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Synthesis and characterization of Ni(II) and Cu(II) complexes based on quercetin Schiff base and using them as heterogeneous catalysts in Henry reaction

Zahra Moodi & Ghodsieh Bagherzade*

Department of Chemistry, College of Sciences, University of Birjand, Birjand 97175-615, Iran

*E-mail: gbagherzade@gmail.com, bagherzadeh@birjand.ac.ir

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Quercetin is a dietary flavonoid, which with its specialized structure can form Schiff bases and their metal complexes. Schiff base ligand has been synthesized through the condensation of quercetin and ethanolamine. In this paper, two new metal complexes involving the Schiff base ligand have been prepared by a simple method at room temperature. SBL-Cu and SBL-Ni have been characterized by elemental analysis, ICP, UV-visible, and FT-IR studies. The activity of the synthesized complexes as a heterogeneous catalyst has been assessed in Henry reaction. The complexes have shown high catalytic performance in Henry reaction. However, SBL-Cu had better catalytic activity than SBL-Ni. These catalysts can be recycled and reused five times without a significant reduction in the activity. A series of benzaldehyde derivatives have been applied in the optimized Henry reaction to investigate the effect of the substituent group on the yield of the reaction. As expected, benzaldehydes containing electron-withdrawing group have a higher yield in Henry reaction.

Keywords: Schiff base complex, Quercetin, Heterogenous catalysis, Henry reaction

Flavonoids are polyphenolic secondary metabolites that have shown different biological and pharmaceutical activities such as antiviral, antibacterial, neuroprotective, cardioprotective, antioxidant, and anticancer properties.[1] Due to the presence of carbonyl, hydroxyl groups, and nitrogen donors in some synthesized compounds, flavonoids can chelate or bind with metal ions. This ability increases the pharmacological and biological properties of the flavonoids and produces these properties of small molecules.[2,3]

Quercetin (3,3',4',5,7-pentahydroxyflavone, Fig. 1) is one of the dietary flavonoids, distributed in medicinal plants, vegetables, and fruits.[4] In the last two decades, quercetin attracted considerable attention because of its biological and pharmaceutical activities such as antioxidant, antibacterial and anticancer.[5-7] Quercetin is built from two aromatic rings (benzoyl ring A and cinnamoyl ring B), having several hydroxy groups joined by O-heterocycle. With the presence of chelating sites in the quercetin structure, quercetin is capable of forming complexes with metal ions. The potential sites of chelation in quercetin are 5-hydroxychromone, 3-hydroxychromone, and 3′,4′-dihydroxyl groups.[8]

Schiff bases are typically formed by the condensation of primary amines with aldehydes and ketones. Schiff bases play an important role in the development of coordination chemistry. These compounds are characterized by the C=N group in their structure.[9] Schiff bases and their metal complexes are considered privileged compounds in coordination chemistry, due to their electron properties, biological activities, industrial applications, availability, solubility in conventional organic solvents, and a wide range of structures.[10-14]

Many of the bioactive compounds used as drugs have modified pharmacological and toxicological potentials when a metal ion coordination with chelating ligands.[15-18] In general, metallic elements are crucial for living systems. Metals can lose their electrons to form positively charged ions that tend to be soluble in biological systems. Biomolecules such as proteins and DNA are electron-rich, and metal ions are electron deficient, so that metals can serve as electron donors, and they bind to biological molecules. The same principle applies according to the affinity of the metal ions for small molecules and ions which are essential to life, such as O_2. Metals play important roles in biological functions such as oxygen and electron transport within the body. Copper ions have demonstrated a wide range of effectiveness against micro-organisms. Copper-containing compounds such as copper sulfate,

