FULL PAPER



Green synthesis and characterization of palladium nanoparticles supported on zeolite Y by sonochemical method, powerful and efficient catalyst for Suzuki-Miyaura coupling of aryl halides with phenylboronic acid

Azadeh Tadjarodi 🕩 | Modarres Dehghani | Mina Imani

Research Laboratory of Inorganic Materials Synthesis, Department of Chemistry, Iran University of Science and Technology, Narmak, Tehran, Iran

Correspondence

Azadeh Tadjarodi, Research Laboratory of Inorganic Materials Synthesis, Department of Chemistry, Iran University of Science and Technology, Narmak, Tehran, Iran. Email: tajarodi@iust.ac.ir In this paper, we report effective, useful and environmental compatible ultrasound method for the synthesis of zeolite–Y. Then, the prepared product was composed with palladium nanoparticles by sonication treatment. The prepared zeolite Y–Pd nanoparticles was used as catalyst in Suzuki-Miyaura coupling of aryl halides (Ar-X, X = I, Br, Cl, F) with phenylboronic acid. Based on our studies, the prepared zeolite Y-Palladium nanoparticles revealed a high catalytic performance in Suzuki-Miyaura coupling reaction so that aryl fluoride can even react with phenylboronic acid by utilizing this catalyst. The advantages of the use of this catalyst in Suzuki-Miyaura coupling reaction are green solvent, short reaction time, high yields, ligandless and recyclable. Structure and morphology of the synthesized zeolite-Y and zeolite-Y-Palladium nanoparticles were characterized by FT-IR (Fourier transform infrared), XRD (X-ray diffraction), SEM (Scanning electron microscopy), TEM (Transmission electron microscopy), EDX (Energy dispersive analysis of X-ray), BET (Brunauer Emmett Teller) and ICP-MS (Inductively coupled plasma mass spectrometry).

KEYWORDS

carbon-carbon cross coupling, palladium nanoparticles, Sonochemical method, zeolite Y

1 | INTRODUCTION

Zeolites are considered as hydrated aluminosilicates composed of crystalline structure with molecular sieving properties.^[1] These compounds are inorganic materials with thermal, chemical and mechanical stability and have been widely used as catalysts,^[2,3] ion exchangers^[4] and adsorbents.^[5] Faujasite (FAU) is one of the zeolite minerals, which is widely used. Its structural framework can be described in terms of a linkage of TO₄ tetrahedral (T = Si, Al) in a truncated octahedron in a diamond-type structure.^[6] Zeolite Y, a highly versatile member of the Faujasite family, has been widely used as industrial catalyst for several commercial processes, as long-chain hydrocarbon cracking and hydro processing technology.^[7]

Recently, the use of ultrasonic irradiation has been extensively studied,^[8-11] as a new source of energy and environmentally compatible. The role of ultrasonic waves in improvement of the formation of zeolite in different heterogeneous^[12] and homogeneous systems^[13] is as well-known as its general contribution to the crystallization of distinct mineralogical phases.^[14] Ultrasound has been successfully used with the sol–gel process in the synthesis of a wide range of nano and micro particles. The use of ultrasonic treatment in synthesis reactions offers several practical advantages compared to the conventional methods due to the low reaction temperature and the short reaction time.^[15] In fact, the ultrasound technique has been increasingly reported for the synthesis of various functional materials due to unique activations based on a physical phenomenon known as acoustic cavitation. The acoustic cavitations are generated during passing the ultrasound waves through a liquid medium and interacting with gas bubbles existing in the liquid. The recent findings have indicated an accurate estimation of temperatures and chemical events of acoustic bubbles. They behave as special microreactors with high temperature and energy, which can increase reaction rates. It was suggested that the ultrasonic technique with the formation of cavitation bubbles provides a mechanical force with a high energy in the liquid phase and also accelerates ionic reactions, molecular diffusion and mass transfer. The ultrasonic treatment enhances reactivity and assists in the breakdown of intermediates, dispersion of particles in a finely divided form. In fact, the created energy by ultrasound irradiation causes the chemical effects in the irradiated liquid; formation, growth and implosive collapse of bubbles, which increase and

