4H, H-1), 6.76 (s, 4H, H-4), 8.56 (s, 8H, OH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 22.0$ (CH_3), 29.0 (C-5), 102.5 (C-3), 123.5 (C-1), 125.7 (C-2), 152.3 (C-4) ppm.

2.2.4 Synthesizing the 3D-Network Polymer Based on Calix[4]resorcinarene(4)

The polymer was combined by adding 42 mmol of formaldehyde to 14 mmol of the produced calix[4]resorcinarene which were mixed in 40 ml NaOH solution (10%) by considering the room temperature. The produced mixture was heated to 90°C and kept at 90°C for 20 h. Then, the surplus alkali was washed by using the gel produced from cold water. In the next procedure, the gel was kept at 100°C for

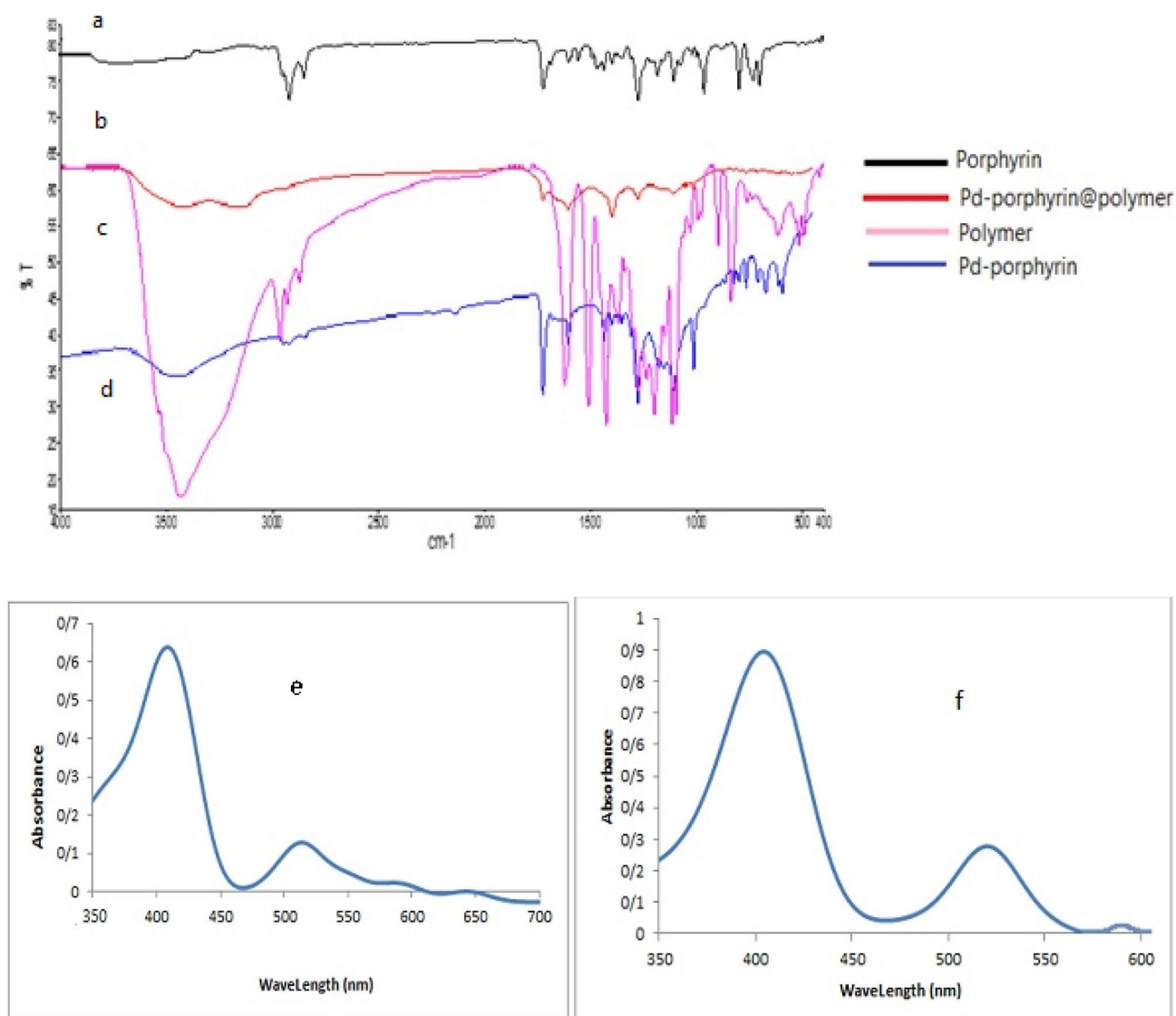


Fig. 1 FT-IR spectra of **a** synthesized porphyrin. **b** Pd-porphyrin@polymer. **c** Polymer. **d** Pd-porphyrin complex and UV-Vis spectrums of Porphyrin (**e**) and Pd-porphyrin@polymer (**f**)

one hour, which changed into the acidic form through treating with the 0.1 M HCl solution. The produced solid was dried at 100 °C for 10 h [38]. In Table 1, elemental analysis was investigated for this polymer. The results shown no nitrogen observed.