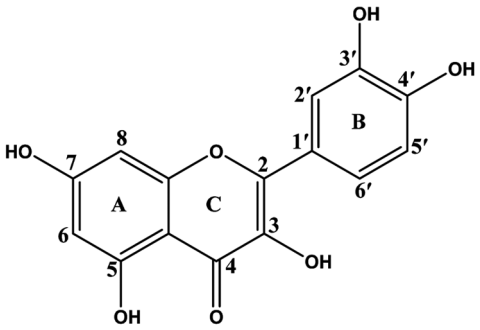


Fig. 1 — structure of quercetin

Bordeaux mixture, etc., are known to control most plants' fungi and bacterial diseases on most crops. Natural chelators reduce copper toxicity by binding available parts of the copper ions, so that they can be considered as a protective agent against fungi and bacteria.[19] Copper (II) complexes have a wide variety of applications, such as catalyst in organic reactions, anticorrosion agents, and a broad spectrum of pharmacological activities.[20] Nickel is an essential trace element required for many biological processes. Nickel ions act as cofactors for several enzymes in an organism from all kingdoms of life. These enzymes catalyze many significant chemical reactions.[21] Nickel complexes have been reported for various biological activities such as antifungal,[22] antibacterial[22–24], and antiproliferative/anticancer properties.[25–28]

Many metal complexes of Schiff bases have a significant role as a catalyst in a variety of reactions. These compounds enhance the yield of the reaction.[29] Nitro aldol or Henry reaction is one of the most important reactions for the construction of C-C bonds in organic chemistry. The product of the Henry reaction is β-nitro alcohol, which can be converted to valuable building blocks of natural products or medicinal such as α-hydroxycarboxylic acids, β-amino alcohols, aziridines, α-nitro ketones, α-hydroxy ketones, sulfides, and aldehydes.[30,31] This study aims to synthesize and characterize the new copper(II) and nickel(II) complexes of quercetin Schiff base as well as to find out their catalytic activity in the Henry reaction.

Experimental Details

General information

All chemicals and solvents were in analytical grade and used without further purification. FT-IR spectra were performed using a JASCO FTIR- 4200 instruments by KBr technique. UV-visible spectra were recorded using SHIMADZU-1800 model UV-visible spectrophotometer. Elemental analysis was done by a Vario EL III CHNOS Elemental Analyzer. Inductively coupled plasma (ICP) atomic emission spectroscopy was performed by OPTIMA 7300DV. A Bruker DPX-250 Avance 300 MHz spectrometer was employed to determine the ^{1}H and ^{13}C NMR spectra in DMSO-d6 with TMS as an internal standard. The thermogravimetric analysis was recorded using a Perkin Elmer thermal analyzer with a heating rate of 10 °C/min under nitrogen atmosphere. PXRD analysis was performed using Philips diffractometer (Model TMe1800).

Synthesis of Schiff base ligand

For preparing Schiff base ligand (SBL), 1 mmol of quercetin (0.302 g) was dissolved in ethanol (7 mL). 57 μL of glacial acetic acid was added to the ethanolic quercetin solution, and after 30 min, ethanolamine (1 mmol, 60.2 μL) was added dropwise. After being stirred and reflux for 8 h at 60 °C, the dark red precipitation was filtered and washed with ethanol. Finally, the synthesized Schiff base ligand was obtained after recrystallization from a hot solution of ethanol and dried in a vacuum oven. SBL has been characterized with the spectroscopy method.[32]

Synthesis of the metal Schiff base complexes

SBL (1.0 mmol) was dissolved in ethanol. Triethylamine (1.0 mmol) was added to the solution. After 5 min, $Cu(OAc)_2.2H_2O$ (1 mmol) was added to the solution quickly. The mixture was stirred for 12 h at room temperature. The final product was obtained by centrifugation of the mixture, washing with ethanol (3 times), and drying in a vacuum oven. This procedure was repeated with $Ni(OAc)_2.4H_2O$ to produce a corresponding complex.

SBL-Cu: Yield: 82%; Anal. Calc. for $C_{21}H_{23}CuNO_{13}$ (%): C (44.98), H (4.10), N (2.47), O (37.06), and Cu (11.39). Found: C (44.96), H (4.13), N (2.50), O (37.08), and Cu (11.33). IR main peaks (KBr, cm^{-1}): 1566 (ν C=N), 1355 (ν C-O-H), 1636 (C=C), 3189 (ν O-H), 1241 (ν C-O-C), 625 (ν Cu-O).