improve the formation of product. This method is introduced as a powerful technique to produce various materials in higher yields, shorter reaction times and milder conditions. It can also assist in conservation of energy and minimization of wastes compared to conventional heating methods.^[16,17] Also the ultrasound technique as a reduction method has been used to prepare palladium nanoparticles from their salts.^[18] Recently, the use of ultrasound in the synthesis of micrometer sized (e.g. Na-A,^[19] Na-X,^[20] MCM-22^[11]) and nanometer-sized (e.g. Na-P,^[21] Na-A,^[22] silicalite-1,^[23] SAPO-34^[24]) molecular sieves has been reported. With this overview we also introduced this procedure to synthesize functionalized zeolite-Y composite in this work.

The palladium-catalyzed Suzuki-Miyaura cross-coupling (Scheme 1) has become one of the most versatile and powerful reactions for the construction of carbon–carbon bonds.^[25] It is widely used in the synthesis of poly-olefins and substituted biaryls,^[26] which are important skeletons in the structures of natural products, agrochemicals, pharmaceuticals, and advanced materials.^[27–29] Generally, phosphine ligands are often used in palladium-catalyzed processes but most of them are toxic, expensive and water or air sensitive. Recently, the use of available aryl chlorides



FIGURE 1 General procedure for the synthesis of Z-Y-Pd NPs catalyst

SCHEME 1 Zeolite Y-Pd nanoparticles catalyzed Suzuki–Miyaura coupling reaction of aryl halide with phenylboronic acid

and aryl fluorides in these transformations has received an increasing attention so that a number of effective catalytic systems have developed for this purpose.^[30]

We previously described an oil–water self-assembly strategy for the synthesis of Pd/reduced-graphene oxide and Pd nanoparticles at room temperature by reduction of the [PdCl₂(cod)] complex and investigated their catalytic activity in S-M coupling reaction. These reported catalysts showed a high performance in coupling reaction with shorter time and higher yield compared to other catalysts.^[31]

In this work, an efficient sonochemical procedure is reported for the synthesis of palladium nanoparticles supported on zeolite Y at rather low temperature of crystallization and short reaction time. The structural and morphological characteristics of the resulting product were studied by FT-IR (Fourier transform infrared), XRD (X-ray diffraction), SEM (Scanning electron microscopy) EDX (Energy dispersive analysis of X-ray) and ICP-MS (Inductively coupled plasma mass spectrometry). Our catalytic studies on the S-M reaction using the prepared zeolite Y-Pd nanoparticles showed this product is a suitable catalyst for carbon–carbon cross coupling (Suzuki-Miyaura coupling reaction).

2 | EXPERIMENTAL

Sodium hydroxide (NaOH, 99% wt., Merck), sodium aluminate (NaAlO₂, [Na₂O, 40-45%wt + Al₂O₃, 50-55%wt.] (Aldrich), NaSiO₂ (Aldrich, Shanghai, China), PdCl₂ (Merck) and deionized water were used as received. Ultrasound radiation was obtained by ultrasonic processor (FAPAN, 400R) with standard probe. The powder X-ray diffraction patterns (XRD) were obtained by a (STOE-STADV) instrument. Scanning electron microscopy (SEM) images was obtained using a TESCAN instrument. FT-IR spectra were taken with a shimadzo spectrometer. Brunauer Emmett Teller (BET) was obtained using a Micromeritics ASAP 2020 instrument. TEM images were obtained using a Zeiss-EM10C-100 KV instrument. ¹H NMR spectra were taken with a Bruker 400 MHz ultrashield spectrometer using CDCl₃ as the solvent and TMS as the internal standard.

