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ORIGINAL PAPER

Binuclear Pd(II) complexes with multidentate Schiff base ligands: synthesis, catalysis, and antibacterial properties

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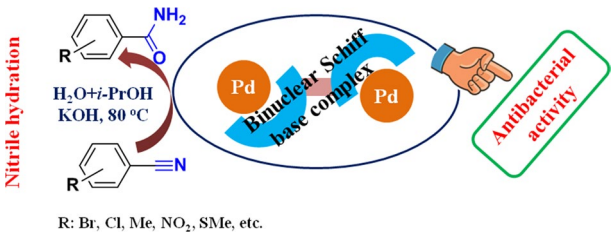
Abstract

Three new binuclear palladium(II) complexes with multidentate Schiff base ligands were synthesized and characterized by FT-IR, UV–Vis, ^{1}H-NMR, ESI–MS, and CHN analysis. The complexes were successfully applied as catalysts for hydration of nitriles to amides. Using one of the complexes, a wide range of aryl/heteroaryl nitriles were efficiently converted to corresponding amides in moderate-to-excellent yields with low catalyst loading (0.8 mol%). In addition, the complexes were also tested for antibacterial activity against two gram-positive and two gram-negative bacteria using Kirby Bauer's disc diffusion method. However, to our surprise, among the three complexes, only one complex showed growth inhibitory effect against gram-positive bacteria, *Bacillus subtilis*, for which minimum inhibitory concentration (MIC) was determined to be 30 μg cm^{-3}.

Graphical abstract



Keywords Binuclear palladium complexes · Schiff bases · Nitrile hydration · Antibacterial activity

Introduction

Schiff bases are one of the most extensively studied classes of ligands that have got widespread applications in transition metal chemistry [1, 2]. Schiff bases have gained the status of 'privileged ligands' owing to their extraordinary stability, synthetic flexibility with tunable stereo-electronic properties, and ability to stabilize metals in various oxidation states [3–6]. During the past few years, Schiff base-palladium complexes have attracted enormous attentions across diverse fields starting from catalysis to biological applications [7–11]. However, majority of the reported Schiff base-palladium systems are mononuclear in nature, while from catalytic or biological perspective, binuclear complexes are more intriguing mainly because of their synergistic potentiality [12]. In fact, one of the key inspirations of designing bi- or polynuclear Schiff base complexes came from metalloenzymes; as the active sites of many such enzymes consist of multiple metal centres acting in concert to drive a specific biochemical process [13, 14]. Hence, efforts are being made to synthesize palladium based homo- or hetero-nuclear Schiff base complexes [15–17]. Some of the complexes of palladium were found to display enhanced catalytic activity

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for a number of catalytic reactions [18]. For example, Nandhini et al. reported homobinuclear Pd(II) complexes containing bis-O,N-bidentate Schiff base ligands that showed excellent activity as catalysts for the Suzuki–Miyaura cross-coupling reaction [19]. Similarly, Handa's group demonstrated utility of a hetero-binuclear Pd/La complex containing a Schiff base ligand that showed excellent catalytic performance for *anti*-selective asymmetric nitroaldol reactions [20].

However, to the best of our knowledge, the potentiality of binuclear palladium-Schiff base complexes has not been explored for an important reaction like hydration of nitriles to amides. It may be noted that hydration of nitriles to corresponding amides is considered as one of the most convenient protocols for the synthesis of targeted amides [21–23] that serve as starting materials in various organic reactions [24, 25]. Mechanistic study revealed that the reaction involves activation of both nitrile substrate and water [26, 27]. Thus, a binuclear complex is expected to accelerate the reaction much faster than their mononuclear counterpart through simultaneous activation of the two reactants at the two metallic centres.

Besides catalytic potentials, palladium Schiff base complexes are also known for their excellent antibacterial behaviour against various gram-positive and gram-negative bacteria species [28, 29]. However, it is surprising to note that compared to mononuclear analogues antibacterial reports with binuclear palladium-Schiff base complexes is very limited [30].

