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Synthesis of novel binuclear Cu(II) complex and its application as catalyst for conversion of amine to imine

Aieshri Swargiary[a], Somali Mukherjee[b], Dulu Brahma[a], Pranjal Kalita[a] & Arunendu Mondal*[a]

[a] Department of Chemistry, Central Institute of Technology Kokrajhar, Kokrajhar 783 370, Assam, India
(Deemed to be University under MHRD, Govt. of India)

[b] Department of Chemistry, Scottish Church College, 1 and 3 Urquhart Square, Kolkata 700 006, India

E-mail: a.mondal@cit.ac.in

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Oxidative coupling reaction of benzylamine to imine has been achieved with almost 90% yield under very mild reaction conditions by applying a novel binuclear Cu(II) complex as a catalyst. Binuclear Cu(II) complex, $[Cu_2(2,3\text{-pydc})(2,2'\text{-bpy})_4](ClO_4)_2.2H_2O$ [**A**] has been synthesised using the ligands 2,3-pyridinedicarboxylic acid (2,3-pydc) and 2,2'-bipyridine (2,2'-bpy). The newly synthesised catalyst has been characterised by using single crystal X-ray diffraction study (SC-XRD), UV-Vis spectroscopy, IR-spectroscopy and elemental analysis.

Keywords: Cu(II) complex, 2,3-Pyridinedicarboxylic acid, 2,2'-Bipyridine, Catalyst, Benzylamine, Imine

Nowadays construction of inorganic coordination materials have received wide interest in the field of applied chemistry. These inorganic coordination materials are generally constructed by using inorganic metal moieties and organic ligands. Depending on the bonding mode of organic ligands, the coordination compounds can form discrete, 1D, 2D or 3D structures with excellent stability[1–4]. These materials have been found to get utilized in various field of applied chemistry *viz.* catalysis[5,6], water harvesting[7], gas storage and separation[8,9], antibacterial activity[10], magnetism[11] and luminescent properties[12] and also in medical field like drug delivery[13,14] and others. The construction of desired materials with required functional properties depends on several factors such as metal ions, nature of ligands, metal to ligand ratio, *p*H, solvent system, *etc*.[15] Selection of potential organic ligands with different coordination modes plays a very important role in the synthesis of these inorganic-organic hybrid materials[16–18].

In the recent study it is seen that various pyridine dicarboxylic acid ligands have emerged interest in the mind of many researchers because both the deprotonated carboxylic acids can act as either bidentate or bridging ligands[19,20]. These ligands have different coordination modes that can coordinate with transition metals through its carboxyl oxygen atoms and aromatic nitrogen atom to provide structurally versatile complexes with desired properties. The chemistry of pyridinedicarboxylic acids provokes the researchers to utilize the ligands in the synthesis of inorganic-organic hybrid materials. Driving force to synthesis new inorganic-organic materials and use it as a catalyst is perceived from the earlier works, where researchers have used different carboxylic acid substituted pyridine as ligands and synthesised structurally diverse compounds with different applications[19–26].

Production of imines by oxidative coupling of benzylamines have been received much more attention because of its applications in chemical synthesis of different useful organic compounds. The presence of unsaturated C = N bond in imines makes it highly reactive and as a result it becomes highly demandable in pharmaceutical industry, agrochemical industries and synthesis of fine chemicals, dyes, fragrances and fungicides, *etc*.[27] Imines also play a very significant role in the synthesis of biologically active nitrogen containing organic compounds[28–33]. Various works on oxidation of benzylamines to imines have been reported earlier by different Cu-salts[34], copper-supported alumina[35], copper and cerium oxide[36], and Cu/chitosan bead[37], *etc*. as catalysts. Recently B. Venuetal[38]. and S. Z. Anbardan *et al*.[39] have reported the use of Cu based MOF as catalyst for oxidative coupling of amines to imines.

Very recently Swargiary *et al.* reported the application of copper based coordination polymer as catalyst for similar reaction[40]. Based on literature report, a new coordination compound has been synthesized using copper with deprotonated 2,3-pyridinedicarboxylic acid as organic bridging ligand and 2,2'-bipyridine as auxiliary chelating ligand to increase the stability of the complex[1,2]. This newly synthesized material has been used as catalyst for oxidative coupling of amine to imine and the result shows that its ability to act as a catalyst for the reaction is excellent. This is the first example where a dimeric Cu-complex has been used as a catalyst for such conversion.