2.1 | Synthesis of zeolite NaY (Z-Y)

Zeolite Y was prepared using a starting aluminosilicate gel with molar ratio $1Al_2O_3$: $4Na_2O$: $9SiO_2$:170H₂O.^[32] In a typical preparation, NaAlO₂ (1.4 g), H₂O (18.1 ml), NaOH (5.1 g), and SiO₂ (17.3 ml) at room temperature were mixed by stirring for 24–36 hr. Then the mixture was subjected to conventional heating, ultrasound

radiation. The delivered power of ultrasound wave was 150 W. During sonication, the temperature of mixture raised from ambient temperature to a steady state of 90 °C within 5 min by using a circulating oil bath. The reactor temperature was kept constant at 90 °C (\pm 1 °C). After 3 h the resultant product was filtered, washed and dried overnight at 80 °C.

2.2 | Zeolite Y-palladium nanoparticles (Z-Y-Pd NPs) catalyst preparation

The acid form of zeolite H-Y was obtained by ion exchange of NaY zeolite (1 g) with a solution of NH_4Cl (1 M, 50 ml) at 80 °C for 4 h. After this process, the exchanged zeolite was filtered, washed with deionized water, dried at 120 °C overnight and then calcined at 500 °C for 4 hr. For the next step, 1 g of H-Y zeolite was added to 50 ml of ethylene glycol in a 100 ml round-bottom flask and then $PdCl_2$ (0.04 g) and KOH (0.45 g) were added to this solution. This mixture was dispersed by ultrasonic bath for 1 hr. After ultrasonic treatment, product was filtered, washed and was kept constant at 200 °C for 4 hr. The elemental analysis of ICP-MS was used to determine the percentage of the loaded palladium on zeolite-Y substrate.

2.3 | General procedure for Suzuki-Miyaura coupling reaction

Aryl halide (0.5 mmol) and phenylboronic acid (0.75 mmol) were added to a flask containing the Z-Y-Pd NPs catalyst (15 mg) and K_2CO_3 (1 mmol) in 10 ml distilled water and ethanol (1:1). The mixture was stirred in an oil bath at 80 °C. After completion of the reaction



FIGURE 2 XRD patterns of the formation of Z-Y at different times by sonochemical method (150w and 90 °C)

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Entry

1

2

3

4

5

6

7

TABLE 1 Synthesized zeolites with hydrothermal method

180

24

24

24

Na-Y

Na-Y

Na-Y

Na-Y

110

60

100

100

(monitored by TLC), the reaction mixture was cooled down to room temperature, and then dichloromethane was added to the reaction vessel. The organic phase was separated and dried over anhydrous MgSO₄. The pure desired product was obtained by the evaporation of the solvent.

3 | RESULTS AND DISCUSSION

In this work, the synthesis of Z-Y-Pd NPs catalyst is reported and it is suitable for Suzuki-Miyaura cross coupling reaction (Figure 1). At the first, we studied the effect of time in synthesis of Z-Y by sonochemical method. Results showed that structure and morphology of Z-Y as shown in Figure 2 is completely formed at 3 hr by ultrasonic irradiation. XRD patterns confirmed the formation of Z-Y at different times with ultrasonic irradiations (150 W) at 90 °C. After crystallization, product shows sharp diffraction peaks at 2 theta of 6.05°, 10.02°, 11.7°, 15.4°, 20.1°, 23.3°, 26.7° and 30.9°. This pattern confirms the formation of Z-Y.

A comparison of the ultrasonic method and hydrothermal method was given in Table 1. It shows the synthesis of zeolites by hydrothermal method needs to consume the high energy and more time (Table 1), whereas the synthesis of zeolites by ultrasonic method is performed in short time, low temperature or pressure. Furthermore, by gel composition and the use of ultrasonic, more convenient reaction conditions can be used to form discrete and well-defined zeolite nanoparticles. This hence results in a significant improvement in nanocrystal properties (electrical, optical, magnetic, catalytic, etc.), which have an important impact on advanced applications and in fundamental studies.^[33–39] In this study, Pd nanoparticles have been prepared by the ultrasonic reduction method. After ultrasonic treatment, the reduction of Pd (II) ions could occur by generated organic radicals as the following reactions (1–3):

$$H_2 O \rightarrow OH + H$$
 (1)