As a part of our ongoing research on Schiff base chemistry [31–34], herein, we have reported two Schiff base ligands and their three binuclear palladium complexes. Catalytic activity of the complexes in nitrile hydration reaction and their antibacterial activity against four bacterial species are presented in this article.

Results and discussion

Synthesis and characterization of the ligands and complexes

The ligands **L1** and **L2** were synthesized using previously reported protocols [35, 36]. The binuclear palladium complex **1** (Fig. 1) was prepared by reacting the ligand **L1** with $Pd(OAc)_2$ in MeOH in 1:2 molar ratio under refluxing condition. Elemental analysis (C, H, N) and the mass spectra of the complex are in good agreement with the proposed molecular composition. The ESI–MS spectra of the complex showed the molecular ion peak $[M]^+$ at $m/z = 840$. In the FT-IR spectrum, the characteristic band for $\nu_{C=N}$ stretching was observed at 1568 cm^{-1} and compared to free ligand (1560 cm^{-1}), the band was shifted to slightly higher frequency, consistent with complexation of the **L1** with palladium. In the ^1H NMR spectrum, the singlet corresponding to the imine protons was observed at $\delta = 10.17$ ppm. Compared to the free ligand (observed at 10.09 ppm) a downfield shift was observed suggesting coordination of the imine ligand with palladium. As expected, the –OH protons of the ligand (observed at 5.51 ppm) disappeared upon complexation attributing an N_2O_2 type coordination environment around the metal centre. The ^{13}C NMR spectrum of the complex also showed peaks for the imine carbons at $\delta = 163.60$ ppm. In the UV–Vis spectrum, a strong absorption peak was observed at $\lambda_{max} = 373$ nm that could be attributed to $n \rightarrow \pi^*$ transition. The peak disappeared in the spectrum of the complex **1** indicating coordination of the ligand **L1** with palladium.

The complexes **2** and **3** (Fig. 1) were synthesized by reacting the ligand **L2** with $Pd(OAc)_2$ and $PdCl_2$, respectively. In the ESI–MS of the complexes **2** and **3**, the peaks corresponding to $[M]^+$ and $[M+1]^+$ species were observed at $m/z = 751$ and 658, respectively. In the FT-IR spectra of

