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FULL PAPER

Preparation and characterization of nano-Co-[4-chlorophenyl-salicylaldimine-methyl pyranopyrazole] Cl_2 as a new Schiff base complex and catalyst for the solvent-free synthesis of 1-amidoalkyl-2-naphthols

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Abstract

By the reaction of 4-chlorobenzaldehyde with ethyl acetoacetate, malononitrile, and hydrazine hydrate, 6-amino-4-(4-chlorophenyl)-3-methyl-2, 4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile was prepared and then reacted with salicylaldehyde and $CoCl_2{\cdot}6H_2O$ to produce nano-Co-[4-cholorophenyl-salicylaldimine-methylpyranopyrazole]Cl_2 (nano-[Co-4CSMP]Cl_2). The prepared nano-Schiff base complex was reported for the first time and fully characterized by Fourier transform-infrared spectroscopy, thermal gravimetric analysis, differential thermal gravimetric analysis, scanning electron microscopy, energy-dispersive X-ray spectroscopy, transmission electron microscopy, and Brunner–Emmett–Teller analyses and applied as an efficient catalyst for the synthesis of some 1-amidoalkyl-2-naphthol derivatives.

KEYWORDS

1-amidoalkyl-2-naphthol, nano-[co-4CSMP]Cl_2, pyranopyrazole, Schiff base

1 | INTRODUCTION

Azomethine compounds, namely, Schiff bases, are prepared by the reaction of primary amine with carbonyl compounds such as aldehydes or ketones. Schiff base compounds are widely introduced as one of the useful kinds of ligands due to their easy and convenient way for the preparation of materials and their interesting coordination chemistry.[1] Schiff base complexes were prepared by the coordination of Schiff base ligands with different metals. These compounds possess some important activities such as antibacterial, antifungal, anticancer, antioxidant, anti-inflammatory, antimalarial, and antiviral properties. Schiff base complexes are also used as efficient catalysts in organic transformations.[2–4] 1-Amidoalkyl-2-naphthol derivatives are beneficial compounds due to their ability to convert into some biological materials such as 1-aminoalkyl-2-naphthols[5–7] and 1,3-oxazine derivatives.[8] In particular, 1,3-oxazine derivatives exhibit some significant activities, including antibiotic,[9] antitumor,[10] analgesic,[11] anticonvulsant,[12] antipsychotic,[13] antimalarial,[14] antianginal,[15] antihypertensive,[16] and antirheumatic properties.[17] One-pot three-component condensation reactions of different phenols with aromatic aldehydes and amide derivatives were reported in the presence of various catalysts such as [Msim]Cl, [Dsim]Cl, [Msim]$AlCl_4$,[18,19] trityl chloride,[20,21] saccharin sulfonic acid,[22] nano SnO_2,[23] and [Msim]$FeCl_4$.[24] Because of the significance of these compounds in various applications and some limitations in previous approaches, the methods proposed herein will be useful are still required.

Recently, we have prepared a new category of Schiff base ligands, by the reaction of various pyranopyrazole derivatives with salicylaldehyde, and then mixed the

ligand with different metals to prepare Schiff base complexes. These complexes were studied and their catalytic activity was tested during the synthesis of various compounds such as pyrano[2,3-d] pyrimidinediones,[25] 4-[(2-hydroxynaphthalen-1-yl)(aryl)methyl]-5-methyl-2-phenyl-1H-pyrazol-3(2H)-ones,[26] pyranopyrazoles,[27] hexahydroquinolines,[28,29] and bis (pyrazolyl)methanes.[30]

In continuation of our previous investigations on the designing of new Schiff base complexes and their catalytic application in the synthesis of various organic compounds, we have now prepared nano-Co-[4-cholorophenyl-salicylaldimine-methylpyranopyrazole]Cl_2 (nano-[Co-4CSMP]Cl_2) as a new Schiff base complex and successfully tested it as a catalyst in the synthesis of 1-amidoalkyl-2-naphthol derivatives (Figure 1 and Scheme 1).

2 | EXPERIMENTAL

All chemicals were purchased from Merck or Fluka Chemical Companies. The known products were identified by comparison of their melting points (MPs) and spectral data with those reported in the literature. The ^{1}H-nuclear magnetic resonance (^{1}H-NMR; 500 MHz) and ^{13}C-NMR (125 MHz) spectra were recorded on a Bruker AVANCE DPX FT-NMR spectrometer (δ in ppm). Infrared (IR) spectrum of products was recorded by Perkin Elmer PE-1600-FTIR.