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$$HOCH2CH2OH + OH(H) \rightarrow HOCH_2C HOH + H_2O(H_2)$$
(2)

$$nPd (II) + 2nHOCH_2C^{\bullet} HOH \rightarrow nPd(0) + 2nHOCH_2CHO + 2nH^+$$
(3)

The proposed mechanism correspond with report Okitsu et al.^[40] In fact, the created energy by ultrasound irradiation causes the chemical effects in the irradiated liquid; formation, growth and implosive collapse of bubbles,



FIGURE 3 (a) FT-IR spectrum and (b) SEM image of synthesized Z-Y



FIGURE 4 (a) XRD pattern and (b) SEM image of Z-Y-Pd NPs

which increase and improve the formation of nanoparticles.

The FT-IR spectrum and SEM image of the synthesized Z-Y were indicated in Figure 3. There are the strong vibrations at 1002, 719, 567 and 460 cm⁻¹ in the FT-IR spectrum of product (Figure 3a). The characteristics bands at 460, 567 and 1002 cm⁻¹ are assigned to T-O (T = Si, Al) bending and Si-O, Al-O tetrahedral vibration, respectively.^[41,42] The SEM image (shown in Figure 3b) revealed a uniform particulate morphology with an average particle size of 84 nm.

XRD pattern and SEM image of the prepared Z-Y-Pd NPs were shown in Figure 4. There are diffraction peaks at 2 theta of 40.04°, 46.63° and 67.99°, which are related to the palladium nanoparticles decorated on zeolite.^[43] The peaks at 2 theta of 6.04°, 13.64°, 17.58° and 28.44° are attributed to Z-Y substrate confirming its structure without any changes.^[41]

The chemical composition of the Z-Y-Pd NPs was determined by elemental analysis of EDX and ICP-MS, confirming the presence of Pd NPs (2.7%) on the zeolite body (Figure 5).

Figure 6 (a-b) shows TEM images of the Z-Y-Pd NPs, which includes a spherical morphology of product with



FIGURE 5 EDAX spectrum of the Z-Y-Pd NPs

the decorated dark spots, which can be probably related to Pd particles. The average diameter of the Z-Y-Pd NPs is about 84 nm with the decorated Pd nanoparticles with the average size of about 5 nm. This indicates desirability of the ultrasonic method for adhering Pd NPs on the surface of zeolite. Also, the average crystallite size of the as prepared Pd nanoparticles were calculated by the Scherer formula: $D = 0.9\lambda/(\beta \cos \theta)$, where D represents the crystallite size (nm), β is the full width of the diffraction line at half the maximum intensity, λ represents the wavelength of X-ray and θ is the Bragg angle.^[44] The average crystallite size of about 7 nm obtained for the prepared product using XRD pattern and Debye-Scherrer equation. This value is much lower than the obtained particle size of 84 nm resulted from TEM images for the Z-Y-Pd particles. It returns to the difference of concept of the crystallite size and particle size. A particle may be made up of several different crystallites or just one crystallite that in this case is equal to particle size. We often report the particle size based on SEM and TEM images because we can't exactly determine whether the particle is one crystallite or composed of many compacted single crystals. The observed difference between the obtained values of XRD and TEM images can reveal that each particle of the prepared product is a composed of small crystallites with the average crystallite size of about 7 nm.