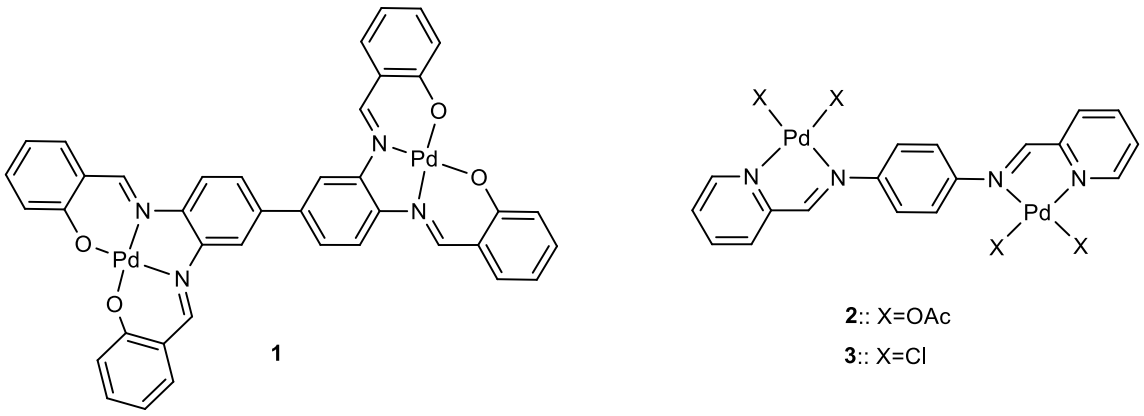


Fig. 1 Binuclear palladium-Schiff base complexes **1**–**3**

the respective complexes, the stretching bands corresponding to the imine bonds appeared at 1560 and 1563 cm^{-1}. Both these values were found to appear nearly at the same position as that of the ligand **L2** unlike the complex **3**. In addition, the band for stretching vibrations corresponding to $\nu_{C=O}$ of acetate groups present in the complex **2** appeared at 1643 cm^{-1} in its FT-IR spectrum. The ^1H NMR spectra of the complexes **2** and **3** showed imine protons as distinct singlets at $\delta = 9.56$ and 9.01 ppm, respectively. The downfield shift of peaks from the corresponding signal observed in the spectrum of **L2** (8.92 ppm) confirmed formation of the complexes. In addition, a singlet of the methyl protons of the acetate groups present in the complex **2** was also observed at 2.40 ppm. In the UV–Vis spectra of the complexes, the sharp electronic transition with $\lambda_{max} = 387$ nm corresponding to $n \rightarrow \pi^*$ transition observed in **L2** disappeared giving an evidence of formation of the complexes. It may be important to note that all the complexes are stable in air and could be kept in desiccator for several months.

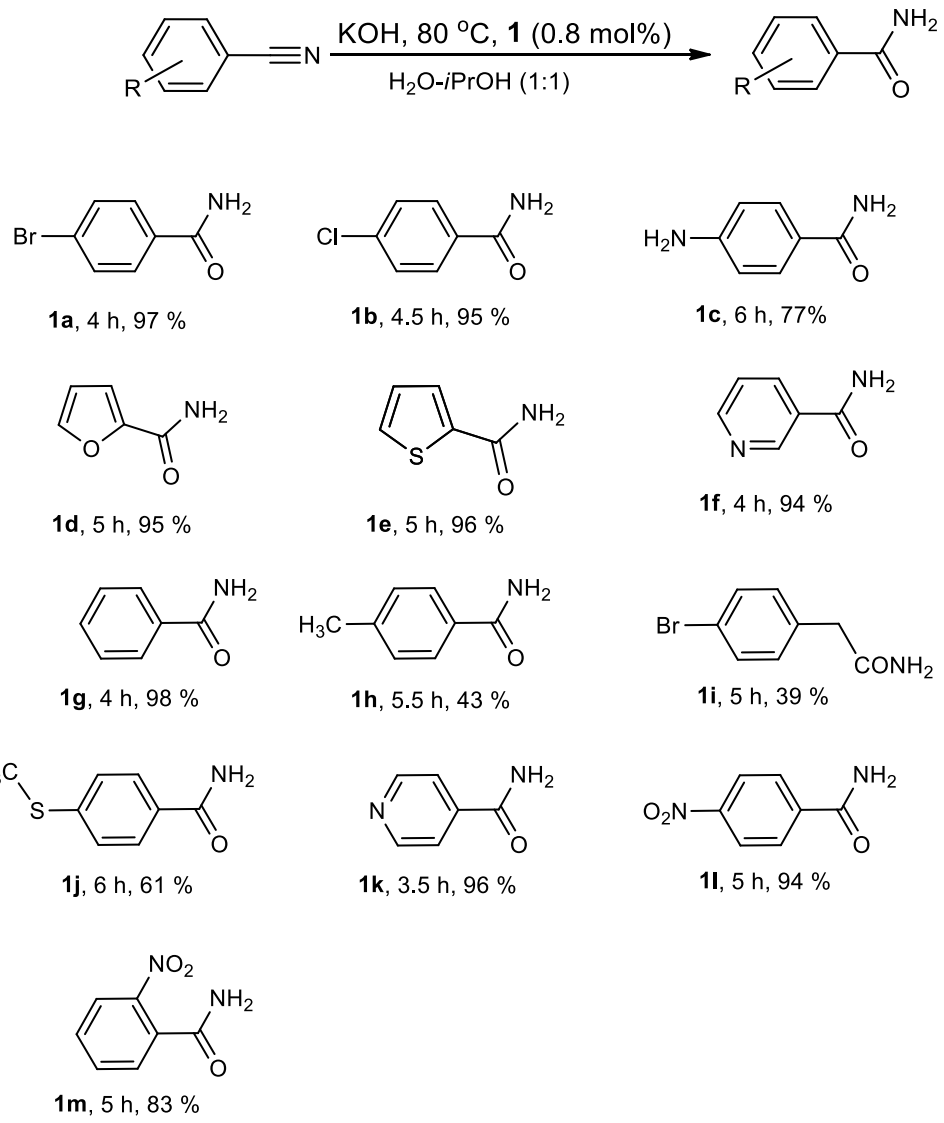
Catalytic activity of 1 in nitrile hydration