Experimental Section

The chemicals like $Cu(ClO_4)_2.6H_2O$ is purchased from Sigma Aldrich, 2,3-pyridinedicarboxylic acid (2,3-pydc) from Hi-Media Laboratories and 2,2'-bipyridine (2,2'-bipy) from Alfa Aesar and are used directly without purification. Benzylamine is taken from Pallav and tert-butyl hydroperoxide 70% in water (TBHP) is taken from Loba Chemie Pvt Ltd respectively. Solvents like methanol and acetonitrile are taken from Emplura; petroleum ether and ethyl acetate are taken from SRL.

Characterization techniques

Fourier-Transform Infra-red (FTIR) spectroscopy data is taken in the range of 4000 to 400 cm^{-1} region from Shimadzu FTIR 8201 spectrophotometer using KBr pellet. Shimadzu UV-2600 UV-Vis spectrophotometer has been used to take solid state UV-spectrum taking $BaSO_4$ as reference material. Elemental analysis study has been done by EuroEA elemental analyzer. SII 6300EXSTAR thermogravimetric analyzer has been utilized for thermal analysis. The heating rate during analysis was 10°C/ min in N_2 atmosphere. Single crystal-XRD data is collected on BRUKER D8 VENTURE SC-XRD using Mo-K∞ as the X-ray source. Powder-XRD data has been taken with EMPYREAN Diffractometer system and DMSO-d_6 solvent has been used to take NMR spectrum with BRUKER 400MHz NMR spectrometer.

Synthesis of $[Cu_2(2,3\text{-pydc})(2,2'\text{-bpy})_4](ClO_4)_2 2H_2O$(A)

$Cu(ClO_4)_2.6H_2O$ (1mmol), 2,3-pydc (0.5mmol), 2,2'-bpy (2mmol) are dissolved in MeOH (10 mL), CH_3CN (10 mL) and H_2O (10 mL) at RT. The precipitate is filtered off and the filtrate is kept undisturbed in an open environment. Blue crystals are obtained after a few days with 36.21% yield. Analytical calculation for $Cu_2C_{47}H_{39}N_9O_{18}Cl_2$ (%): C 49; N 11; H 3.3. Found: C 45; N 13; H 3.2. It should be noted that because of poor quality data the exact number of solvent molecules could not be determined through single crystal X-ray analysis. However, considering experimental elemental analysis and spectral data, the most probable formula of the compound turns out to be $[Cu_2(2,3\text{-pydc})(2,2'\text{-bpy})_4](ClO_4)_2\ 2H_2O$ (A). The key unit of binuclear Cu(II) center could easily be identified through X-ray single crystal data analysis.

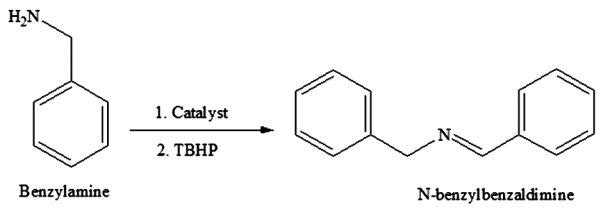
Catalytic activity

15 mg of catalyst is added slowly into the mixture of 10 mmol benzylamine and 15 mmol TBHP. The reaction starts immediately as soon as the catalyst is added and continued to stir for about 30 minutes at RT. The reaction is monitored by taking in TLC at different time interval. Column chromatography has been performed to isolate the pure imine product. The formation of imine product is confirmed by taking NMR spectroscopy. The catalytic reaction is represented in Scheme 1.

Results and Discussions

IR Spectroscopy

In the IR-spectrum (Fig. 1) of compound **A,** it is seen that the strong and broad peak in the range of 3343 cm^{-1} is for water molecule. The weak peaks of C-H stretches of ligand are observed in the range of 3122 cm^{-1} and 3076 cm^{-1} respectively[41]. The asymmetric (ν_{as}) and symmetric (ν_{s}) stretching modes of the COO groups are seen at the range of 1658-1592 cm^{-1} and 1442-1389 cm^{-1} respectively which signify the different coordination modes of C-O of the carboxylic groups of the 2,3-pydc with the Cu(II) ions. The characteristic peaks observe at 1063 cm^{-1} and 768 cm^{-1} are due to pyridine ring compound and O-C-O vibrations of 2,3-pydc ligand[42,43]. The peaks