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Nitrogen adsorption-desorption measurements were carried out at -196 °C on a Micromeritics ASAP 2020 instrument to determine the Brunauer Emmett Teller (BET) surface area and to estimate the mesopore size distribution using the Barrett Joyner Halenda (BJH) calculation procedure. Before each measurement, samples were evacuated 3 hr at 300 °C. The N₂ adsorption-desorption isotherm curves for the synthesized Z-Y-Pd NPs (Figure 7) revealed a hysteresis loop in the range of $0 < P/P_0 < 1$, which is in a close accordance with the category of type II, which represents a porous nature of the



FIGURE 6 (a, b) TEM images and (c) histogram of particle size distribution of Z-Y-Pd NPs

prepared product. BET analysis using nitrogen adsorption–desorption experiments indicated the surface area of 80.66 m² g⁻¹. The pore volume of 0.001 cm³ g⁻¹ was determined by the t-plot method. In addition, the micropore diameter was obtained at about 14.79 nm using BJH curve. Based on the BET results, the synthesized Z-Y-Pd NPs possess a good porous surface that is necessary to make it an effective catalyst.

The Suzuki-Miyaura coupling reactions of aryl halides with phenylboronic acid were investigated by employing the Z-Y-Pd NPs as catalyst. The weight percent of Pd in



FIGURE 7 Adsorption-desorption isotherm from Z-Y-Pd NPs

the Z-Y-Pd NPs catalyst, as determined by ICP-MS, was 2.72 wt%.

A series of reactions were performed with various amounts of catalyst to test the reaction feasibility with catalyst concentration (Table 2). The optimal amount of the Z-Y-Pd NPs catalyst was determined by the reaction of bromobenzene with phenylboronic acid at 80 °C. Based on the Table 2, it is evident that an increase in the amount of Z-Y-Pd NPs catalyst leads to an increase in yield of the cross-coupled product. The optimum amount of the Z-Y-Pd NPs catalyst is 15 mg.

To study the effects of different solvents in our catalytic system, the coupling of bromobenzene with phenylboronic acid was performed in the presence of various solvents. The results of the reactions have been presented in Table 3. According to our study, when polar solvents are used, better catalytic performance can be achieved in product (Entry 1, 2). Also, aprotic solvents (Entries 5–7) have relatively less yields. The lowest conversion (63%) was obtained with THF (Entry 4) and the highest conversion was obtained with H₂O: EtOH (1:1) (Entry 3, 98%). K₂CO₃ as a base and reflux at 80 °C were used for optimizing the solvent of the reaction.

Table 4 shows the catalytic activity of Z-Y-Pd NPs for various aryl halides (aryl iodide, aryl bromide, aryl chloride and even aryl fluoride) in cross coupling reaction. In these reactions, aryl iodide and bromide with various WILEY Organometallic 7 of 10 Chemistry

TABLE 2 Investigating the effect of catalyst amount in the synthesis of biphenyl from bromobenzene and phenylboronic acid at 80 $^{\circ}$ C inH2O: EtOH

Br	+ B(OH) ₂ H ₂ O:EtOH, 10 ml (1:1) 1 h Z-Y-Pd NPs as catalyst	
Entry	Amount of catalyst (mol %)	Yields ^a (%)
1	0.007	73
2	0.01	90
3	0.02	98
4	0.04	98

^aIsolated yields.

TABLE 3 Suzuki-Miyaura coupling reaction in various solvents in the presence of 0.02 mol% of Pd NPs

Br +	B(OH) ₂ Solvent 1 h Z-Y-Pd NPs as catalyst	
Entry	Solvent	Yields ^a (%)
1	EtOH	94
2	H ₂ O	88
3	EtOH:H ₂ O (1:1)	98
4	DMF	78
5	DMSO	72
6	Toluene	65
7	THF	63

^aIsolated yields.