Direct addition of water to nitriles in presence of catalyst is one of the most attractive and convenient routes for preparation of corresponding amides which are used as intermediates in fine chemical industry [23]. At the outset of this study, we intended to evaluate catalytic activity of the synthesized complexes for the reaction under different experimental conditions. For initial optimization, benzonitrile was chosen as model substrate and hydration was carried out at 80 °C in presence of KOH as base in three different solvent systems: H_2O, *i*-PrOH-H_2O (1:1), and *i*-PrOH. In fact, the catalyst **1** was found to be the best candidate to produce benzamide in all the three solvent systems (Table S1). Therefore, the complex **1** was appointed as catalyst for further optimization of various reaction parameters. Using the catalyst **1**, several pure and mixed solvent systems were tested where *i*-PrOH-H_2O in 1:1 was found to be best media for the model reaction (Fig. S1). Following this, a bunch of inorganic bases were also screened where KOH could execute maximum conversion of benzonitrile to benzamide (Fig. S1). Further, it was observed that temperature also played a crucial role in the catalytic activity of **1** as the formation of benzamide went on increasing with gradual increase of temperature up to 80 °C (Fig. S2). At 80 °C, yield of the product was recorded to be 71% with 0.5 mol% of the catalyst. But, further increase of temperature to 100 °C with the same quantity of catalyst did not render significant increase of product. However, in presence of 0.8 mol% of the catalyst, yield of the product appreciably increased to 98% even at 80 °C (Fig. S2). Therefore, these conditions (*i*-PrOH-H_2O (1:1 v/v), KOH, 80 °C, and 0.8 mol% of **1**) were chosen as the optimum reaction conditions for further study of substrate scope. It may be noted that there are examples in the literature that nitrile hydration could take place under basic condition without the use of any transition metal catalyst [37]. To confirm this, we have performed a blank experiment with benzonitrile under the optimized condition without using the catalyst and only 34% product formation was observed. However, under similar experimental condition, the product yield significantly improved when the complex **1** was used as catalyst.

Scope of the catalyst was then extended for various sterically and electronically diverse nitriles under the optimized reaction conditions (*i*-PrOH-H_2O (1:1), KOH, 80 °C, 0.8 mol % of **1**). In the study, it was observed that nitriles containing electron withdrawing substituents could effectively be converted to amides using **1** as catalyst with excellent yields. For instance, 4-Br, 4-Cl and 4-NO_2 benzonitriles gave corresponding amides with 97, 95, and 94% yields in 4, 4.5, and 5 h of reaction time, respectively (Table 1, **1a**, **1b**, and **11**). However, yield of benzonitrile with electron withdrawing group like -NO_2 at *ortho* position produced lower yield of product in same duration of reaction time (Table 1, 1**m**). On the contrary, substrates with electron donating substituents could be converted to corresponding amides with moderate yields. For instance, benzonitrile with 4-NH_2 and 4-CH_3 substituents gave 77 and 43% of yields, respectively (Table 1, **1c** and **1h**), although, the reactions were carried out for quite longer time. Electrically neutral benzonitrile could also be activated by the catalyst towards hydration producing 98% benzamide in just 4 h of reaction time (Table 1, **1g**). Notably, the catalyst **1** could also effectively tolerate some heteroaromatic nitriles that are usually reluctant to catalytic hydration. Consequently, nicotinonitrile isomers were converted to corresponding nitriles with significant conversion of 94 and 96% with exclusive selectivity (Table 1, **1f** and **1k**). Moreover, nitriles with heterocyclic five membered aromatic ring, viz. furan-2-carbonitrile and thiophene-2-carbonitrile were also compatible for hydration with the present catalytic system which could produce 95 and 96% yields of corresponding amides, respectively (Table 1, **1d** and **1e**). But, benzylic nitrile with 4-Br substituent could be converted to corresponding hydrated product with only 39% yield (Table 1, **1i**). It might be due to the presence of an intervening sp^3 carbon that makes the nitrile carbon less electrophilic, thereby deactivating the substrate.