2.1 | Procedure for the synthesis of nano-co-[4-cholorophenyl-salicylaldimine-methylpyranopyrazole]Cl_2 (nano-[co-4CSMP]Cl_2)

A mixture of ethyl acetoacetate (0.13 g, 1 mmol), hydrazine hydrate (1.25 mmol), and isonicotinic acid (0.1 mmol, 0.0123 g, 10 mol%) was added into a 10-mL round-bottomed flask connected to a reflux condenser, and stirred at 85 °C for 5 min, and then 4-chlorobenzaldehyde (1 mmol) and malononitrile (0.066 g, 1 mmol) were added to the reaction mixture. Upon completion of the reaction, followed by thin-layer chromatography, the reaction mixture was cooled down to room temperature. Water was added to the reaction mixture that was then dissolved with isonicotinic acid. Finally, the aqueous layer was separated from the reaction mixture, and the solid residue (crude product) was triturated using a mixture of ethanol and water (9:1) to prepare 6-amino-4-(4-chlorophenyl)-3-methyl-2,4-dihydropyrano [2,3-c]pyrazole-5-carbonitrile as an amine.[31] The prepared amine (1 mmol) and $CoCl_2{\cdot}6H_2O$ (1 mmol) were added into a 25-mL round-bottomed flask containing salicylaldehyde (1.5 mmol), which was connected to a reflux condenser, and stirred at 100 °C for 72 hr. Finally, the reaction mixture was washed with ethyl acetate three times to purify nano-[Co-4CSMP]Cl_2 from excess salicylaldehyde (Scheme 2).

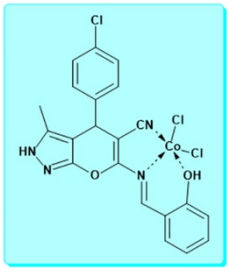


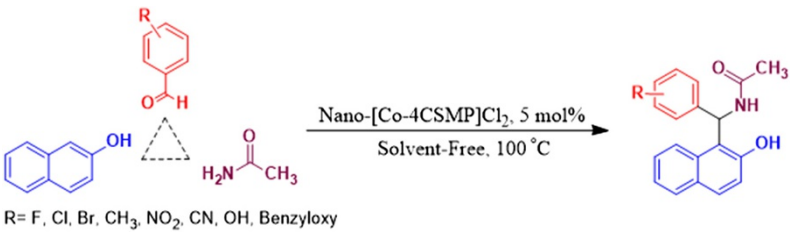
FIGURE 1 Structure of nano-[Co-4CSMP]Cl_2

2.2 | General procedure for the synthesis of 1-amidoalkyl-2-naphthols

A mixture of 2-naphthol (0.288 g, 2 mmol), acetamide (2.5 mmol), aromatic aldehyde (2 mmol), and nano-[Co-4CSMP]Cl_2 (5 mol %) was added into a 25 mL-round-bottomed flask connected to a reflux condenser and stirred at 100 °C for an appropriate period. After the completion of the reaction, as monitored by thin-layer chromatography, the reaction mixture was cooled down to room temperature and the product and remaining starting materials were extracted with warm ethanol (20 ml) to filter them from the catalyst (the product and remaining starting materials are soluble in warm ethanol; however, the catalyst is not). The product was purified by recrystallization of the reaction mixture in ethanol (90%).

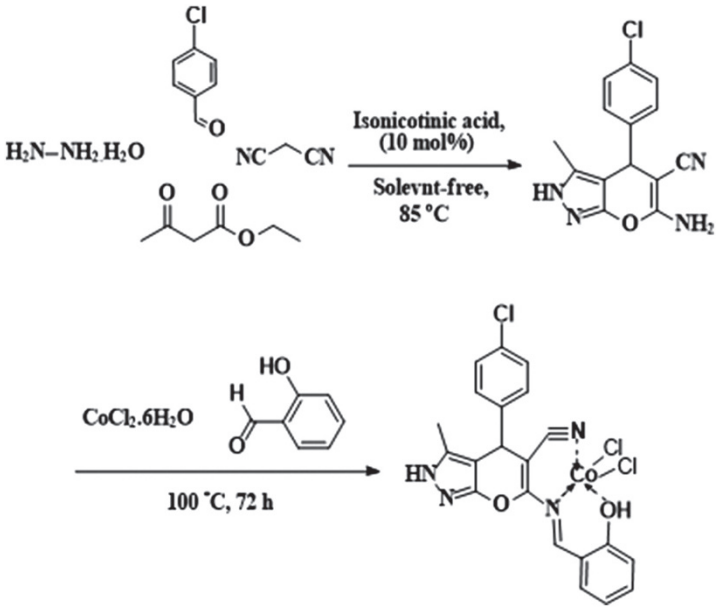
2.2.1 | *N*-([4-(benzyloxy)phenyl][2-hydroxynaphthalen-1-yl]methyl) acetamide

White solid; MP: (226–230 °C); IR (KBr cm^{-1}): 3424, 3064, 3032, 2905, 2863, 1627, 1509, 1437, 1378, 1269, 1247, 1090, 1062, 1024, 816, 742, 696, 559; ^{1}H-NMR [500 MHz,



SCHEME 1 Preparation of 1-amidoalkyl-2-naphthols

SCHEME 2 Preparation of nano-[Co-4CSMP]Cl_2



dimethyl sulfoxide (DMSO)-d_6]: 1.95 (s, 3H, CH_3), 5.02 (s, 2H), 6.88 (d, 2H, J = 8.85 Hz) 7.04–7.07 (m, 3H), 7.19–7.41 (m, 8H), 7.74 (d, 1H, J = 8.85 Hz), 7.79 (d, 1H, J = 7.55 Hz), 7.84 (s, 1H), 8.40 (d, 1H, J = 8.45 Hz), 9.95 (s, 1H); ^{13}C-NMR (125 MHz, DMSO-d_6): 22.7, 47.4, 69.1, 114.3, 118.5, 118.9, 122.3, 126.2, 127.2, 127.6, 127.7, 128.4, 128.5, 129.1, 132.3, 134.6, 137.1, 153.0, 156.7, 169.09.