SBL-Ni: Yield: 80%; Anal. Calc. for $C_{21}H_{23}NNiO_{13}$ (%): C (45.38), H (4.16), N (2.54), O (37.37), and Ni (10.55). Found: C (45.36), H (4.17), N (2.52), O (37.40), and Ni (10.55). IR main peaks (KBr, cm^{-1}): 1550 (ν C=N), 1361 (ν C-O-H), 1630 (C=C), 3310 (ν O-H), 1265 (ν C-O-C), 621 (ν Ni-O).

Typical catalytic procedure for Henry reaction

After optimizing the reaction, the best results were obtained using 0.5 mmol (0.051 mL) benzaldehyde, 2.5 mmol (135 μL) nitromethane, 1 mol% SBL-Cu or

SBL-Ni as a catalyst in the presence of ethanol as a solvent. The reaction was performed at 70 °C. The amount of catalyst, temperature, and solvent nature were the significant factors in the optimizing process (Table 1). The progress of the reaction was monitored by thin-layer chromatography (TLC). The reaction was completed after 24 h and was cooled at room temperature. The catalyst was separated from the reaction mixture by centrifuging, washed with EtOAc, and used for the next run. The product was purified on a silica gel plate. To evaluate the performance of the catalysts, the Henry reaction was performed with different benzaldehydes and nitromethane, and the yield of the reaction was evaluated.

Results and Discussion

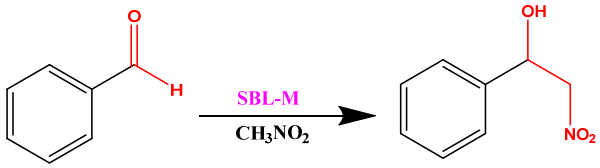
Characterization of SBL-Cu and SBL-Ni

SBL, SBL-Cu, and SBL-Ni were stable at room temperature. The ligand and complexes were characterized by elemental analysis, UV-visible spectra, PXRD analysis, TGA, and FT-IR data. NMR spectroscopy has been performed for the ligand (Fig. S1 and S2, Supplementary Data), but since Cu^{2+} and Ni^{2+} are paramagnetic, ^{1}H NMR was not performed. Some physical properties and elemental analysis data of the ligand and its copper(II) and nickel(II) complexes are listed in Table 2.

Absorption spectroscopic study of SBL, SBL-Cu, and SBL-Ni

The UV-visible absorption spectra of the ligand and synthesized complexes are shown in the Fig. 2. SBL shows two absorbance bands in the absorption spectrum. The band at 365nm (band I) is related to B-ring absorption in quercetin structure (cinnamoyl system), and the absorption band at 255 nm (band II) is represented A-ring (benzoyl system) in the structure of the quercetin. The absorption spectra of the complexes in comparison to the ligand show bathochromic shifts. The spectral changes confirmed that the complexation took place between the ligand and metal ions. Thus, the UV-visible spectrum showed significant information about the coordination sites. Since the absorption band at 365 nm in ligand

Table 1 — Optimized conditions for Henry reaction between benzaldehyde and nitromethane catalyzed by SBL-M (M=Cu, Ni)[a]



	Entry	Solvent	Catalyst amount (%)	Time (h)	Time (°C)	Yield (%)[b]
	1	H_2O		48	70	10
	2	H_2O	0.5	48	60	50
	3	H_2O	0.5	48	70	60
	4	H_2O	1	48	60	65
	5	H_2O	1	48	70	70
M=Cu	6	EtOH		24	60	20
	7	EtOH	0.5	24	60	68
	8	EtOH	0.5	24	70	75
	9	EtOH	1	24	70	83
	10	CH_3CN	1	48	60	25
	11	THF	1	48	60	50
	1	H_2O		48	70	8
	2	H_2O	0.5	48	60	30
	3	H_2O	0.5	48	70	40
	4	H_2O	1	48	60	48
	5	H_2O	1	48	70	55
M=Ni	6	EtOH		24	60	14
	7	EtOH	0.5	24	60	45
	8	EtOH	0.5	24	70	55
	9	EtOH	1	24	70	70
	10	CH_3CN	1	48	60	10
	11	THF	1	48	60	30

a: reaction condition benzaldehyde (0.5 mmol) and nitromethane (2.5 mmol), b: Isolation yield