TABLE 4 Suzuki reaction^a of different aryl halides, with phenylboronic acid

	$R \xrightarrow{Z} X + X + B \xrightarrow{OH} B \xrightarrow{Z-Y-Pd h} B \xrightarrow{Arychystar} B Ary$	$\begin{array}{c} \text{NPs} \\ \text{yst} \\ \text{CO}_{3} \end{array} \qquad $	
Entry	Substrate	Product	Time (h): Yield ^b (%)
1	C ₆ H ₅ I	a	0.25: 99
2	C ₆ H ₅ Br	a	1.0: 98
3	C ₆ H ₅ Cl	a	1.5: 88
4	4-MeC ₆ H ₄ I	b	1.0: 98
5	$4-NO_2C_6H_4Br$	С	2.5: 99
6	$4-NO_2C_6H_4Cl$	С	2.0: 91
7	4-CNC ₆ H ₄ Cl	d	2.0: 92
8	4-CNC ₆ H ₄ F	d	3.0: 63
9	C ₅ H ₄ NCl	e	2.5: 89

^aAryl halide (0.5 mmol), phenylboronic acid (0.75 mmol), Z-Y-Pd NPs (0.02 mol%), K_2CO_3 (1.0 mmol), and water/ethanol (1:1, ν/ν). ^bIsolated yields. 8 of 10 WILEY-Organometallic

TABLE 5 Catalytic performance of different Pd-based catalysts in the coupling of chlorobenzene and phenyl boronic acid

Entry	Catalyst	Solvent	Base	Temp (°C)	Time (h)	Yield (%)	References
1	CelFemImiNHC@Pd complex	EtOH	Cs_2CO_3	RT	2.0	54	[46]
2	IPrPdCl ₂	H ₂ O/ <i>i</i> -PrOH	K_3PO_4	RT	5.5	99	[47]
3	Pd/3-ampy-GO	H ₂ O/EtOH	K_2CO_3	80	0.5	85	[48]
4	PtPdCu	H_2O	K_2CO_3	Reflux	1.5	80	[45]
5	PdPtZn	H ₂ O-CTAB	K_2CO_3	Reflux	2.0	80	[49]
6	Pd/r-GO	H_2O	K_2CO_3	Reflux	3.0	80	[31]
7	Pd/Fe ₃ O ₄ /r-GO	H_2O	K_2CO_3	Reflux	2.5	85	[30]
8	Fe ₃ O ₄ /P (GMA-AA-MMA)–Pd	H ₂ O/EtOH	K_2CO_3	80	3.0	9	[50]
9	PdCl ₂	PEG	K_2CO_3	RT	6.0	98	[51]
10	Pd-1/FSG	H_2O	K_2CO_3	100	12.0	26	[52]
11	Pd/C (1% mol)	TBAB,H ₂ O	Na ₂ CO ₃	Microwave heating, 120 °C	0.5	48-94	[53,54]
12	Pd/C (1% mol)	TBAB,H ₂ O	Na ₂ CO ₃	Microwave heating, 120–150 °C	0.2	80-90	[54,55]
13	Z-Y-Pd NPs	H ₂ O/EtOH	K ₂ CO ₃	Reflux	1.5	88	This work

substituents give excellent yields with conversions ranging from 98 to 99% (Entries 1, 2, 4 and 5). Also, when the aryl chloride substrates are used, good efficiency is achieved with conversions ranging from 88 to 92% (Entries 3, 6, 7 and 9). An interesting case with this catalyst is the reaction between an inactive fluorine substrate with phenylboronic acid (Entry 8) with conversion 63%. To the best our knowledge there are a few catalyst that able to react with aryl fluorides. A frequent observation in Suzuki-Miyaura coupling reaction states that a better conversion is achieved with arvl halides containing an electron-withdrawing group.^[31] For example, when aryl halide containing a cyanide group, electron-withdrawing group, yield of reaction is better than when it involves methyl group, electron-donating group. The oxidative addition of palladium into a carbon-halogen (C-X) bond occurs in the order I > Br > Cl > F, based mainly on the strength of the C-X bond.^[45] In addition, we carried out S-M reaction without using the prepared catalyst in



FIGURE 8 Reusability of Z-Y-Pd NPs catalyst in the Suzuki-Miyaura cross-coupling reaction between bromobenzene and phenylboronic acid

the same conditions specially by utilizing CNC_6H_4F substrate and monitored the progress of process by TLC. The results did not show any progress in coupling reaction after several hours, which can reveal the role of catalyst in the progress of mentioned reactions.