2.2.2 | *N*-[(4-bromophenyl) (2-hydroxynaphthalen-1-yl)methyl] acetamide

White solid; MP: (220–225 °C**)**; IR (KBr cm^{-1}): 3393, 3055, 2962, 2874, 2788, 2703, 2615, 1636, 1620, 1580, 1522, 1488, 1361, 1331, 1245, 1180, 1064, 1072, 1011, 934, 864, 820, 747, 680, 585, 512; ^{1}H-NMR (500 MHz, DMSO-d_6): 1.99 (s, 3H, CH_3), 7.07–7.09 (m, 3H), 7.11–7.29 (m, 2H), 7.36–7.39 (m, 1H), 7.44 (d, 2H, J = 8.55 Hz), 7.76–7.81 (m, 3H), 8.46 (d, 1H, J = 8.2 HZ), 10.04 (s, 1H); ^{13}C-NMR (125 MHz, DMSO-d_6): 22.7, 47.6, 118.4, 118.5, 119.2, 122.5, 126.5, 128.4, 128.5, 128.6, 128.7, 129.5, 130.9, 131.2, 132.3, 142.3, 153.3, 169.5.

2.2.3 | *N*-[(2,4-dichlorophenyl) (2-hydroxynaphthalen-1-yl)methyl] acetamide

White solid; MP: (200–202 °C); IR (**KBr cm^{-1}**): 3406, 3143, 3067, 1650, 1632, 1582, 1517, 1471, 1439, 1370, 1322, 1298, 1267, 1164, 1147, 1089, 1063, 986, 942, 870, 816, 750, 750, 583, 458; ^{1}H-NMR (500 MHz, DMSO-d_6): 1.93 (s, 3H, CH_3), 7.00 (d, 1H, J = 7.85 Hz), 7.11 (d, 1H, J = 8.85 HZ), 7.27 (t, 1H, J = 7.75 Hz), 7.39–7.45 (m, 2H), 7.46 (d, 1H, J = 2.2 Hz), 7.59 (d, 1H, J = 8.5 Hz), 7.74 (d, 1H, J = 8.85 Hz), 7.79 (d, 1H, J = 8.10 Hz), 7.96 (d, 1H, J = 8.70 HZ), 8.62 (s, 1H), 9.82 (s, 1H); ^{13}C-NMR (125 MHz, DMSO-d_6): 22.4, 47.4, 116.4, 118.7, 122.4, 122.6, 126.4, 126.5, 128.3, 128.5, 128.7, 129.7, 131.3, 131.7, 132.8, 132.9, 139.4, 153.8, 168.9.

2.2.4 | *N*-[(2-hydroxynaphthalen-1-yl) (4-nitrophenyl)methyl]acetamide

White solid; MP: (250–254 °C), IR (**KBr cm^{-1}**): 3392, 3264, 3052, 2958, 1626, 1641, 1523, 1439, 1281, 853, 825, 581, 512; ^{1}H-NMR (500 MHz, DMSO-d_6): 2.02 (S, 3H, CH_3), 7.17–7.29 (m, 3H), 7.40 (d, 3H, J = 8.30 Hz), 7.81 (t, 3H, J = 8.75 Hz), 8.13 (d, 2H, J = 8.90 Hz), 8.57 (d, 1H, J = 7.90 Hz), 10.11 (s, 1H); ^{13}C-NMR (125 MHz, DMSO-d_6): 22.6, 48.0, 117.9, 118.5, 122.6, 123, 123.3, 123.5, 126.8, 127.2, 127.8, 128.5, 128.7, 129.9, 132.3, 145.9, 151.3, 153.4, 169.9.

2.2.5 | *N*-[(2-hydroxynaphthalen-1-yl) (3-nitrophenyl)methyl]acetamide

White solid; MP: (245–250 °C); IR (**KBr cm^{-1}**): 3375, 3226, 3087, 3063, 1648, 1630, 1579, 1525, 1530, 1350, 1209, 1094, 990, 806, 734, 584; ^{1}H-NMR (500 MHz, DMSO-d_6): 2.01 (s, 3H, CH_3), 7.17 (d, 1H, J = 7.9 Hz), 7.22 (d, 1H, J = 8.8 Hz), 7.29 (t, 1H, J = 7.5 Hz), 7.39–7.42 (m, 1H), 7.54 (m, 2H), 7.80–7.84 (m, 3H), 8.00 (s, 1H), 8.05 (d, 1H, J = 7.25 Hz), 8.64 (d, 1H, J = 8.00 Hz), 10.16 (S, 1H, OH); ^{13}C-NMR (125 MHz, DMSO-d_6): 22.4, 47.5, 117.7, 118.3, 120.3, 121.1, 122.5, 122.7, 126.7, 128.3, 128.6, 129.5, 129.8, 132.08, 132.7, 145.3, 147.6, 153.3, 169.6.