2.2.5 Synthesis of Pd-porphyrin@polymer(5)

According to the method reported by Fareghi-Alamdari [39], first, 5 g of polymer and 0.5 g Pd-porphyrin(2) were added in 20 ml DMF after heating at 130 °C for 36 h. Then, the reaction mixture was cooled and filtered. In the next procedure, it was exhaustively washed by using DMF and deionized water. Finally, the prepared catalyst was dried and stored under vacuum (Scheme 1).

2.3 General Method for the Synthesis of Suzuki C–C Coupling

In an ordinary experiment, bromobenzene (0.5 mmol), phenylboronic acid (0.75 mmol), catalyst (20 mg), K_2CO_3 (1.5 mmol) and $H_2O:DMF$ (1:1, 3 ml) were added to a 10 ml balloon and heated at 80 °C. Then, they were reacted for the necessary time. The solid was extracted with ethyl acetate, along with concentrating the liquid. In addition, the product was created through preparing TLC. Further, Pd-porphyrin@polymer was separated by centrifugation in order to recover the catalyst, and accordingly washed with ethanol, along with water. Additionally, it was dried under the decreased pressure. Finally, the catalyst was utilized by fresh charge of solvent and reactant for a subsequent reaction under the same conditions.

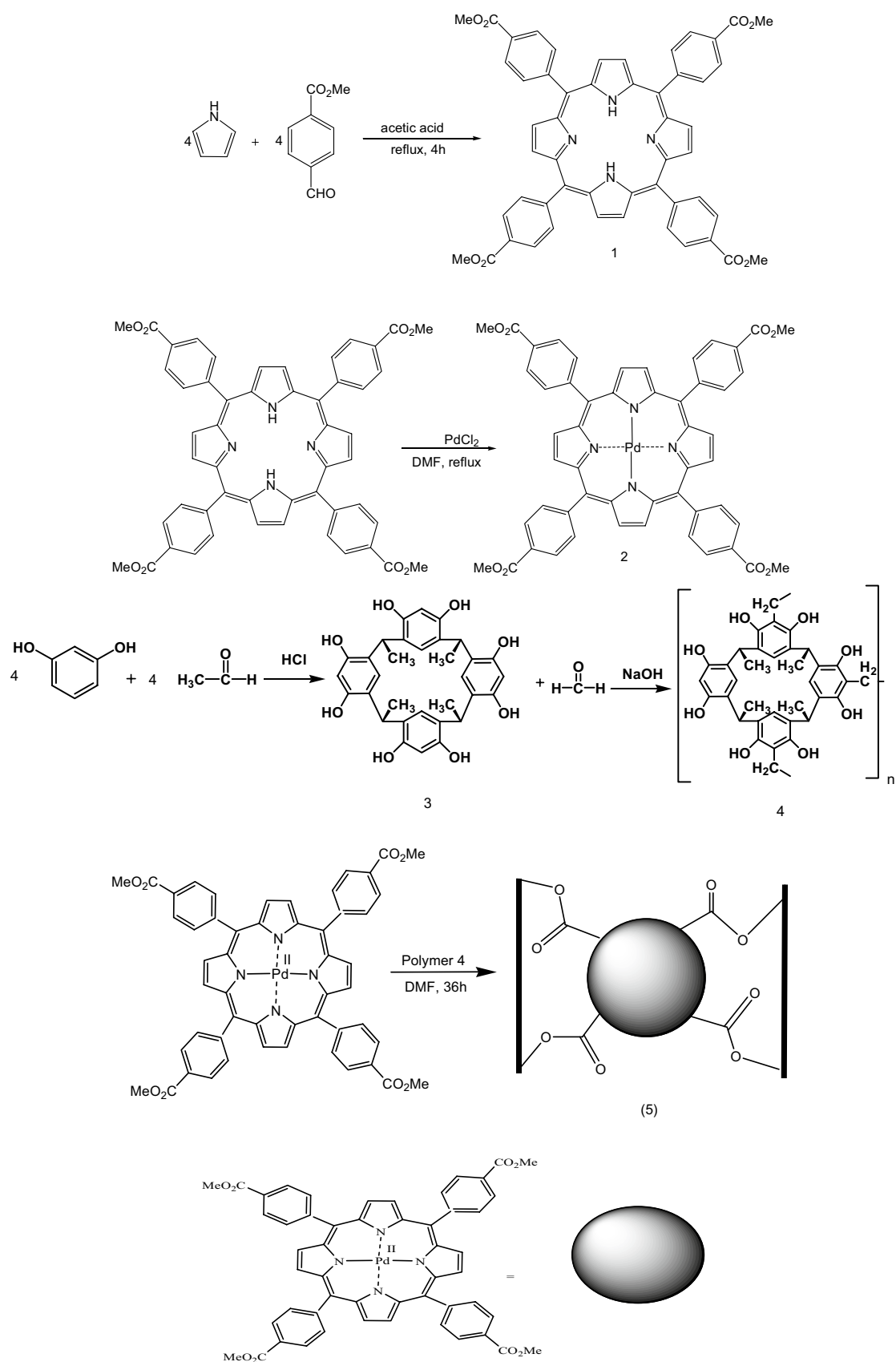
3 Results and Discussion

As it was already mentioned, one of the most important challenges in modern chemistry is related to the search for more efficient catalytic systems which may integrate the advantages of both homogeneous and heterogeneous catalysis. Thus, Pd-porphyrin was immobilized onto a 3D-network polymer based on calix[4]resorcinarene. First, the porphyrin ligand was synthesized based on the results of the previous studies [35]. Then, the porphyrin ligand was metalated utilizing $PdCl_2$ and attached onto modified network polymer through creating the covalent bonding between the carboxylic acid group of Pd-porphyrin and the hydroxyl group related to network polymer. Scheme 1 displays the route for

preparing the highly ordered Pd-porphyrin onto modified network polymer. In addition, FTIR were recorded to determine the catalyst and confirm the covalent interactions between porphyrin and polymer and palladium(II) complex in Pd-porphyrin@polymer (Fig. 1a–d). As shown in Fig. 1a, b, different IR spectra were observed for porphyrin and Pd-porphyrin complex. The results indicated the stretching and bending frequencies of the N–H bond related to free base porphyrin placed at ~ 3310 and 960 cm^{-1} . However, the palladium ion was entered into the porphyrin