Table 2 — Molecualr formula, colour yield and elemetal analysis of SBL, SBL-Cu and SBL-Ni

Compound (Molecualr formula)	Colour	Yield (%)	Found (Calculated)				
			C (%)	H (%)	N (%)	O (%)	M (%)
SBL ($C_{17}H_{15}NO_7$)	Orange	87%	59.10 (59.13)	4.41 (4.38)	3.98 (4.06)	32.51 (32.43)	
SBL-Cu ($C_{17}H_{15}NO_9$)	Black	88%	45.83 (46.00)	4.16 (4.08)	3.20 (3.16)	32.42 (32.44)	14.39 (14.32)
SBL-Ni ($C_{17}H_{15}NO_9$)	Brown	80%	46.47 (46.51)	4.18 (4.13)	3.25 (3.19)	32.68 (32.80)	13.42 (13.37)

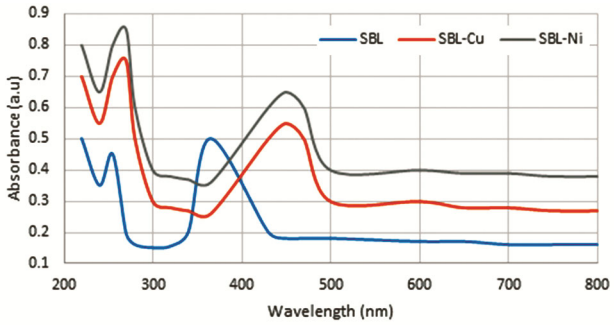


Fig. 2 — UV-visible absorption spectra of SBL, SBL-Cu, and SBL-Ni

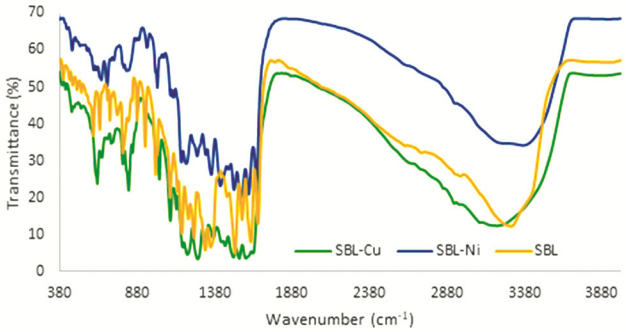


Fig. 3 — FT-IR spectra of SBL, SBL-Cu, and SBL-Ni

has a more bathochromic effect than the other one, thus the cinnamoyl system is the better site for coordination. As the 3-OH group has a more acidic proton, it is expected to have coordination among the azomethine group with metal ions.

FT-IR studies of the complexes

The FT-IR spectra of the SBL ligand, SBL-Cu, and SBL-Ni are shown in the Fig. 3. The main peaks of SBL, SBL-Cu, and SBL-Ni are listed in Table 3. The Schiff base ligand showed a characteristic peak at 1660 cm^{-1}, indicated C=N stretching frequency, while this band appeared at 1566 cm^{-1} in SBL-Cu and at 1550 cm^{-1} in SBL-Ni. It could be suggested that the coordination takes place between the azomethine groups with metal ions. In the SBL spectrum, the bands at 1559 cm^{-1} and 1247 cm^{-1} are related to ν (C=C) and ν (C-O-C) frequency, respectively, which are shifted in the complexes. The broad bands for ν (O-H) at 3220 cm^{-1} (SBL-Cu) and 3310 cm^{-1} (SBL-Ni) may be assigned the presence of water in the structure of the complexes. Moreover, the presence of ν (M-O) stretching vibration at 625 cm^{-1} and 621 cm^{-1} for SBL-Cu and SBL-Ni, respectively indicates the formation of Copper (II) and Nickel (II) complexes. The ligand (SBL) doesn't exhibit this band.

