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A novel Ni-Zn-NPs as a catalyst for the preparation of tetrahydrobenzo[*b*]pyran

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Abstract In this report, a new precursor binuclear complex of Zn(II) and Ni(II) ions under formula [Zn(phen)(L)(OH2)(μ-SCN)Ni(phen)(L)(SCN)]**(1)** that phen=1-10-phenanthroline, L=2-aminobenzoate has been synthesized and then the facile synthesis of Ni-Zn-NPs has been reported under thermal decomposition. The complex **(1)** was characterized using cyclic voltammetry (CV), elemental analysis, and spectroscopic methods (IR, UV-Vis). FT-IR spectrum of complex **(1)** show vibrational frequency of NH group of anthranilic acid ligand appeared in the 3421 cm^{-1}. UV–visible spectrum of the compound **(1)** show d-d transitions of Ni(II) ion. Characterization of the nano-particle was performed with scanning electron microscopy (SEM), FT-IR spectroscopy, and X-ray powder diffraction (XRD). The X-ray diffraction pattern showed a pure system $Ni_{0.9}Zn_{0.1}O$ with a monoclinic structure. The average particle size was 27 nm using the Debye- Scherrer equation. Finally, the nano-particle has been used as a catalyst for the preparation of tetrahydrobenzo[b]pyran. The best datas were achieved when the reaction was carried out at 60 °C in the presence of $Ni_{0.9}Zn_{0.1}O$ (5 mol %) in 2:1 EtOH/H_2O.

Keywords: binuclear complex; Cyclic voltammetry; scanning electron microscopy

1. Introduction

Multi nuclear transition metal compounds with variety of ligands have become an important subject due to their potential activities. In recent years, researchers have synthesized polynuclear compounds with iron, nickel, cobalt, copper and zinc ions [1-7]. These compounds are also of great interest due to their ability to modify chemical, catalytic and photochemical properties as well as facile electrochemical processes[7-13]. 4*H*-pyrans belong to an important class of heterocyclic compounds having important biological activities [14]. They are reported to have diverse pharmacological activities such as anti-coagulant, anti-cancer, spasmolytic, diuretic, anti-ancaphylactia [15], Alzheimer's disease, AIDs associated dementia and myoclonus [16]. They are also reported to serve as an important regulator for potassium cation channel and photochemical activities [17-18]. Because of the intense interest in the biological activity of these compounds, several improved procedures for the synthesis of pyrans were reported. One of the most important methods for the synthesis of these compounds is one-pot condensation of an aldehyde, dimedone and malononitrile., Various catalysts such as Aspirin [19], $CeMgxZr_1$-xO_2 [20], HTMAB [21], TEBA [22], basic quaternary ammonium salt [23], $RE(PFO)_3$ [24], and tartaric acid [25] have been used for this reaction. We decided to prepare tetrahydrobenzo[b]pyran with a new nano catalyst. The results are represented in the following section.

2.Experimental

2.1. Materials and physical measurements

The materials applied in this study were of high purity (99%). IR spectra and Melting points of all samples were determined by FT-IR-JASCO-460 plus spectrometer and Electro thermal 9100 apparatus The UV–Vis spectra were taken on a JASCO 7850 spectrophotometer. The ^{13}C and ^{1}H-NMR spectra of samples were obtained on a Bruker DRX-400 and 300 Avance instrument in DMSO solution at 300 MHz. Chemicals were prepared from Fluka (Buchs, Switzerland) and chemical producer Merck. The redox activities of the complex were investigated using the technique of cyclic voltammetric. The Cyclic voltammogram was performed with a SAMA 500 Electro Analyzer. X-ray powder diffraction (XRD) measurements were recorded by an X'pert diffractometer of Philips Company with graphite monochromatic Cu Kα(λ= 0.15418 nm) radiation in the 2θ range of 10° to 60°.

3.Synthesis of binuclear complex (1)

3.1. Preparation of [Zn(phen)(L)(OH2)(μ-SCN)Ni(phen)(L)(SCN)] (1)

For the synthesis of this complex, the cationic and anionic parts were prepared separately.

3.1.1. Prepare cationic part

To obtain cationic complex, aqueous solution of 2-aminobenzoic acid (1mmol, 0.137gr) neutralized with NaOH is added to methanol solution of 1-10 phenanthroline (1mmol, 0.180gr). Then this mixture is added drop by drop to aqueous solution (20mL) of $Zn(NO_3)_2{\cdot}6H_2O$ (1mmol, 0.297gr). The resulting mixture was refluxed for 1 hours at a temperature of 90-100°C.

3.1.2. Prepare anionic part.

To obtain anionic complex, a mixture of 2-aminobenzoic acid (1mmol, 0.137gr) neutralized with NaOH, 1-10 phenanthroline (1mmol, 0.180gr), and Sodium thiocynate (2mmol, 0.162gr) was added to the aqueous solution of $Ni(NO_3)_2{\cdot}6H_2O$ (1mmol, 0.182gr). The resulting mixture was refluxed for 2 hours at a temperature of 90-100°C. Then cationic and anionic parts added together and have refluxed for 5 hours and the final product **(1)** was obtained. Crystals

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of complex **(1)**were obtained by slow evaporation of solvents and kept for further characterization. Yield 69%. Anal. Calcd. for: C, 53.93; H, 3.39; N, 12.58. Found: C, 54.02; H, 3.45; N, 13.05%. FT-IR(KBr, cm-1)); FT-IR (KBr, cm^{-1}): 3421ν (N-H), 2884-3007 cm^{-1} ν (C-H), 1514 ν (C=C).