ring, the N–H bond vibration frequency of porphyrin ligand was vanished, and the functional groups of Pd–N bond were established at $\sim 1000\text{ cm}^{-1}$, which resulted in forming Pd-porphyrin complex [40]. Additionally, the bands as 1722 and 1604 cm^{-1} were determined for carbonyl related to ester and C=N, respectively. In addition, based on a broad band at $3200\text{--}3600\text{ cm}^{-1}$, all of the phenolic hydroxyl groups of polymer 4 were not performed completely due to steric hindrance, compared to the IR spectrum of polymer 4 in the ESI⁺. Therefore, the quantitative FTIR data demonstrated grafting the Pd-porphyrin onto the polymer surface the successfully. Along with FT-IR spectrum, other techniques were implemented for confirming the catalyst better (UV–VIS, SEM, XRD, EDS and TGA). UV–Vis spectroscopy was used to evaluate the Pd-porphyrin@polymer. As represented in Fig. 1e, f, the absorptions peaks at 425, 525, 560, 600, and 658 nm are relayed on H_2TMCPP , while the sample gives new absorption peaks at 422, 535, and 580 nm after metalation with the palladium cation (solvent: $CHCl_3$). Morphology and size of the catalyst were observed by using scanning electron microscopy (SEM). As displayed in Fig. 2a, the Pd-porphyrin is placed on the polymer. The energy dispersive spectroscopy (EDS) was analyzed to specify the Pd-porphyrin@polymer, which confirms the presence of nitrogen and palladium in the aforesaid catalyst (Fig. 2b). The wide-angle X-ray diffraction was performed to follow the crystalline structure and size of the supported Pd. As shown in Fig. 2c, four prominent peaks at 2θ values of about 40° , 46° , 68° , and 81° related to the features of crystalline Pd. (111), (200), (220), and (311) verified a decrease in Pd ions [41]. The thermal stability of Pd(II)-porphyrin-based polymer network was examined by thermogravimetric analysis (TGA). The initial slight weight loss occurs before 150 °C, which can be attributed to the evaporation of hydrated water or residual solvent. The second and third one are occurred at 150–400 °C and they related to the decomposition of metalloporphyrin. Thus, TGA analysis demonstrates the high thermal stability of catalyst. The later loss weight is attributed to decomposition of polymer network at $\sim 450^\circ\text{C}$ (Fig. 3). Pd-porphyrin@polymer was utilized as an excellent catalyst for C–C coupling reactions after the catalyst was confirmed by implementing spectroscopic techniques (FT-IR, UV–Vis, SEM, EDS, XRD, TGA). In addition, the