Thermal study of the complexes

Thermal studies of complexes have provided helpful information to investigate the complex structure. TGA curves of SBL-Cu and SBL-Ni were similar (Fig. 4). The mass loss in these complexes at 50 °C has corresponded to the superficial moisture. The weight loss at 150 °C was attributed to the loss of two coordinated water. Furthermore, 50% of the mass loss was observed at 510 °C, which is indicated the high thermal stability of the complexes.

Powder XRD studies of the complexes

The crystal structure of the complexes were confirmed by powder X-ray diffraction (XRD) analysis. XRD patterns of SBL-Ni (Fig. 5) were showed remarkable peaks at 2θ of 37.22°, 43.15°, 62.85°, 75.27°, and 79.25° are indexed as (111), (200), (220), (311) and (222), respectively. The crystalline structure of SBL-Ni has belonged to the cubic system, space group Fm-3m. XRD patterns of SBL-Cu (Fig. 6), peaks at 2θ 32.6°, 35.7°, 38.8°, 48.6°, 53.2°, 61.6°, 66.1°, 68.2°, 72.4°,and 76.1° are corresponded to the values of (110), (002), (111), (202), (020), (113), (311), (220), (311) and (222) respectively. The results confirmed that SBL-Cu belonged to monoclinic structure.

Determination of stoichiometry of SBL-Cu and SBL-Ni

Job's method was applied to determine the stoichiometry ratio of metal-ligand complexes. In this method, a series of solutions with different ligand-metal concentration ratios were prepared. To find the metal-ligand ratio in SBL-Cu and SBL-Ni, the optical

Table 3 — FT-IR main peaks of SBL-SBL-Cu, and SBL-Ni

Compound	υ (C=N)	υ (C=C)	υ (O=H)	υ (C-O-C)	υ (M-O)
SBL	1660	1559	3304	1247	
SBL-Cu	1566	1636	3220	1241	625
SBL-Ni	1550	1630	3310	1239	621