2.2.6 | *N*-[(4-chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl] acetamide

White solid; MP: (230–232 °C); IR (**KBr cm^{-1}**): 3393, 3268, 3055, 2962, 2870, 2789, 2703, 2616, 1638, 1625, 1581, 1515, 1492, 1439, 1332, 1374, 1362, 1332, 1279, 1245, 1172, 1092, 1064, 1014, 934, 848, 819, 748, 500; ^{1}H-NMR (500 MHz, DMSO-d_6): 1.98 (s, 3H, CH_3), 7.09 (d, 1H, J = 8.20 Hz), 7.15 (d, 2H, J = 8.45 Hz), 7.22 (d, 1H, J = 8.85 Hz), 7.25–7.39 (m, 4H), 7.76–7.81 (m, 3H), 8.46 (d, 1H, J = 8.25 Hz), 10.03 (s, 1H, OH); ^{13}C-NMR (125 MHz, DMSO-d_6): 22.6, 47.5, 118.4, 118.5, 122.5, 126.5, 127.9, 128.2, 128.3, 128.5, 128.6, 129.5, 130.7, 132.3, 141.8, 153.2, 169.5.

3 | RESULTS AND DISCUSSION

For the synthesis of nano-[Co-4CSMP]Cl_2, by the reaction of 4-chlorobenzaldehyde with ethyl acetoacetate, malononitrile, and hydrazine hydrate, 6-amino-4-(4-chlorophenyl)-3-methyl-2,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile was prepared according to a previous study.[31] The prepared pyranopyrazole was then reacted with salicylaldehyde and $CoCl_2{\cdot}6H_2O$ to produce nano-Co-[4-cholorophenyl-salicylaldimine-methylpyranopyrazole]Cl_2 (nano-[Co-4CSMP]Cl_2) as a nano-Schiff base complex (Scheme 2). The structure of nano-[Co-4CSMP]Cl_2 was studied by IR, scanning electron microscopy, energy-dispersive X-ray spectroscopy (EDX), transmission electron microscopy (TEM), Brunner-Emmett-Teller (BET), thermogravimetric analysis, and derivative thermogravimetry analyses.

First, 4CSMP as a Schiff base was synthesized alone and its structure was studied by ^{1}H-NMR and ^{13}C-NMR spectra (Schemes **S1** and **S2**). After the preparation of nano-[Co-4CSMP]Cl_2 by the reaction of 4CSMP with $CoCl_2{\cdot}6H_2O$, EDX images from nano-[Co-4CSMP]Cl_2 were studied, which further confirmed the successful preparation of the catalyst. According to EDX analysis,