Scheme 1 — Oxidative coupling reaction of benzylamine

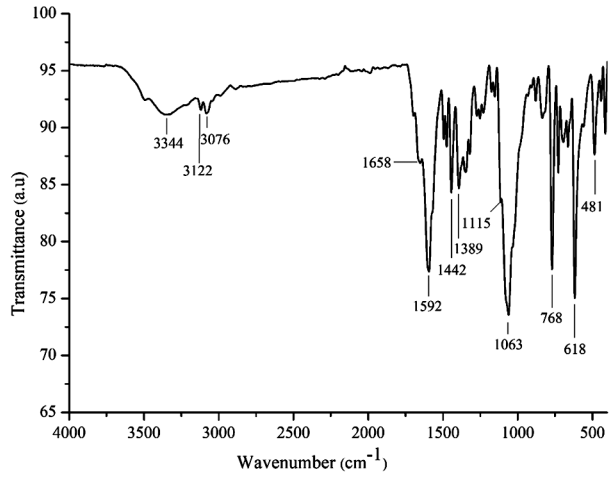


Fig. 1 — IR spectrum of compound A

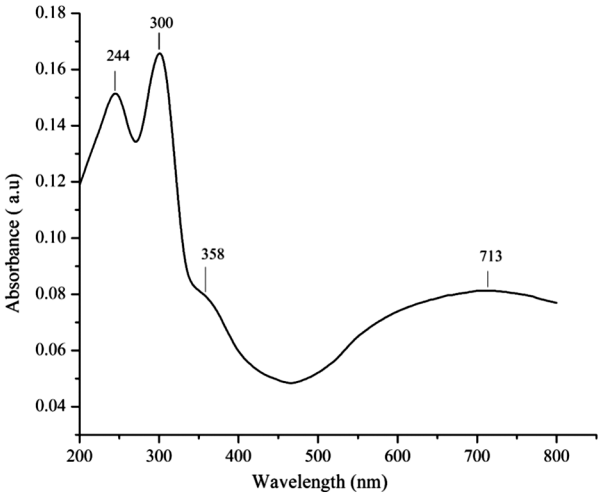


Fig. 2 — UV-Vis spectrum of compound A

observe at 618 cm^{-1} and 481 cm^{-1} may be mainly due to vibrations of Cu-O and Cu- N respectively[44].

UV-Visible spectroscopy

The UV-Visible spectrum (Fig. 2) of compound **A** in solid state is taken at RT in the range of 200 – 850 nm taking $BaSO_4$ as reference. The absorption bands of intra ligand π - π^* transitions are generally seen below 320 nm[45]. In this compound the absorption bands at 244 nm and 300 nm are assumed to be for π – π^* transitions[45]. The absorption band for ligand to metal charge transfer is assumed to be seen at 358 nm[45]. This band indicates that the ligand is coordinated to the metal center. Very broad band around 713 nm is generally assigned for d-d transitions[44].

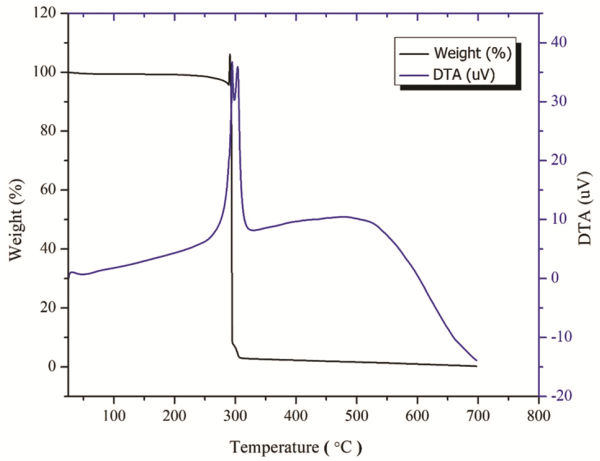


Fig. 3 — TGA-DTA curve of compound A

TGA -DTA analysis

The TGA-DTA curve in nitrogen atmosphere is shown in Fig. 3. The simultaneous TGA-DTA data have been taken in the temperature range of 25 – 800°C with heating rate of 10°C/ min. From the graph it is seen that the compound is stable up to 295°C without melting, after that complete decomposition of ligands takes place. It is also revealed that there is steady loss of solvent molecules from RT to 290°C. DTA graph corresponding to the decomposition reveals a two steps exothermic decomposition process.