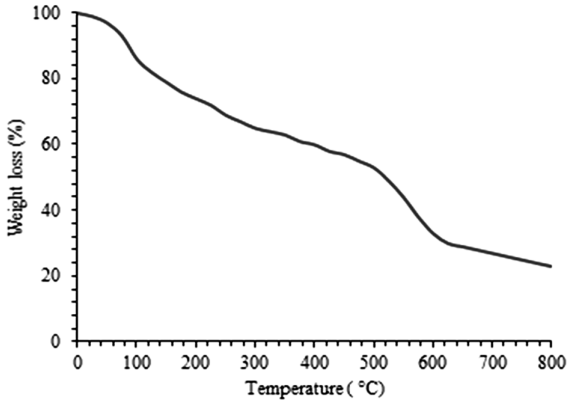


Fig. 4 — TGA curve of SBL-Cu and SBL-Ni

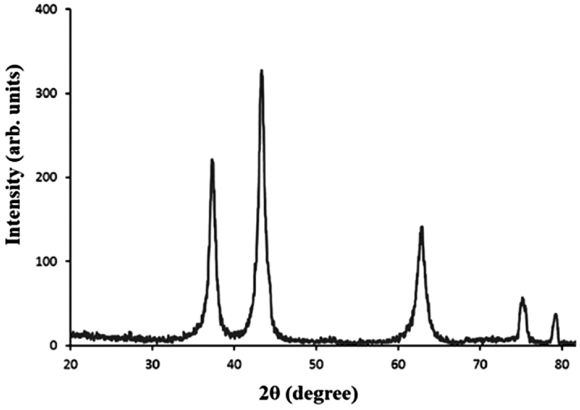


Fig. 5 — XRD pattern of SBL-Ni

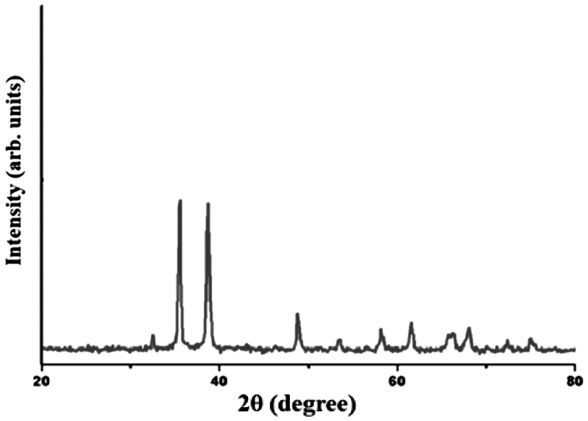


Fig. 6 — XRD pattern of SBL-Cu

density (OD) amounts were plotted against C_M/C_L, where C_M is the concentration of the metal ions (Cu and Ni), and C_L is the ligand concentration

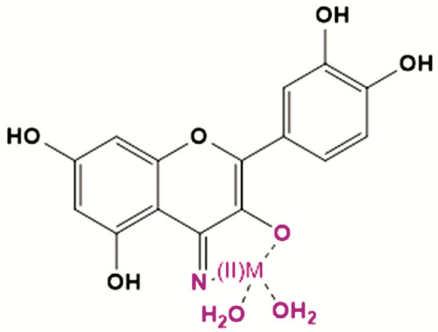


Fig. 7 — Proposed structure for the SBL-Cu and SBL-Ni (M=Cu, Ni)

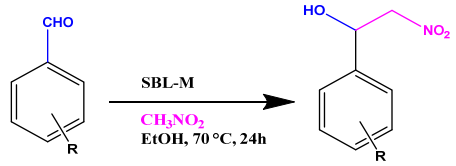
(SBL). The maximum absorbance was observed in C_M/C_L=1, which confirmed that the stoichiometric ratio for the SBL-Cu and SBL-Ni was 1:1 (Supplementary Data, Fig. S3). According to the analysis performed and Job's method, the structure of the complexes is shown in Fig. 7.

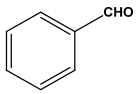
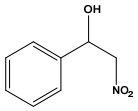
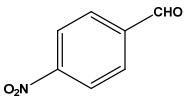
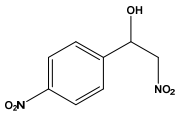
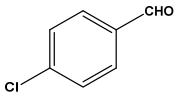
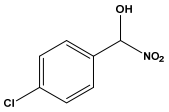
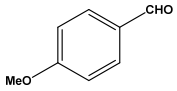
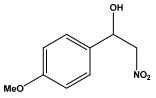
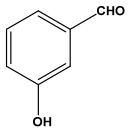
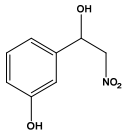
The catalytic activity of the SBL-Cu and SBL-Ni in the Henry reaction

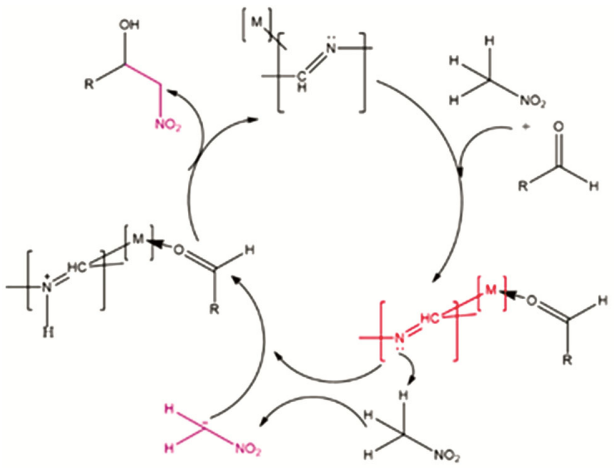
To find optimal conditions for the Henry reaction, different solvents, a various number of catalysts, and different temperatures were chosen. (Table 1) The best results were obtained by applying 1 mol% of catalysts, ethanol as a solvent, and 70 °C for 24 h. Both catalysts showed remarkable activity in Henry reaction, but SBL-Cu had a higher yield in this reaction. To investigate the effect of substitution group on the reactivity of different benzaldehydes with nitromethane under optimized conditions, sex substituted benzaldehydes were examined, as shown in Table 4. NMR spectra of the products were obtained by Henry reaction are shown in Fig. S4-S15 in Supplementary Data. All the studied benzaldehydes showed moderate to high yields in the Henry reaction in the presence of SBL-Cu or SBL-Ni as a catalyst. As expected, benzaldehydes containing electron-withdrawing groups gave higher yields than those including electron-donating groups.