Based on literature findings, a plausible mechanistic cycle of the reaction is presented in Scheme 1 [38, 39]. The reaction is believed to proceed via intermediary involvement of a Pd-hydroxo complex, formed through opening of N,O-chelate. Most likely, nitrile substrate gets coordinated to one of the palladium centre at *cis* to the hydroxo group through the second ring opening, thereby activating the nitrile carbon towards a nucleophilic attack. Similar type of ring opening mechanism was reported for a Ni(II)-based catalytic system containing N,C-bidentate ligand [38].

Table 1 Hydration of nitriles using the complex **1** as catalyst



Reaction conditions: nitrile substrate 0.3 mmol, KOH 0.15 mmol, *i*-PrOH + H_2O (1:1) 4 cm^3: Yields were recorded in GC

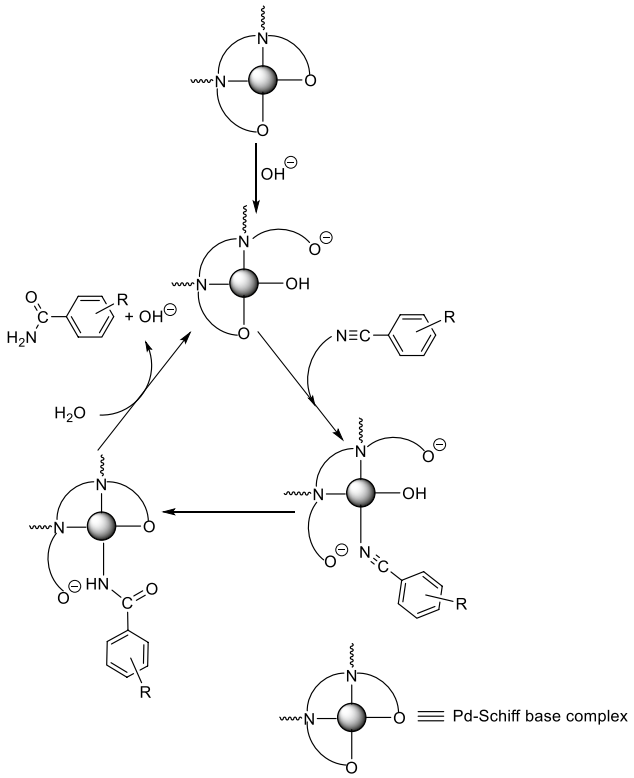
Antibacterial study

Schiff base compounds show active biological properties due to the presence of azomethine (–C=N–) group in their parent structure [40]. Therefore, an antibacterial study of the synthesized compounds was performed to observe the growth inhibitory property of the compounds **1**–**3** against four bacterial species (Table 2). An effective zone of inhibition of 10 ± 2.64 mm against *Bacillus subtilis* indicated a positive result by the compound **2** after 24 h of incubation (Fig. 2). On the other hand, DMSO shows no activity against any test organism in the control.

The MIC value of the compound **2** against the test bacterial sample *Bacillus subtilis* was determined by the standard broth dilution method. The change in colour from yellow to dark brown after adding 2-(4-iodophenyl)-3-(4-nitrophenyl)-2*H*-tetrazolium chloride (INT) solution in compound **2** treated bacterial sample indicated a positive antibacterial result. From the result, it was observed that the microtitre-plate well containing 30 μg cm^{-3} of the compound **2** significantly reduced the bacterial growth and was considered as minimum inhibitory concentration (MIC) value of the compound, and the concentration of 50 μg cm^{-3} was determined as the minimum bactericidal concentration (MBC) value of the compound which completely ceased the test bacterial growth. Surprisingly, minimum concentration values of the compound are found to be better than many other reported Schiff base complexes (Table 3).