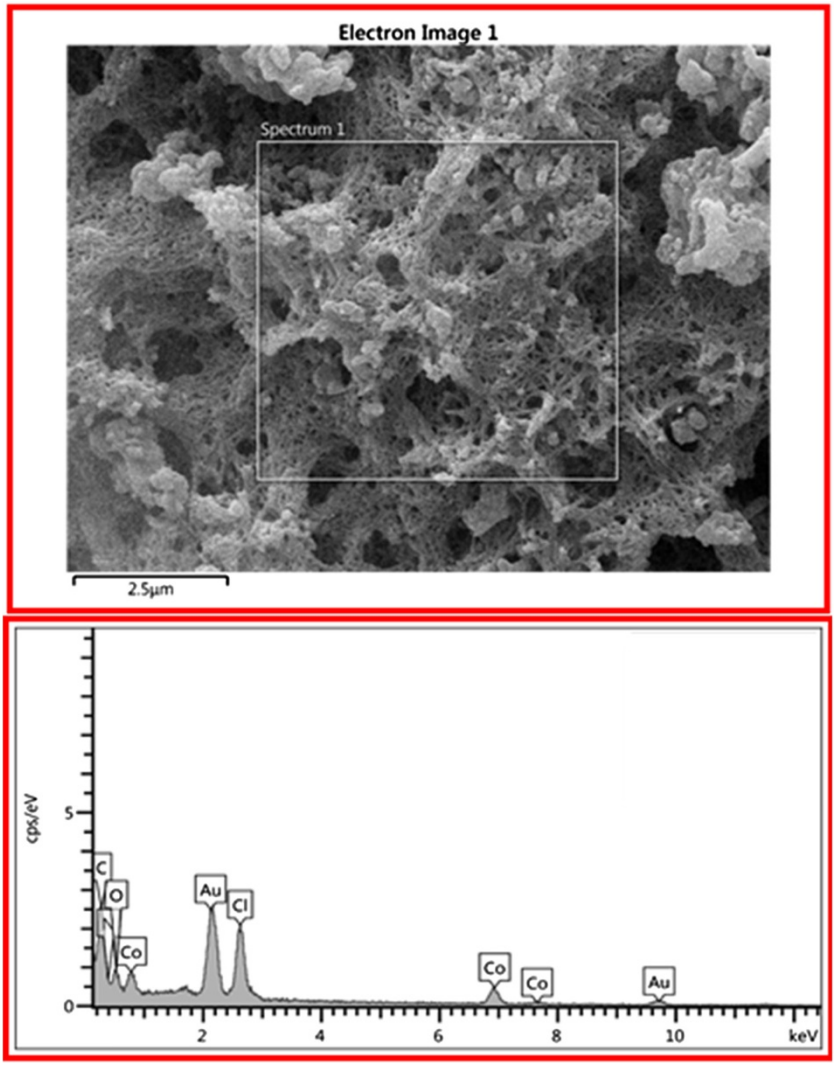


FIGURE 2 Energy-dispersive X-ray spectroscopy of nano-[Co-4CSMP]Cl_2

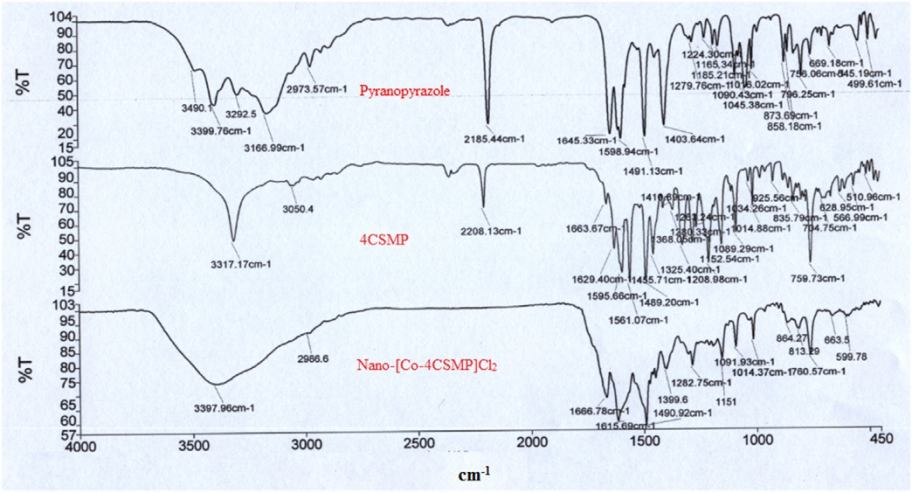


FIGURE 3 Infrared spectrum of nano-[Co-4CSMP]Cl_2

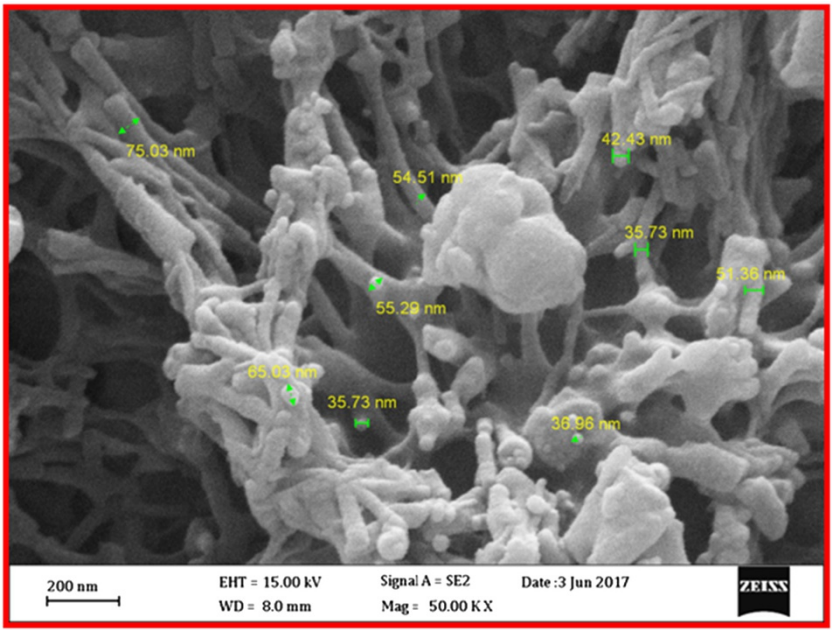


FIGURE 4 Scanning electron microscopy image of nano-[Co-4CSMP]Cl_2

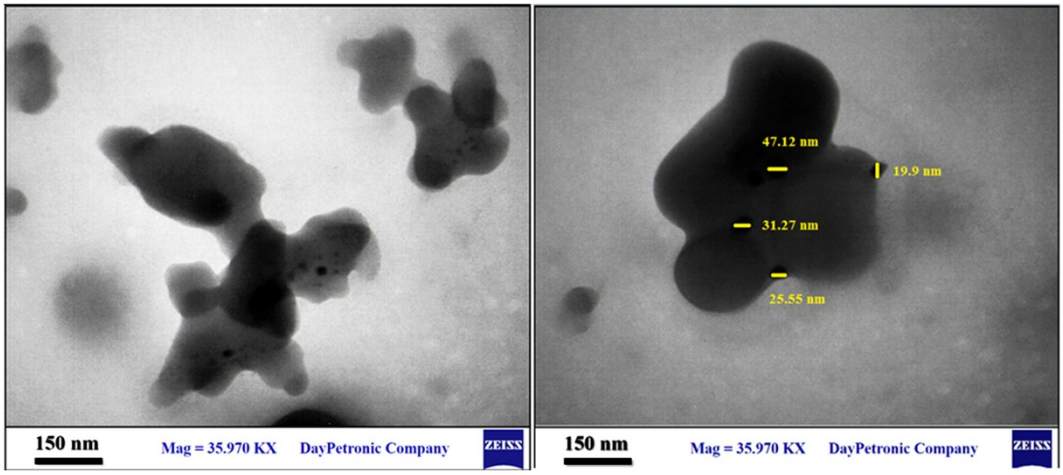


FIGURE 5 Transmission electron micrographs of nano-[Co-4CSMP]Cl_2

TABLE 1 Specific surface area (S_{BET}), diameter pore, and total pore volume of nano-[Co-4CSMP]Cl_2

Sample	Brunner–Emmett–Teller (BET) surface area ($m^2\ g^{-1}$)	Diameter (nm)	Pore volume ($cm^3\ g^{-1}$)
Nano-[Co-4CSMP]Cl_2	16.042	1.88	0.07

the desired elements, namely, carbon, oxygen, nitrogen, chlorine, and cobalt, were found in the structure of the catalyst (Figure 2).

The IR spectrum of nano-[Co-4CSMP]Cl_2 showed a broad peak at 2900–3600 cm^{-1}, which could be attributed to the O–H stretching of the OH group and the peak which appeared at 1666 cm^{-1} to the stretching mode of C=N bond of azomethine (Figure 3). To demonstrate the steps involved in the synthesis of nano-[Co-4CSMP]Cl_2 as a Schiff base complex, the IR spectra of 