Table 1 Elemental analysis data for polymer4

Entry	C (wt%)	H (wt%)
Polymer	45.74	4.97



Scheme 1 Schematic preparation of catalyst

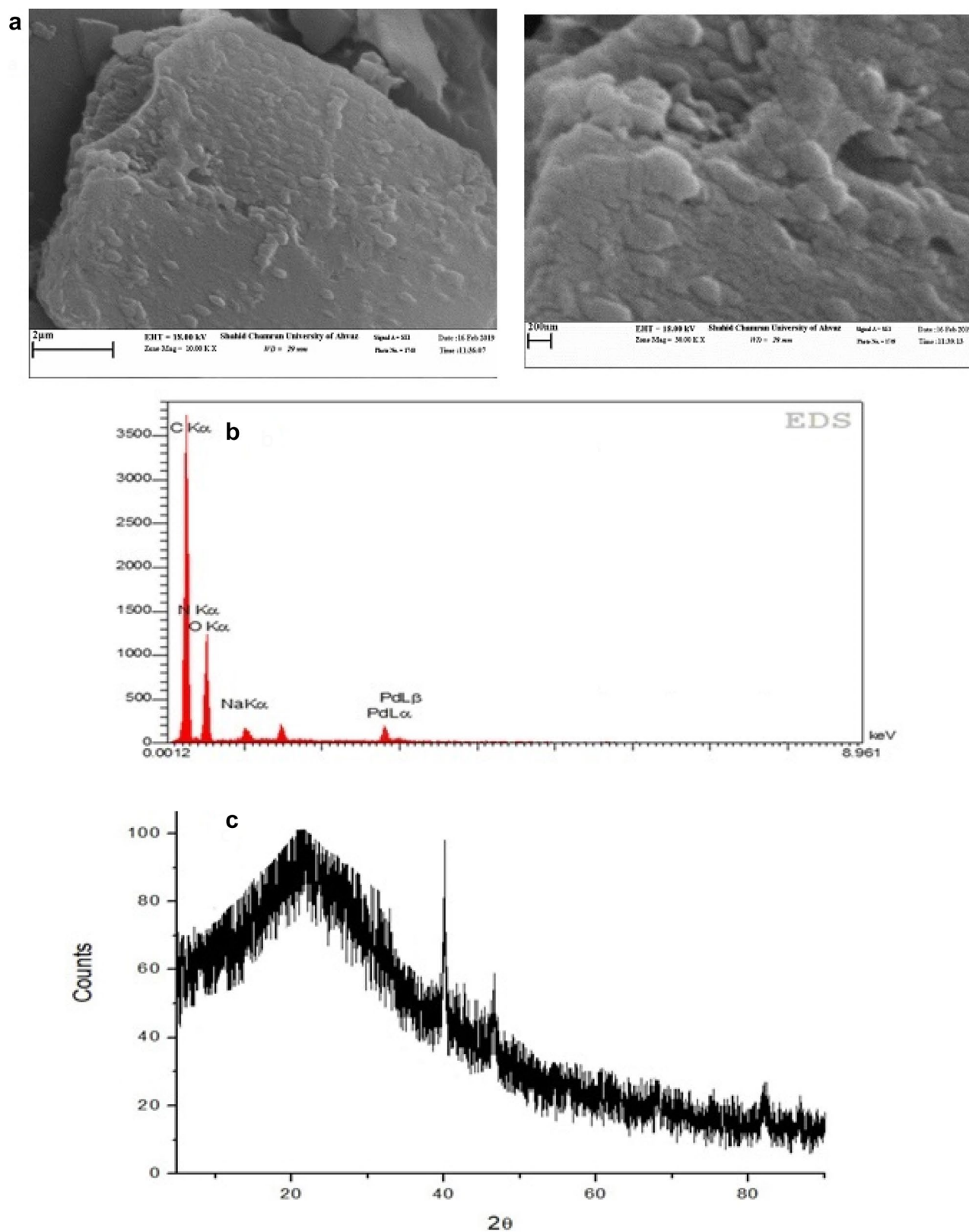
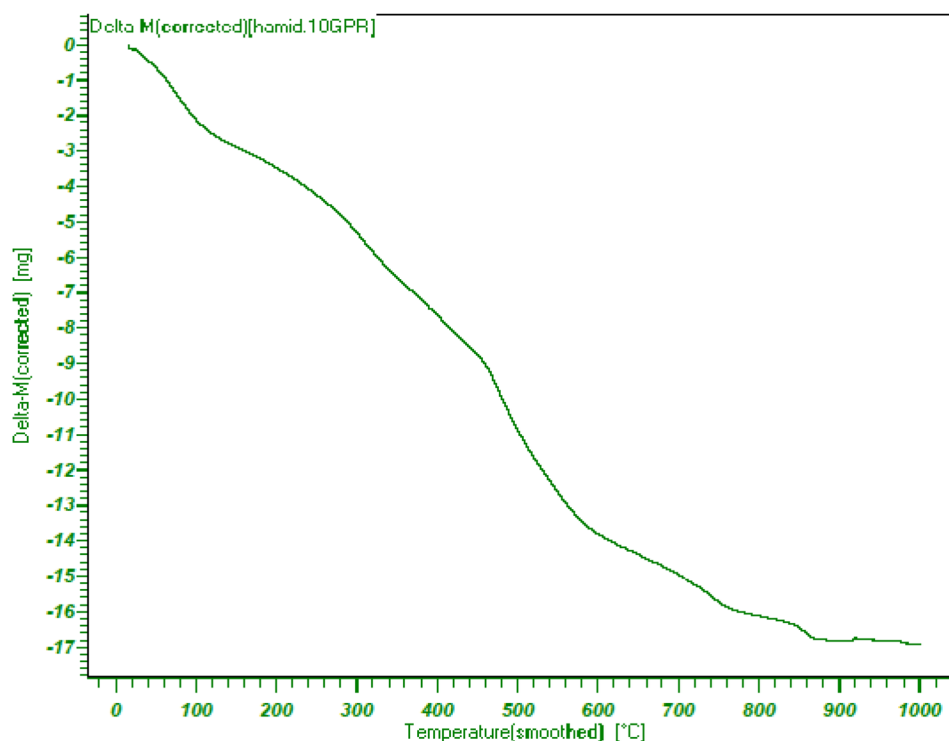
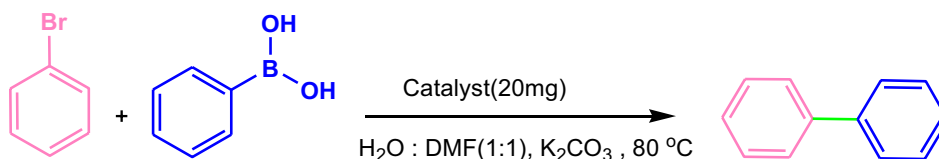