Single crystal XRD analysis

The crystallographic study of compound **A** reveals that the compound crystallizes in orthorhombic crystal system with space group Aba2. The cell parameters of the crystal structure are a = 23.477(4) Å, b = 23.644(3) Å and c = 19.619 (3) Å with cell angles $\alpha = \beta = \gamma = 90^o$ respectively. The cell volume is 10890.3 $Å^3$. Because of the poor-quality data, missing residues (exact number of solvent molecules and perchlorate ions) could not be located in the Fourier map. In addition to that, shift error could not be fixed. So, we were only able to reach primary solution which reveals that the key unit surrounding the metal coordination site is a binuclear moiety. The ORTEP view of the crystal structure of the compound is shown in Fig. 4. The asymmetric unit of the dimeric copper (II) compound consists of one 2,3-pydc and four N-donor chelating ligand 2,2'-bpy. From the crystal structure it is seen that each Cu(II) atoms are exhibited by distorted square pyramidal geometry. The Cu (1) atom is coordinated by one oxygen atom O1 and one nitrogen atom N5 from 2,3-pydc and four

nitrogen atoms N1, N3 and N2, N4 from two different 2,2'-bpy. The other Cu (2) atom is coordinated by one oxygen atom O4 from the same 2, 3-pydc and four N-atoms N6, N8 and N7, N9 from two different 2,2'-bpy. The ligand 2,3-pydc acts as bridge between the two copper atoms as one oxygen atom O1 of one carboxyl and one nitrogen atom N5 is coordinated to one Cu1 atom and the one oxygen atom O4 of other carboxyl of the same 2,3-pydc is coordinated to other Cu2 atom. The other two oxygen atoms of the two different carboxyl of the same 2,3-pydc remains uncoordinated. The Cu…..Cu distance in the complex is 8.086Å respectively. Some selected bond distances, bond angles and hydrogen bonding parameters of $[Cu_2(2,3\text{-pydc})_1(2,2'\text{-bpy})_4](ClO_4)_2.2H_2O$ are given in Table 1, Table 2 and Table 3 respectively.

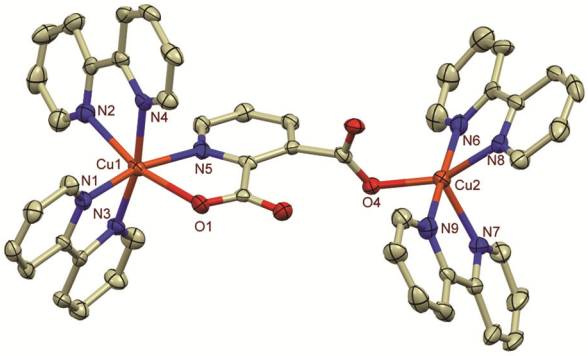


Fig. 4 — ORTEP view of compound **A** with 20% ellipsoid probability. The lattice solvent molecules and hydrogen are not shown for clarity

Table 1 — Selected bond distances of $[Cu_2(2,3\text{-pydc})(2,2'\text{-bpy})_4](ClO_4)_2\ 2H_2O$(A)

	Bond Distances (Å)
Cu1-O1	2.246(9)
Cu1-N1	2.057(10)
Cu1-N2	2.272(12)
Cu1-N3	2.017(10)
Cu1-N4	2.044(11)
Cu1-N5	2.105(10)
Cu2-O3	2.812(10)
Cu2-O4	2.005(8)
Cu2-N6	1.983(12)
Cu2-N7	2.158(12)
Cu2-N8	2.034(10)
Cu2-N9	1.974(11)