6-amino-4-(4-chlorophenyl)-3-methyl-2,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile as an amine and 4CSMP as an imine were compared with nano-[Co-4CSMP]Cl_2 (Figure 3). When cobalt coordinates with imine, the peak at 2208 cm^{-1} related to the CN bond is omitted, suggesting that the cobalt has interacted with the CN group and changed the triple bond in CN (Figure 3). An analysis of scanning electron microscopy images of nano-[Co-4CSMP]Cl_2 also confirmed the formation of nano-sized catalyst particles (Figure 4).

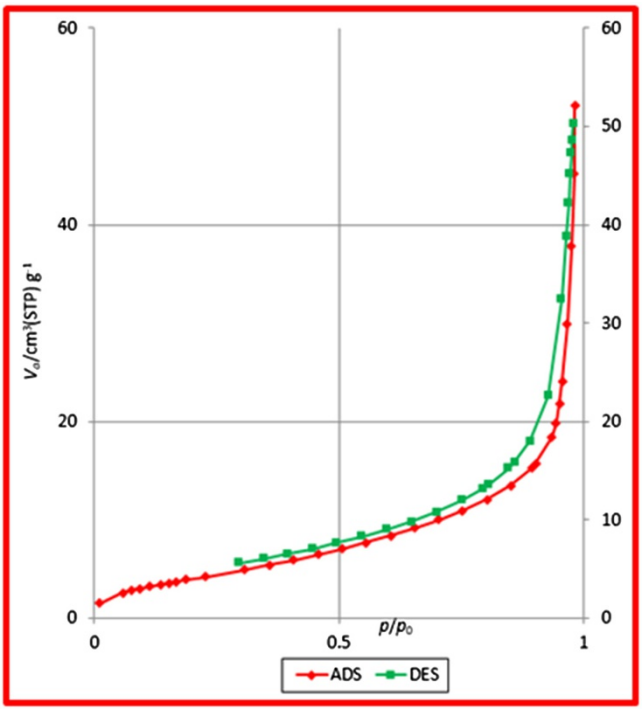


FIGURE 6 Nitrogen adsorption (ADS)–desorption (DES) isotherms of nano-[Co-4CSMP]Cl_2

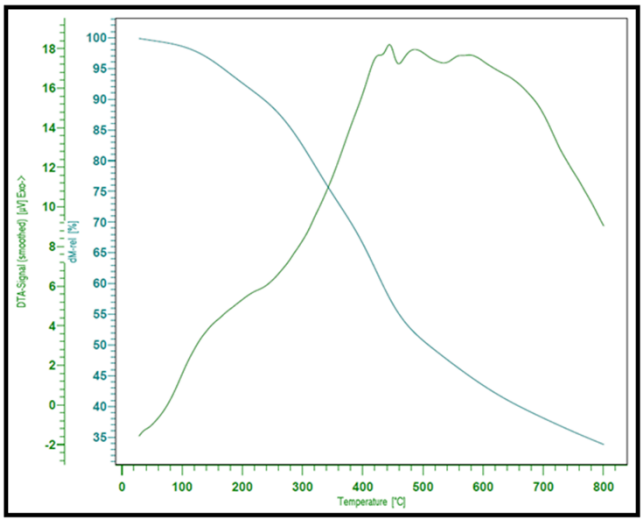


FIGURE 7 Thermal gravimetric analysis and differential thermal analysis (DTA) of nano-[Co-4CSMP]Cl_2

The size of nano-[Co-4CSMP]Cl_2 was also studied by TEM analysis. TEM measurements further confirmed that the particles prepared were nano sized (Figure 5). Because the catalyst prepared has a porous structure, the volume and size of the cavities contained in it were investigated. The BET surface area, pore size, and pore volume of nano-[Co-4CSMP]Cl_2 are 16.042 $m^2\ g^{-1}$, 1.88 nm, and 0.07 $cm^3\ g^{-1}$, respectively (Table 1 and Figure 6).