Fig. 2 SEM images of catalyst (**a**), EDS spectrum of catalyst (**b**) and XRD pattern of catalyst (**c**)

Fig. 3 TGA curves of Pd-porphyrin@polymer

reaction of bromobenzene and phenylboronic acid was selected as a pattern system to obtain optimum conditions for the purpose of evaluating the catalytic activity of Pd-porphyrin onto a 3D-network polymer based on calix[4]resorcinarene as heterogeneous catalyst for synthesizing C–C coupling. The solid was extracted with ethyl acetate, and the liquid was concentrated after running the reaction

(Table 2). The progress of the reaction was followed by TLC. It is worth noting that the reaction stopped in the absence of the catalyst. Further, the impacts of different parameters such as solvent, base, and time were evaluated to obtain the highest conversion. Additionally, some experiments were performed to discern the effect of solvent and base in this reaction. Based on the results in Table 2,

Table 2 Optimization of the reaction conditions

Entry	Base	Solvent	Catalyst (mg)	T (°C)	Yield (%)
1	K ₂ CO ₃	H ₂ O	20	80	80
2	K ₂ CO ₃	EtOH	20	80	70
3	K ₂ CO ₃	DMF	20	100	75
4	K ₂ CO ₃	H ₂ O:EtOH (2:1)	20	80	78
5	K ₂ CO ₃	H ₂ O:DMF (2:1)	20	80	85
6	K ₂ CO ₃	H ₂ O:DMF (1:1)	20	80	95
7	NaOH	H ₂ O:DMF (1:1)	20	80	75
8	Na ₂ CO ₃	H ₂ O:DMF (1:1)	20	80	77

Reaction conditions: phenylboronic acid (0.75 mmol), bromobenzene (0.5 mmol), catalyst (20 mg), base (1.5 mmol), solvent (3 ml), 80 °C

H₂O:DMF as solvent and K₂CO₃ as base had the highest conversion. Finally, the amount of Pd-porphyrin@polymer as heterogeneous catalyst was evaluated to synthesize C–C coupling. As displayed in Table 3, the best result was achieved when the reaction was performed in the presence of 20 mg of Pd-porphyrin@polymer. By considering the conditions of the optimized reaction, the substrate scope of the Suzuki coupling reaction was specified by implementing phenylboronic acid with various aromatic halides in the presence of 20 mg of Pd-porphyrin@polymer as the preferred quantity of catalyst. Based on the results in Table 4, the catalytic activity of Pd-porphyrin@polymer relied on the halide in the order ArCl < ArBr < ArI (entries 1–3, 4, 5 and 6, 7). Further, different aryl halides with electron-withdrawing and electron-donating groups resulted in producing excellent yields at normal time of the reaction (Table 4). Based on the literatures and experimental results [39], the mechanism of generation of free Pd(0) and continued its application in catalytic cycle was represented in Scheme 2. Way A or way B could be happened. After releasing of free Pd(0), the first step is oxidative addition of Pd(0) to the aromatic halides to form the organopalladium specie (I). The reaction with KOH that it generated from aqueous K₂CO₃, gives intermediate (II) which via transmetalation with the boronate complex (III) forms the organopalladium specie (IV). The reductive elimination of the biaryl product (final product) restores original free Pd(0) which completed catalytic cycle and to beginning next catalytic cycle or go back to the hole of porphyrin. Detecting a catalyst which remains consistent even under the harsh conditions is essential. The Pd-porphyrin@Polymer is considered as a thermal and chemical stable complex, complete coordinative saturation of the central atom, as well as a precedence for the Pd(II) oxidation state. Pd-porphyrin leads to the very limited release of Pd when heated under coupling reaction conditions since it involves a very rigid structure and extraordinarily high complex stability. Additionally,