Catalytic applications

Oxidative-coupling reactions of benzylamine to imine

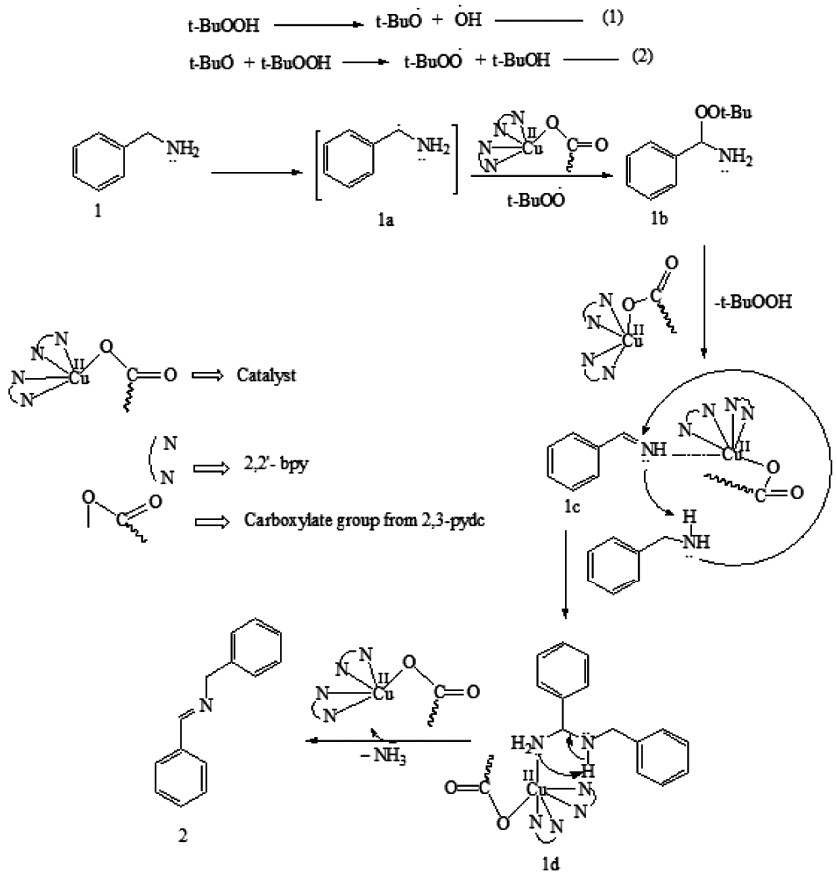
A number of reports for oxidative coupling reactions of benzylamine with different copper-based catalysts have been reported earlier. Use of Cu/chitosan beads as catalyst for oxidative coupling of benzylamine in acetonitrile at 80 °C with TBHP as

Table 2 — Selected bond angles of $[Cu_2(2,3\text{-pydc})(2,2'\text{-bpy})_4](ClO_4)_2\ 2H_2O$ (A)

	Bond Angle (°)		Bond Angle (°)
O1-Cu1-N1	92.1(4)	N4-Cu1-N5	91.4(4)
O1-Cu1-N2	165.3(4)	O3-Cu2-O4	51.8(3)
O1-Cu1-N3	99.6(4)	O3-Cu2-N6	90.8(4)
O1-Cu1-N4	92.2(4)	O3-Cu2-N7	151.4(4)
O1-Cu1-N5	76.3(3)	O3-Cu2-N8	105.1(4)
N1-Cu1-N2	97.9(4)	O3-Cu2-N9	88.3(4)
N1-Cu1-N3	80.0(4)	O4-Cu2-N6	92.1(4)
N1-Cu1-N4	96.4(4)	O4-Cu2-N7	102.7(4)
N1-Cu1-N5	166.3(4)	O4-Cu2-N8	155.6(4)
N2-Cu1-N3	92.7(4)	O4-Cu2-N9	91.8(4)
N2-Cu1-N4	76.1(4)	N6-Cu2-N7	104.4(5)
N2-Cu1-N5	94.9(4)	N6-Cu2-N8	79.0(5)
N3-Cu1-N4	167.7(4)	N6-Cu2-N9	174.1(5)
N3-Cu1-N5	94.7(4)	N7-Cu2-N8	101.6(5)

Table 3 — Hydrogen bonding parameters of $[Cu_2(2,3\text{-pydc})(2,2'\text{-bpy})_4](ClO_4)_2\ 2H_2O$ (A)