We compared our results with those of noble metalbased catalysts reported in the past few years for Suzuki-Miyaura reactions, taking the reaction of chlorobenzene with phenylboronic acid as an example (Table 5). As shown, catalytic performance of Z-Y-Pd NPs synthesized by ultrasonic treatment is excellent during short time and more yield in compare with different methods and catalysts.

The reusability of the Z-Y-Pd NPs catalyst was tested for the reaction of bromobenzene and phenylboronic acid. The catalyst was separated from the reaction mixture by centrifugation, washed with ethyl acetate and dried at room temperature and then reused in another reaction with same materials of previous reaction. It was found that the Z-Y-Pd NPs catalyst could be recycled for ten times without losing the catalytic activity (Figure 8).

4 | CONCLUSIONS

In summary, a simple and green sonochemical method was applied to synthesize zeolite Y and then, palladium nanoparticles were deposited on the prepared zeolite Y with ultrasonic treatment. The catalytic performance of Z-Y-Pd NPs, we studied for the Suzuki-Miyaura coupling reaction. The Z-Y-Pd NPs showed a high activity in the carbon–carbon coupling reaction that even aryl fluoride revealed a rather good yield for reaction with phenylboronic acid in presence of this catalyst. This work introduces the advantages of green solvent, short reaction time, high yields, ligand-free and reusability of the catalyst.

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ANALYTICAL DATA FOR COMPOUNDS

Biphenyl (a). [28] White solid, mp: 68-70 °C (lit. mp 69-71 °C); FT-IR (KBr/cm⁻¹): 3030,1569, 1477, 1424, 1167,1008, 731, 695; ¹H NMR (400 MHz, CDCl₃): $\delta = 7.46 \text{ (m, 2H)}, 7.55 \text{ (m, 4H)}, 7.71 \text{ (m, 4H)}.$

4-Methylbiphenyl (b). [28] White solid, mp: 44-46 °C (lit. mp44–46 °C); FT-IR (KBr/cm⁻¹): 3077, 3024, 2924, 2851, 1603, 1438, 1348, 1123, 1019, 700; ¹H NMR (400 MHz, CDCl₃): $\delta = 2.45$ (s, 3H, CH₃), $\delta = 7.33$ (m, 2H), 7.37 (m, 1H), 7.59 (m, 2H), 7.76 (m, 2H), 7.88 (2H).

4-Nitrobiphenyl (c). [28] Yellow solid, mp: 112-115 °C (lit. mp113–115 °C); FT-IR (KBr/cm⁻¹): 3100, 3021, 1610, 1578, 1441, 1310, 1089, 1024, 703; ¹H NMR (400 MHz, $CDCl_3$): $\delta = 7.47$ (m, 3H), 7.67 (m 2H), 7.77 (m, 2H), 8.46 (m, 2H).

4-Phenylbenzonitrile (d). [28] White solid, mp: 84-87 °C (lit. mp84-88 °C); FT-IR (KBr/cm⁻¹): 3046, 3024, 2229, 1650, 1600, 1441, 1346, 1083, 1024, 703; ¹H NMR (400 MHz, CDCl₃): $\delta = 7.53$ (m, 3H), 7.63 (m, 2H), 7.83 (m, 2H), 8.23 (m, 2H).

2-Phenylpyridine (e). [28] Colorless oil, FT-IR (KBr/ cm⁻¹): 3089, 3058, 3033, 1743, 1575, 1563, 1466, 1444, 1265, 1022, 801, 743, 690; ¹H NMR (400 MHz, CDCl₃): $\delta = 6.89$ (m, 1H), 7.28 (m, 1H), 7.32 (m, 2H), 7.37 (m, 1H), 7.45 (m, 1H), 7.96 (m, 2H), 8.56 (m, 1H).

ORCID

Azadeh Tadjarodi D http://orcid.org/0000-0003-0496-2322

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.

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