Surprisingly, many of the Schiff base-derived compounds show enhanced antimicrobial activity in presence of some specific groups [45]. In the present case, antibacterial

Scheme 1



activity of the compound **2** is probably more pronounced owing to the presence of acetate groups.

Conclusion

In conclusion, three binuclear palladium-Schiff base complexes are synthesized and characterized using various spectroscopic techniques. Further, the catalytic activity of the complexes is screened for hydration of nitriles to amides. One of the complexes showed excellent catalytic performance under mild reaction condition. Interestingly, under the optimized condition, usually less reactive heteroaryl nitriles like 2-furonitrile, 2-thiopenenitrile could also be converted to corresponding amides in excellent yields. In addition, one of the complexes showed effective antibacterial property against *Bacillus subtilis* with MIC value

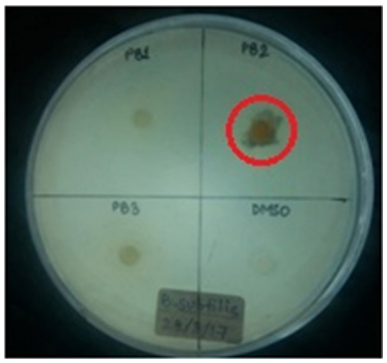


Fig. 2 Zone of inhibition created by compound **2**

30 μg cm^{-3}. Compared to other reported mononuclear Schiff base-derived compounds the present binuclear compound shows superior antibacterial activity which might be attributed to cooperative influence between two metal centres.

Experimental

The chemicals required for different experiments were purchased from Sigma Aldrich, TCI, Merck, and Spectrochem. All bacteriological media used in the work were purchased from HiMedia Laboratories Pvt. Ltd. FT-IR and UV–Vis spectra of the compounds were recorded using Thermo Scientific, Nicolet-iS5 and Shimadzu, UV-1700 spectrophotometers, respectively. NMR spectra of the compounds were recorded in Bruker Ascend 500 MHz FT spectrometer. The elemental analysis (C, H, N) was carried out using PerkinElmer 2400 Series II analyzer. The ESI–MS analysis was carried out using LC-Thermo Scientific Ultimate 3000 MS-TSQ Endura mass analyzer and GC–MS analysis was performed using Agilent 7820A analyzer equipped with Agilent 5975 Series MSD mass detector fitted with HP-5MS capillary column of dimensions 30 m × 0.25 mm × 0.5 mm. Yields of the products were determined using GC analysis of the reaction mixtures in Perkin Elmer, Clarus 480 analyzer fitted with capillary column of dimensions 30 m × 0.25 mm × 0.25 mm.

Table 2 Activity of bacterial strain against different compounds

Name of the bacteria	Type of the bacteria	Result of antimicrobial assay			
		1	**2**	**3**	DMSO
Escherichia coli	Gram negative rod shaped	− ve	− ve	− ve	− ve
Salmonella typhimurium	Gram negative rod shaped	− ve	− ve	− ve	− ve
Bacillus subtilis	Gram positive rod shaped	− ve	+ ve	− ve	− ve
Staphylococcus aureus	Gram positive cocci shaped	− ve	− ve	− ve	− ve

+ ve: The compound shows antibacterial activity, − ve: The compound does not show antibacterial activity

Table 3 Antibacterial activity comparison of different compounds against *Bacillus subtilis*

Sl. No	Compound	MIC / μg cm^{-3}	MBC / μg cm^{-3}	References
1	Kanamycin	3.6	4	[41]
2	Chloramphenicol	4	4.2	[41]
3	Ni-Schiff base complex	75	150	[41]
4	Cu-Schiff base complex	150	300	[41]
5	Cu-Schiff base-bipyridine complexes	125, 62, 62	250, 125, 125	[42]
6	Cu(II) complex of Schiff base and N-donor heterocyclic ligands	150	300	[43]
7	Cu(II) complex of pyrazolone based Schiff base	31.2	62.5	[44]
8	Binuclear Pd(II)-Schiff base complex	30	50	Present work