The proposed reaction mechanism is given in Scheme 1. The nitrogen in the structure of the catalyst can be assisted in the deprotonation of the methylene group of nitromethane. On the other hand, benzaldehydes were activated by the metal center

Table 4 — Compression the reaction of different derivatives of benzaldehyde with nitromethane catalyzed by SBL-M (M=Cu, Ni)



Entry	Substrate	Product	Yield (%) by using SBL-Cu	Yield (%) by using SBL-Ni
1			83	70
2			86	75
3			85	73
4			40	32
5			75	65



Scheme 1 — Proposed catalytic cycle for the Henry reaction catalyzed by SBL-Cu and SBL-Ni

concerning its electrophilic attack. Finally, the C-C bond was formed by adding the nitronate to the carbonyl group of the aldehyde upon nucleophilic attack. Deprotonation of nitromethane and protonation of the coupled spices can be attributed to the azomethine group and ethanol as a solvent. Therefore, the good performance of the catalysts in the presence of ethanol is expected. The performance of SBL-Cu and SBL-Ni was compared with some other similar compounds. (Table 5) The synthesized catalysts are easy to make and cheap. Those are heterogeneous in nature and can be used several times. Also, the solvent applied was ethanol which is a green and environmentally preferable solvent.

Recyclability and heterogeneity tests

The possibility of recycling using SBL-Cu and SBL-Ni was tested for the reaction of benzaldehyde and nitromethane. To this purpose, the catalysts were recovered from the reaction mixture by centrifugation and were applied for the next run. After five reaction cycles, a significant reduction in the yield of the reaction in the presence of the catalyst was not observed (Fig. 8).

To investigate the catalytic performance of SBL-Cu and SBL-Ni, FT-IR analysis was performed after five times reaction. The FT-IR analysis showed no

Table 5 — Comparison of activities of other catalysts in Henry reaction using benzaldehyde and nitromethane

Catalyst	Aldehyde	Solvent/Temp./Time	Yield (%)	ref
$[Cu(L)(H_2O)_4]$(L=2-propionamideoterephthalate)	Benzaldehyde	H_2O/70 °C/30 h	71	33
[Cu(L)-(DMF)].DMF.H_2O(H_2L:5-{(pyridine-4-ylmethyl)amino}isophthalic acid	Benzaldehyde	H_2O/75 °C/40 h	84	34
$[Cu_3(pdtc)L_2(H_2O)_3]$.2DMF.10H_2O (HL:4-(2-(pyridine-4-yl)vinyl)benzoic acid)	Benzaldehyde	1,4-dioxane/70 °C/36 h	50	35
$[Cd_2(L\text{-}glu)_2(bpe)_3(H_2O)].2H_2O]$	Benzaldehyde	MeOH/R.T./30 h	89	36
$[Cd\text{-}(L)]_n$(H_2L:5-{(pyridine-4-ylmethyl)amino} isophtalic acid)	Benzaldehyde	H_2O/75 °C/40 h	69	34
This work (SBL-Cu)	Benzaldehyde	EtOH/70 °C/24 h	83	
This work (SBL-Ni)	Benzaldehyde	EtOH/70 °C/24 h	70	