D-H···A	D-H (Å)	H···A (Å)	D···A (Å)	<D-H··· A (°)	Symmetry
O6--H6A··O5	0.8400	2.1200	2.83(4)	141.00	1–x,2–y,z
O6--H6B··O5	0.8500	2.1800	2.83(4)	133.00	x, y, z
O7--H7A··O6	0.8500	2.4100	2.71(4)	101.00	x, y, z
O8--H8A··O2	0.8500	2.2300	2.776(17)	122.00	x, y, z
O10--H10A··O10	0.8500	2.6000	3.07(3)	116.00	–x,2–y,z
C2--H2··O3	0.9300	2.3300	3.248(16)	167.00	1/2–x,–1/2+y,z
C7--H7··O3	0.9300	2.5400	3.450(16)	168.00	1/2–x,–1/2+y,z
C17--H17··O2	0.9300	2.4700	3.389(19)	169.00	1/2–x,y,1/2+z
C20--H20··O1	0.9300	2.5000	3.090(18)	122.00	x, y, z
C34--H34··O1	0.9300	2.5500	3.364(17)	146.00	1/2–x,1/2+y,z
C47--H47··O3	0.9300	2.5700	3.25(2)	130.00	x, y, z
C48--H48A··O4	0.9600	2.4900	3.42(4)	165.00	x, y, z
C48--H48C··O10	0.9600	1.7400	2.60(5)	146.00	x, y, z
C49--H49A··O8	0.9600	2.1000	2.91(4)	141.00	x, y, z



Scheme 2 — Mechanism for catalytic reaction

oxidising agent gives good yield. However when the same reaction condition is used for $Cu(OAc)_2$, benzylamine is fully converted to imine with low yield[37]. Venu *et al*.[38] have reported that low yield of imine is obtained when Cu-BTC MOF is used as catalyst for oxidative coupling of benzylamine in acetonitrile at 80 °C with O_2 as oxidant. However when the same reaction is performed in absence of solvent gives good result. Anbardan *et al*.[39] have also reported the exploration of Cu-BDC MOF as catalyst for oxidative coupling of benzylamine to imine in THF at RT to yield high to moderate imine product. Similar conversion of amine to imine with copper containing coordination polymer as catalyst has been reported by Swargiary et.al very recently[40].On the basis of the earlier reported works, we have synthesised the binuclear copper(II) complex material as catalyst for oxidative coupling reaction of benzylamine using TBHP as oxidising agent in absence of solvent. The experimental results obtain by loading different amount of catalysts is shown in Table 4. It is seen that 15 mg of catalyst loaded shows good catalytic activity with good yield (Table 4, entry 3). The blank test is also performed to know the effect of the catalyst at the same reaction condition (Table 4, entry 5).

Confirmation of imine product

The imine product is confirmed by ^{1}H NMR (Fig. S1) and ^{13}C NMR (Fig. S2) spectroscopy. The ^{1}H NMR data reveals that the peaks in the range of δ 7.25 to 7.80 are for the two aromatic rings. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.50 (s, 1H), 7.798-7.342 (m, 5H), 7.333-7.232 (m, 5H), 4.77(s, 2H); ^{13}C NMR (101 MHz, DMSO-d_6): δ 161.72, 139.57, 136.02, 130.71, 128.64, 128.32, 127.93, 127.86, 126.74, 63.94.

Mechanism of the catalytic reaction

The possible route for the oxidation of benzylamine to imine with oxidising agent TBHP in the presence of catalyst (**A**) is shown in Scheme 2. The proposed mechanism is very much similar to that

Table 4 — Optimization of reaction parameters of oxidation of benzylamine

Entry	Substrate	Catalyst (mg)	Yield (%)
1	Benzylamine	5	50
2	Benzylamine	10	65
3	Benzylamine	15	90
4	Benzylamine	20	91
5	Benzylamine	–	0

Reaction condition: Benzylamine (10 mmol), TBHP (15 mmol), Temperature (35 °C) in absence of solvent

of reported one[39, 40]. It is assumed that because of coordinative un-saturation, the five coordinated Cu-atom is the active site for the catalytic reaction. The mechanism shows that the catalyst **A,** oxidized TBHP into tert-butylperoxy radical. Then, this peroxy radical abstract hydrogen from benzylamine to convert into **1b** which subsequently transform into imine **1c** by elimination of TBHP. Further addition of benzylamine to imine along with removal of ammonia results N-benzylbenzaldimine **2**.

Conclusion

A new Cu(II) coordination compound with 2,3-pydc and 2,2'-bpy as ligands is successfully synthesised by very simple solvolysis method. The Cu(II) material has been characterized by FTIR, TGA-DTA, UV-Visible and SC-XRD. The SC-XRD study reveals that the crystalline material is dimeric in structure. The material is then explored for oxidative coupling of benzylamine to imine and found that catalyst shows good result with 90% yield even at RT.

Supplementary Information

Supplementary information is available in the website http://nopr.niscpr.res.in/handle/123456789/58776. The CIF file of X-ray single crystal data has been deposited to CCDC (CCDC No: 2224998).

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