The ligands 2,2′,2″,2‴-[(1*E*,1′*E*,1″*E*,1‴*E*)-[[1,1′-biphenyl]-3,3′,4,4′-tetrayltetrakis(azanylylidene)]-tetrakis(methanylylidene)]tetraphenol (**L1**) and N^1,N^4-bis(pyridine-2-ylmethylene)benzene-1,4-diamine (**L2**) were synthesized by following the previously reported procedures [35, 36].

2,2′,2″,2‴-[(1*E*,1′*E*,1″*E*,1‴*E*)-[[1,1′-Biphenyl]-3,3′4,4′-tetrayltetrakis(azanylylidene)]tetrakis(methanylylidene)]-tetraphenoxypalladium(II) (1, $C_{40}H_{26}N_4O_4Pd_2$) The complex **1** was prepared by adding a solution of 0.32 g ligand **L1** (0.50 mmol) dissolved in 7 cm^3 MeOH drop-wise to a methanolic solution of 0.23 g $Pd(OAc)_2$ (1.00 mmol). The mixture was then refluxed for 3 h under stirring condition. The complex was supposed to form when colour of the solution changed from chocolate brown to pale brown. The solid compound thus obtained was separated by filtration and washed with MeOH followed by diethyl ether to remove the unreacted ligand and $Pd(OAc)_2$. Solubility: soluble in DMSO and sparingly soluble in alcohols (EtOH, *i*-PrOH, etc.). Colour: orange; yield: 83%; FT-IR (KBr): $\overline{\nu}$ = 1568 ($\nu_{C=N}$), 751 (ν_{M-N}), 642 (ν_{M-O}) cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.17 (s, 4H, CH = N), 7.89–6.74 (m, 22H, Ar–H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ = 163.60 (C = N), 147.21 (Ar, C-N), 133.52 (Ar–C), 132.53 (Ar–C), 132.35 (Ar–C), 131.51 (Ar–C), 128.79 (Ar–C), 119.29 (Ar–C), 119.23 (Ar–C), 118.88 (Ar–C), 118.65 (Ar–C), 117.28 (Ar–C), 116.98 (Ar–C) ppm; MS (ESI): *m/z* = 840 ($[M]^+$).

***N^1,N^4*-Bis(pyridine-2-ylmethylene)benzene-1,4-diaminepalladium(II) acetate (2, $C_{26}H_{26}N_4O_8Pd_2$)** For the synthesis of complex **2**, a solution of 0.14 g ligand **L2** (0.50 mmol) in 7 cm^3 of MeOH was added drop-wise to 8 cm^3 methanolic solution of 0.23 g $Pd(OAc)_2$ (1.00 mmol). After refluxing for 3 h, the complex was supposed to form when colour of the solution changed from brown to maroon. The mixture was filtered followed by evaporation of solvent from the filtrate part under reduced pressure resulted a crude solid. The solid was then washed with MeOH and diethyl ether to obtain the pure complex. Solubility: soluble in DMSO and sparingly soluble in alcohols (EtOH, *i*-PrOH, etc.). Colour: brownish black; yield: 87%; FT-IR (KBr): $\overline{\nu}$ = 1643 ($\nu_{C=O}$), 1560 ($\nu_{C=N}$), 772 (ν_{M-N}), 642 (ν_{M-O}) cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.56 (s, 2H, CH = N), 7.36–6.35 (m, 12H, Ar–H), 2.40 (s, 6H, CH_3) ppm; MS (ESI): *m/z* = 751 ($[M]^+$).