Pd-porphyrin@Polymer is considered as a very beneficial pattern system for catalyzing the reactions for C–C coupling. However, the catalyst was removed by centrifugation after 5-min reaction and the reaction proceeded for 1 h without forming the product in order to evaluate the leaching of Pd-porphyrin from Pd-porphyrin@polymer in Suzuki coupling of phenylboronic acid and bromobenzene. By considering the above results, excellent heterogeneity is observed by Pd-porphyrin@polymer. The recovery potency from the reaction medium and activity is considered as one of the most main specifications of the heterogeneous catalyst. Further, the reusability of the Pd-porphyrin@polymer catalyst was examined by recovering consecutively, along with reusing the catalyst up to six times. As displayed in Fig. 4, the reaction was repeatedly conducted under one constant set of operating conditions. Thus, the recyclability of catalyst results in creating an economical and potential process for commercial applications. Also, after recycling reactions, the structural and nature of the reused catalyst was investigated by UV–Vis spectroscopy, FT-IR analysis (Fig. 5). As shown in Fig. 5, no difference was observed with the former one. Reaction conditions: phenylboronic acid (0.75 mmol), bromobenzene (0.5 mmol), catalyst (20 mg), base (1.5 mmol), solvent (3 ml), 80 °C

The catalytic efficiency of the Pd-porphyrin@Polymer as heterogeneous catalyst was compared with that of a number of previously reported catalysts in Suzuki reaction (Table 5). It is clearly displayed that the presented methodology in this paper is superior to the most of the previously reported methods in terms of catalyst amount, reaction time or product yield.

4 Conclusion

The present study introduce a novel and simple method for synthesizing Pd-macrocyclic complex supported on the surface of network polymer. First, meso-tetrakis[4-(methoxycarbonyl)phenyl]porphyrin was produced based on Adler's method. Then, meso-tetrakis[4-(methoxycarbonyl)phenyl]porphyrin immobilized on 3D- network polymer based on calix[4]resorcinarene and the product was characterized successfully by FT-IR, SEM, XRD and EDS. In addition, the catalytic activity of functionalized polymer was evaluated in the reaction related to carbon–carbon coupling. Finally, clean reaction profiles, short reaction times, easy procedure, high activity, good recyclability, and no leaching were considered as some of the advantages of using this heterogeneous catalyst.

Table 3 Optimization of the amount of the Pd-porphyrin@polymer

Entry	Catalyst (mg)	Time (min)	Yield%
1	–	80	–
2	5	60	70
3	10	15	85
4	20	15	95
5	30	15	95

Reaction conditions: phenylboronic acid (0.75 mmol), bromobenzene (0.5 mmol), base (1.5 mmol), solvent (3 ml), 80 °C

Table 4 Synthesis of cross-coupling reactions catalyzed by polymer supported Pd-porphyrin