Thermogravimetric analysis and differential thermal analysis of nano-[Co-4CSMP]Cl_2 were also performed, with the corresponding results presented in Figure 7. The nano-[Co-4CSMP]Cl_2 decomposed after 200 °C, which is clearly seen in the figure.

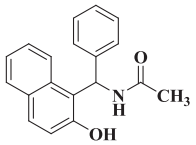
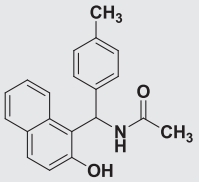
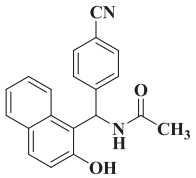
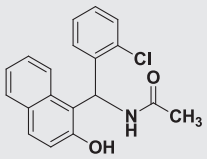
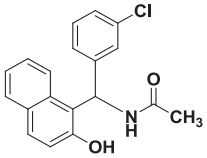
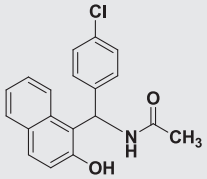
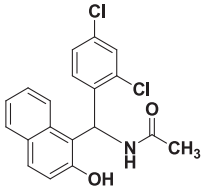
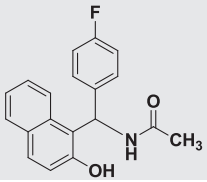
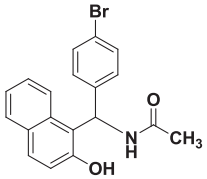
To optimize the reaction condition, the reaction of 2-naphthol (2 mmol) with 4-chlorobenzaldehyde (2 mmol) and acetamide (2.4 mmol) was selected as the model

TABLE 2 Effect of nano-[Co-CSMP]Cl_2 amounts, temperature, and solvent on the reaction between β-naphthol with 4-chlorobenzaldehyde and acetamide

Mol% of catalyst	Solvent	Temp. (°C)	Time (min)	Yield[a] (%)
—	—	100	120	—
3	—	100	5	64
5	—	100	5	90
8	—	100	5	90
5	—	50	40	45
5	—	80	20	80
5	—	120	5	74
5	H_2O	Reflux	25	80
5	Ethanol	Reflux	20	85

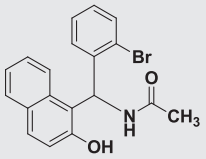
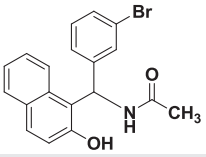
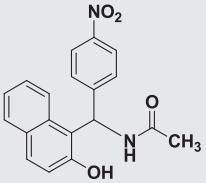
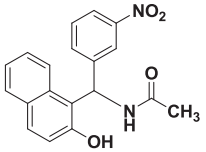
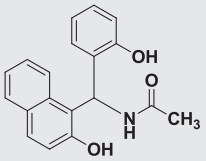
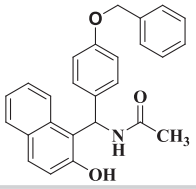
[a]Isolated yield.

TABLE 3 Synthesis of 1-amidoalkyl-2-naphthols using nano-[Co-4CSMP]Cl_2

Entry	Product	Time (min)	Yield[a] (%)	Melting point (°C; literature value)
1		15	90	246–248 (238–240)[18]
2		5	95	225–227 (223–225)[18]
3		5	90	252–253 (260–262)[32]
4		10	90	195–199 (197–199)[18]
5		5	85	237–239 (237–238)[33]
6		5	90	225–227 (220–222)[18]
7		10	95	200–202 (206–208)[32]
8		5	85	230–232 (229–229.5)[34]
9		10	90	222–225 (226–228)[18]

(Continues)

TABLE 3 (Continued)

Entry	Product	Time (min)	Yield[a] (%)	Melting point (°C; literature value)
10		15	85	200–204 (190–191)[33]
11		10	90	245–246 (250–252)[35]
12		5	95	235–237 (246–248)[18]
13		5	80	245–250 (238–240)[18]
14		10	90	246–250 (245–246)[34]
15		5	90	230–235

[a]Isolated yield.

reaction and tested in the presence of different amounts of catalyst, at 50–100 °C under solvent-free condition (Table 2). As shown in Table 2, the best result was obtained using 5 mol% of catalyst at 100 °C. Furthermore, to show the superiority of the solvent-free condition, the model reaction was tested in the presence of various solvents such as H_2O and ethanol in which the yield of product was lower than that in the solvent-free condition. Moreover, the model reaction was tested in the absence of catalyst, in which no progress in reaction was noted (Table 2).