***N^1,N^4*-Bis(pyridine-2-ylmethylene)benzene-1,4-diaminepalladium(II) chloride (3, $C_{18}H_{14}Cl_4N_4Pd_2$)** For the synthesis of complex **3**, initially $[Pd(CH_3CN)_2Cl_2]$ was prepared by dissolving 0.18 g $PdCl_2$ (1.00 mmol) in 10 cm^3 MeCN. The complex thus obtained by filtration was dissolved in 7 cm^3 EtOH and 8 cm^3 ethanolic solution of 0.14 g ligand **L2** (0.50 mmol) was added drop-wise to it. The mixture was refluxed for 3 h until colour of the solution became dark brown. The mixture was filtered followed by evaporation of the solvent under reduced pressure to obtain a crude solid. It was washed with cold EtOH and diethyl ether, dried in vacuum to get the pure complex. Solubility: soluble in DMSO and sparingly soluble in alcohols (EtOH, *i*-PrOH, etc.). Colour: yellowish brown; yield: 82%; FT-IR (KBr): $\overline{\nu}$ = 1563 ($\nu_{C=N}$), 770 (ν_{M-N}), 538 (ν_{M-Cl}) cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ = 9.01 (s, 2H, HC = N), 8.30 (d, 1H, near Py-N), 8.29 (d, 1H, near Py-N), 7.53–7.09 (m, 10H, Ar–H) ppm; MS (ESI): *m/z* = 658 ($[M+1]^+$).

General procedure of catalytic nitrile hydration

To perform nitrile hydration reaction, a round bottomed reaction flask was charged with a nitrile substrate, base, solvent and the catalyst. The mixture was stirred at room temperature or refluxed for a few hours as needed. Progress of the reaction was monitored using TLC under UV lamp and after completion of the reaction, the organic product was extracted with ethyl acetate (15 $cm^3 \times 3$) from the reaction mixture. The exact percentage yields of the desired products were determined from area of the peaks appeared in gas chromatograms of the mixtures. For this purpose, calibration curves were initially recorded using pure amides. Further,

the sample containing the corresponding amide was run in the GC using the same method. Anisole was used as the internal standard in the analysis. Identities of the desired compounds were confirmed from GC–MS analysis.

Evaluation of antibacterial property

The antibacterial property of the synthesized compounds was evaluated by Kirby-Bauer's disc diffusion method using paper discs (diameter = 6 mm) soaked with the respective compounds (**1**–**3**) dissolved in DMSO at a concentration of 1 mg cm^{-3}. The paper discs were carefully placed over individual Muller-Hinton agar (composition (g dm^{-3}): HM infusion B from 300, acicase 17.5, starch 1.5, agar–agar 17, pH = 7.3 ± 0.1 at 25 °C) plates pre-inoculated with 1000 mm^3 respective Gram-positive (*Bacillus subtilis* and *Staphylococcus aureus*) and negative (*Escherichia coli* and *Salmonella typhimurium*) bacterial culture ($OD_{600\ nm}$ = 1.0) followed by incubation at 37 ± 2 °C for 24 h. Appearing a clear zone of inhibition around the impregnated disc(s) after incubation indicates effective antimicrobial activity against the respective test bacterial sample.

After analyzing the results of the disk diffusion method, the potent compound was subjected to determination of minimum inhibitory concentration (MIC) by broth dilution method. For this, 100 mm^3 of test overnight bacterial culture ($OD_{600\ nm}$ = 1.0) was loaded in a 96-well microtitre plate. The bacterial culture was inoculated with different concentrations of the potent compound in such a way so that it exerted the concentration of 0.0, 10, …, 50 μgcm^{-3} in each tube containing the pre-inoculated respective test bacterial culture. After gentle mixing, the inoculated wells were incubated at 37 °C and 135 rpm for 16 h. Precisely, 50 mm^3 of 2-(4-iodophenyl)-3-(4-nitrophenyl)-2*H*-tetrazolium chloride (INT) was added to each well including the control and observed colour development after 2 h to visually differentiate among the live and dead cells. INT on reduction produced a red formazan dye that could be used for quantitative redox assays.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s00706-022-02929-5.

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