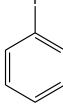
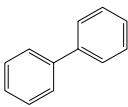
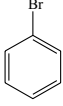
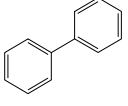
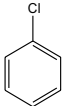
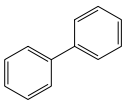
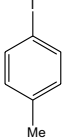
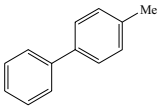
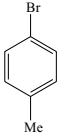
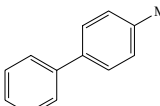
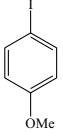
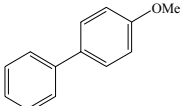
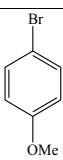
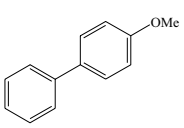
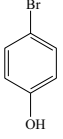
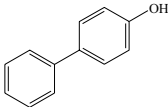
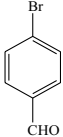
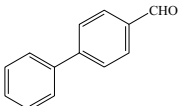
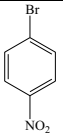
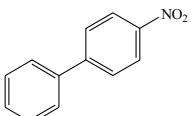
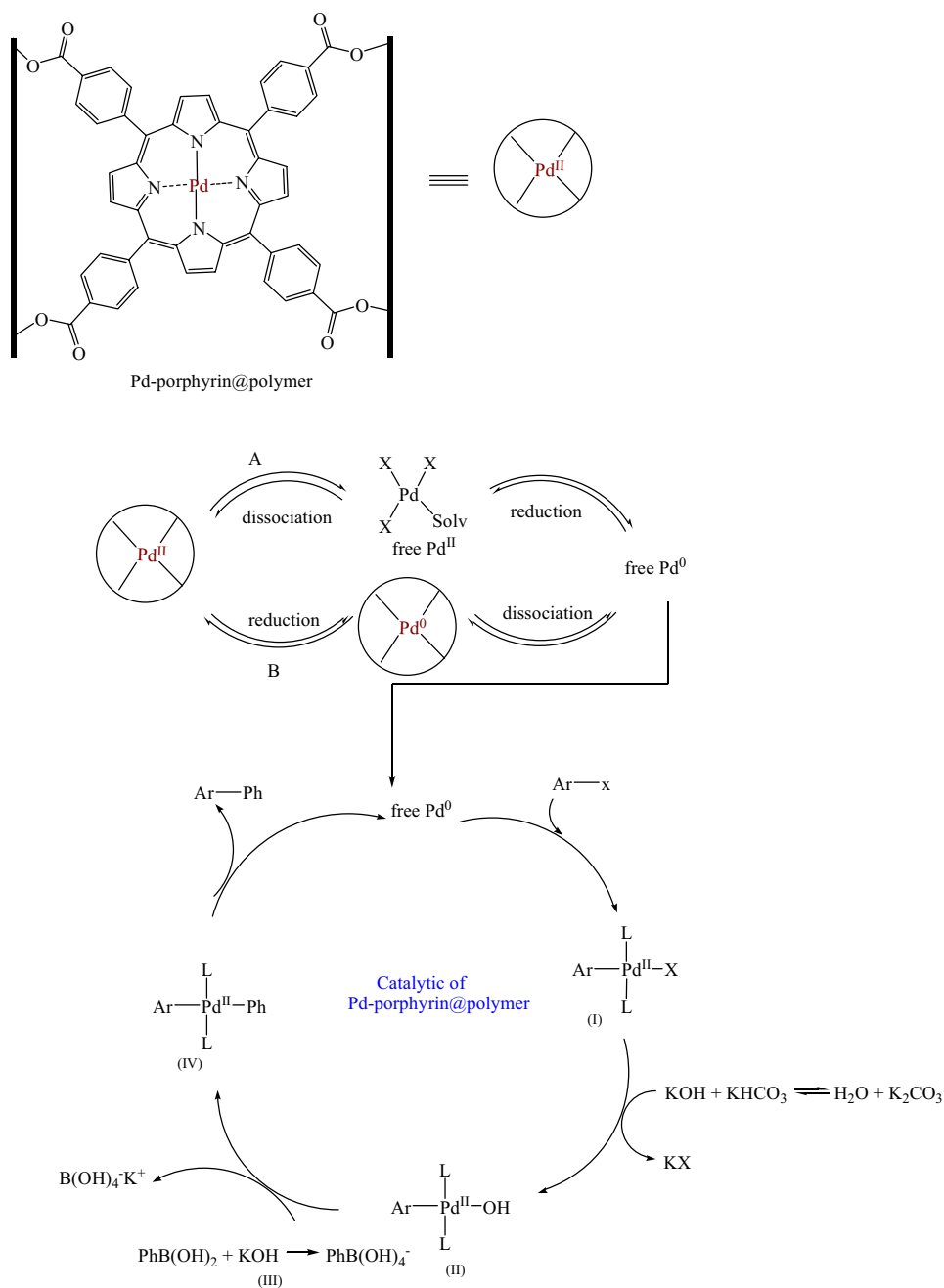
Entry	Aryl halide	Product	Time(min)	Yield%	M.P
1			10	93	66-68
2			15	95	66-68
3			140	88	66-68
4			10	95	47-49
5			15	93	47-49
6			10	89	86-88
7			15	90	86-88
8			15	88	155-158
9			105	86	55-57
10			110	90	103-106

Table 4 (continued)

Reaction conditions: phenylboronic acid (0.75 mmol), bromobenzene (0.5 mmol), catalyst (20 mg), base (1.5 mmol), solvent (3 ml), 80 °C

Scheme 2 The plausible mechanism for Suzuki reaction in the presence of Pd-porphyrin@Polymer