In the next step, the reaction of 2-naphthol with various aryl aldehydes and acetamide was examined in the presence of nano-[Co-4CSMP]Cl_2 at 100 °C and in the absence of solvent to estimate the domain and the generality of the catalysts. Aryl aldehydes containing electron-releasing substituents, electron-withdrawing substituents, and halogens on their aromatic rings reacted successfully in the reaction, and afforded the expected products in acceptable yields and reaction times (Table 3).

According to the proposed mechanism of reaction and based on the previous literature,[32–44] first, aromatic aldehyde is activated by the catalyst and reacted with 2-naphthol to afford **I**. In the next step, orthoquinone methide (**II**) is prepared by the elimination of one molecule of water from **I**. Finally, by Michael addition of acetamide, which is activated with the catalyst, **II** affords the expected 1-amidoalkyl-2-naphthol (Scheme 3).

To compare the applicability and the efficiency of nano-[Co-4CSMP]Cl_2 with previously reported catalysts for the synthesis of 1-amidoalkyl-2-naphthols, the results of using these catalysts in the reaction of 2-naphthol with 4-chlorobenzaldehyde and acetamide are summarized in

SCHEME 3 Proposed mechanism for the preparation of 1-amidoalkyl-2-naphthols

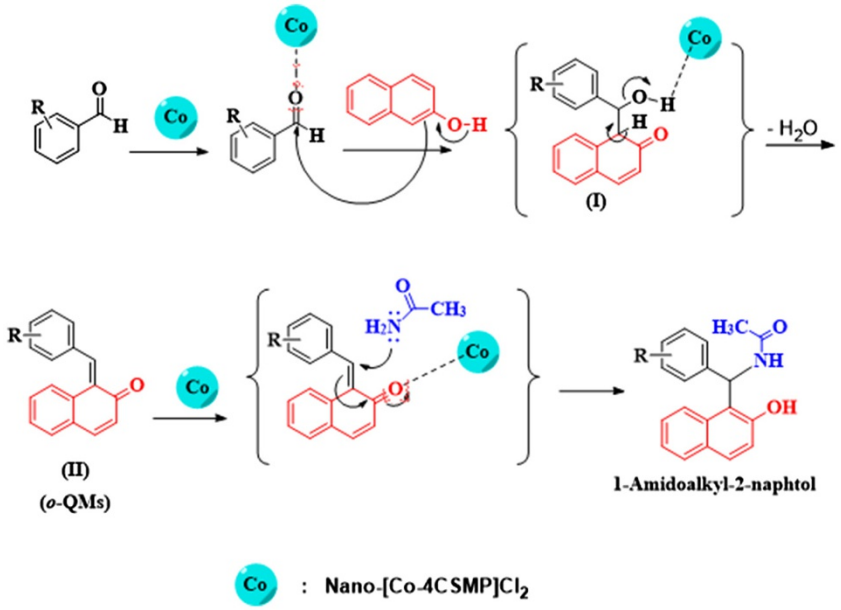


TABLE 4 Comparison of the results of the reaction of 2-naphthol with 4-chlorobenzaldehyde and acetamide catalyzed by nano-[Co-4CSMP]Cl_2 with some previously reported catalysts

Catalyst and temperature	Catalyst amount (mol%)	Time (min)	Yield (%)	Turnover frequency[a] (min^{-1})	Ref.
Nano-[Co-4CSMP]Cl_2, 100 °C	5	5	90	3.6	This work
Cyanuric chloride, 100 °C	10	10	90	0.9	[36]
$H_3PW_{12}O_{40}$, 100 °C	2	80	88	0.55	[37]
N-(4-sulfonic acid)butyl triethylammonium hydrogen sulfate, 120 °C	5	10	85	1.7	[38]
$Cu_{1.5}PW_{12}O_{40}$, 100 °C	2	90	78	0.433	[39]
H_3BO_3, 120 °C	80.8	15	72	0.059	[40]
ZnO nanoparticles, 120 °C	20	30	87	0.145	[41]
Phthalimide-*N*-sulfonic acid, 100 °C	10	6	90	1.500	[42]
Citric acid, 120 °C	10	18	91	0.505	[43]
$CoCl_2{\cdot}6H_2O$, 100 °C	5	20	61	0.630	This work

[a]Turnover frequency.

Table 4. The presented catalyst remarkably improved the synthesis of 1-amidoalkyl-2-naphthols in terms of reaction time, yield, and turnover frequency. This reaction was also performed in the presence of $CoCl_2{\cdot}6H_2O$ alone; however, compared with the reaction performed over nano-[Co-4CSMP]Cl_2 at 100 °C under solvent-free conditions, there was no improvement in results and the reaction overall was weaker (Table 4).

4 | CONCLUSIONS

We have introduced nano-Co-[4-cholorophenyl-salicylaldimine-methylpyranopyrazole]Cl_2 (nano-[Co-4CSMP]Cl_2) as a Schiff base complex and efficient catalyst for the synthesis of 1-amidoalkyl-2-naphthols by the condensation reaction of 2-naphthol with various aryl aldehydes and acetamide at 100 °C under solvent-free conditions.

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SUPPORTING INFORMATION

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