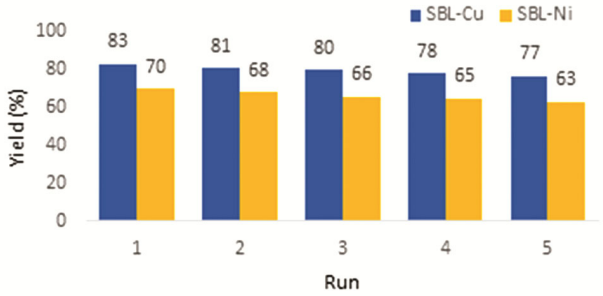


Fig. 8 — Catalyst recycling test for the Henry reaction

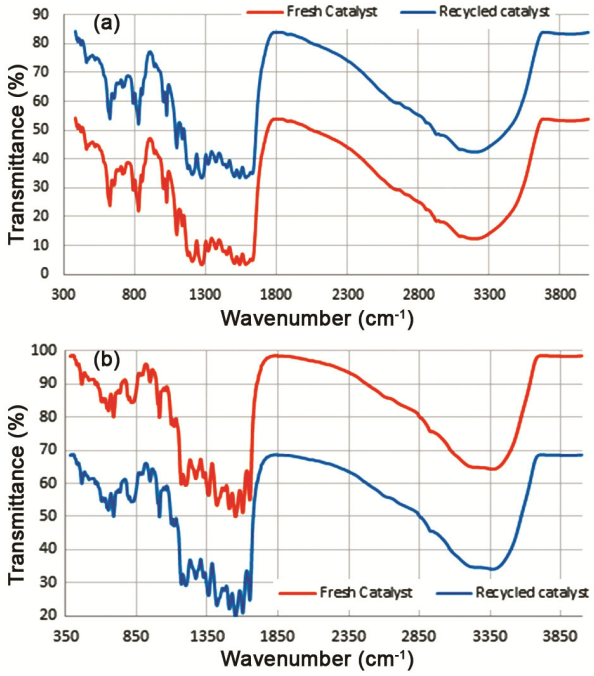


Fig. 9 — Comparison of FTIR spectrum of the (a): recycled SBL-Cu and (b): recycled SBL-Ni after five reaction runs with that of the fresh catalyst.

significant spectral differences between these spectra. (Fig. 9 (a), (b)). To confirm the heterogeneous nature of the catalysts, hot filtration test was carried out for the model reaction. During this test, after 12 h of the

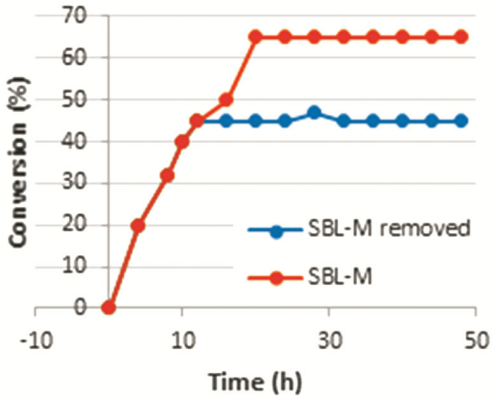


Fig. 10 — Hot leaching test for the Henry reaction based on SBL-M (M=Cu, Ni)

reaction, the catalyst was removed from the reaction by filtration. The filtrate was continued the previous reaction for 36 h. The results showed no conversion in the absence of the catalyst. This process was carried out for SBL-Cu and SBL-Ni. After the separation of the catalysts, there was no catalyst to perform the reaction, and the reaction has not continued (Fig. 10). This experiment confirmed that the synthesized complexes are heterogeneous.

Conclusion

In summary, we have synthesized highly efficient metal complexes using copper (II) and nickel (II) acetate. For the preparation of the complexes, we used a Schiff base as a ligand, which was previously synthesized by our group. We applied these complexes as a catalyst to improve Henry reaction. These catalysts are heterogeneous in nature and have significant activity in Henry reaction to produce β-nitroalkanols. SBL-Cu had shown better catalytic activity than SBL-Ni. These catalysts could be recycled and reused several times without significant loss of activity. The synthesized catalysts were effective in reacting different derivatives of

benzaldehyde with nitromethane to produce corresponding β-nitroalkanols.

Supplementary Data

Supplementary data associated with this article are available in the electronic form at http://nopr.niscair.res.in/jinfo/ijca/IJCA_61(02)YYYYYYYY_SupplData.pdf.

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