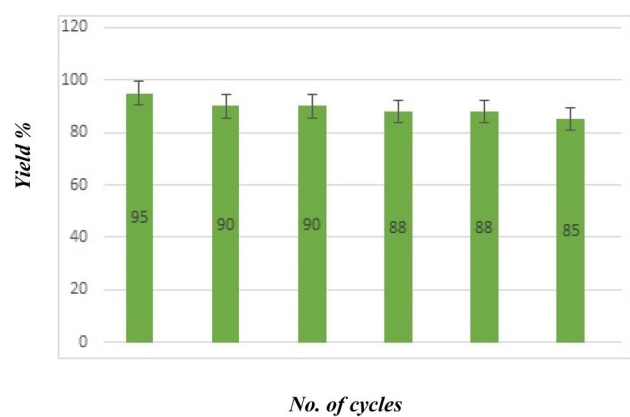


Fig. 4 Recyclability of catalyst

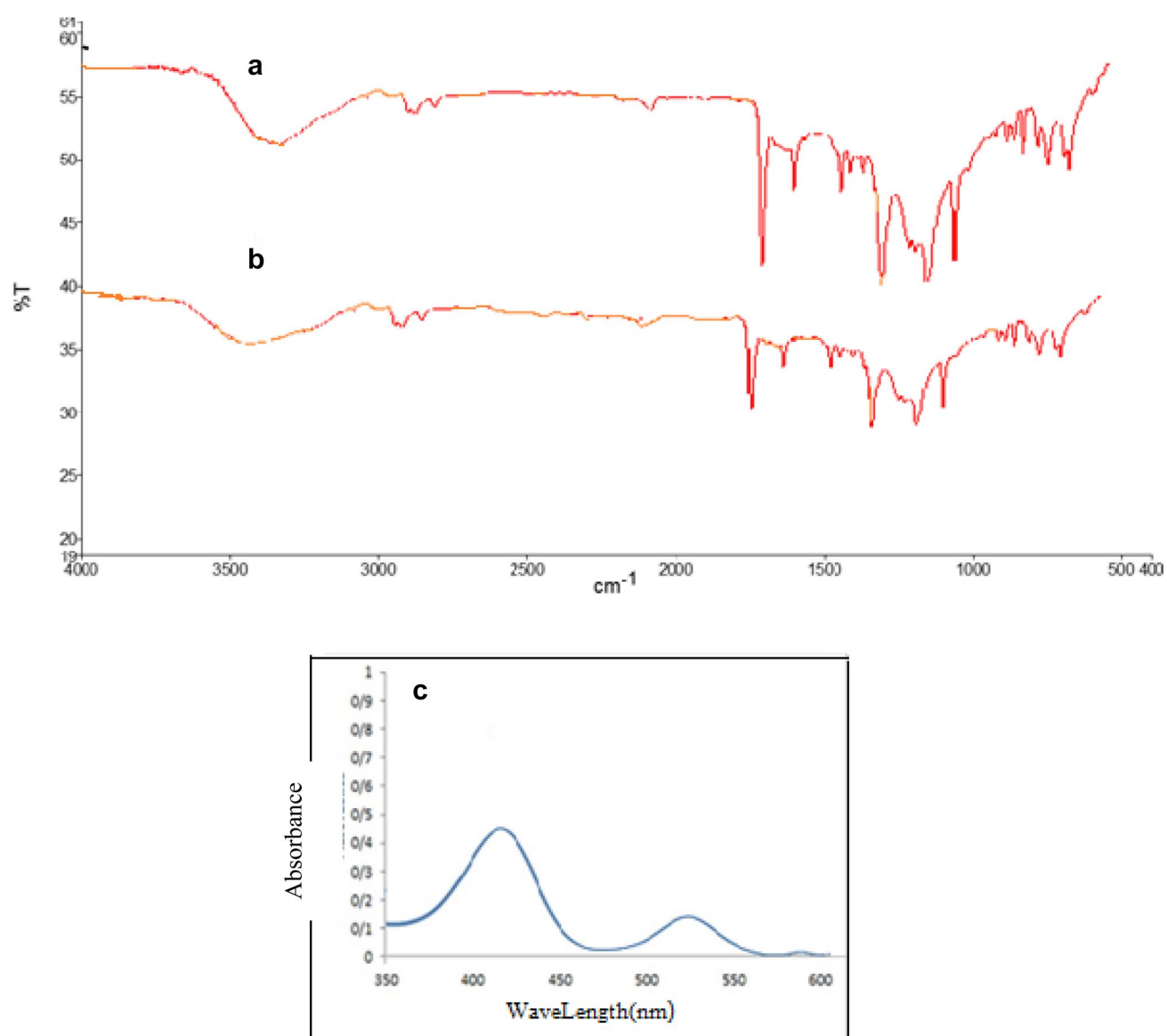


Fig. 5 FT-IR spectra of **a** fresh catalyst; **b** recycled catalyst and UV-Vis spectra of Fresh catalyst (**c**)

Table 5 Comparison of catalytic activity of Pd-porphyrin@polymer with other heterogeneous catalysts for the C–C coupling

Entry	Catalyst	Reaction conditions	Yield (%)	References
1	d/KAPs (DCM-TPP) (20 mg)	K ₃ PO ₄ ·3H ₂ O, H ₂ O, 100 °C	98	[42]
1	GO-NHC-Pd(II) (0.25 mol% Pd)	K ₂ CO ₃ , EtOH:H ₂ O, reflux, 18 h	89	[43]
3	Pd(II)-TMCPP-GO (0.7 mol% Pd)	K ₂ CO ₃ , DMF:H ₂ O, 70 °C, 40 min	92	[39]
4	Pd-Schiff base@MWCNT (0.1 mol% Pd)	K ₂ CO ₃ , DMF:H ₂ O, 60 °C, 3 h	99	[44]
5	Pd/Fe ₃ O ₄ /r-GO (0.36 mol%)	K ₂ CO ₃ , H ₂ O, 80 °C, 25 min	97	[45]
6	Pd-porphyrin@polymer (15 mg)	K ₂ CO ₃ , H ₂ O:DMF, 80 °C, 10 min	96	This work

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