3.2. Characterization of complex (1)

3.2.1. FT-IR spectrum complex (1)

FT-IR spectrum of this compound is taken in KBr tablet method and is depicted in **Fig.1.** The peak of the NH group of anthranilic acid ligand appeared in the 3421 cm^{-1}. The tensile vibrations of the C-H ring in the regions of 2884-3007 cm^{-1} [26]. The tensile vibrations of the bond are C = N, while the NCS is a bridge between the two metals in the complex and in 2084 cm^{-1} it has a sharp peak. The C = C bond also peaked in the 1514 cm^{-1} region[27-29].

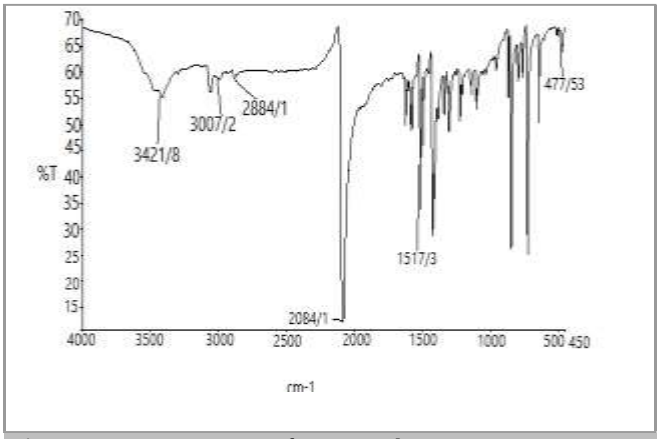


Figure 1. FT-IR spectrum of compound1 as KBr

3.2.2. Study of the electronic spectrum

UV–visible spectra of the compound **(1)**, obtained in DMF solution in the wavelength range 200-800 nm at 298 K. **(Fig. 2)**. In the ultraviolet region, the metal complex **(1)** exhibits intense absorption bands at 225-310 nm, that can be dependent to the n→ π * and π→ π * transitions of 2-aminobenzoate, 1-10 phenanthroline, and thiocynate ligands. The bands in Visible region at 540 nm and 760 nm can be relevant to $^3A_{2g}\rightarrow{}^3T_{1g(F)}$ and $^3A_{2g}\rightarrow{}^3T_{2g(F)}$ d-d transitions of Ni(II) respectively [30].

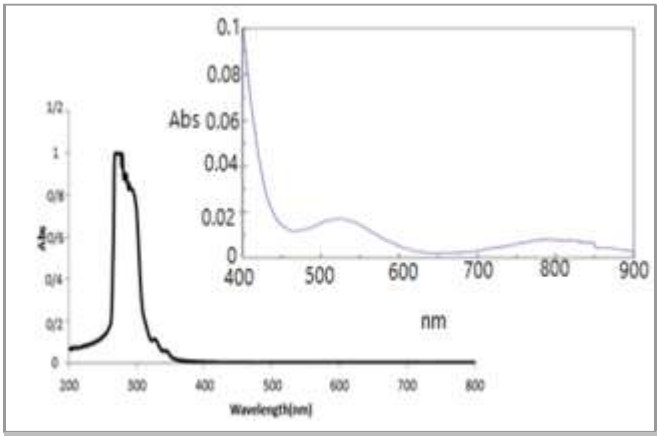


Figure 2. UV–visible absorption spectra in DMF solution

3.2.3. Electrochemical Studies

Fig. 3 shows cyclic voltammogram of **(1)** that were performed in DMF solution in the presence of TBAH as supporting electrolyte [31]. The reduction windows of 2-aminobenzoate and 1,10-phenanthroline ligands are observed in the -0/6V to -1/8V regions. A further irreversible step can be assumed at approximately 1.3 V for reduction of Ni(II)→Ni(I). For the Zn^{+2} in this domain is electrochemically inactive [32,33].

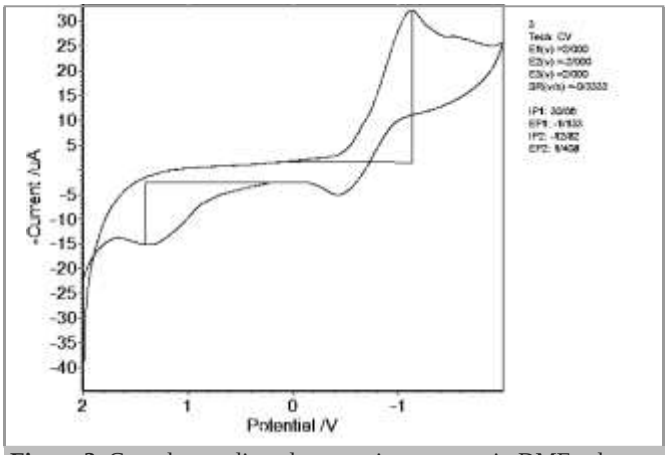


Figure 3. Complex cyclic voltammetric spectrum in DMF solvent

3.3. Synthesis of the Ni-Zn-NPs

Ni-Zn-NPs were prepared from title compound **(1)** using the physical method. 1.0 g of compound **(1)** calcinated at 600°C for 4 h in the furnace to formed Ni-Zn-NPs. The final product ($Ni_{0.9}Zn_{0.1}O$) was collected and washed with distilled water and absolute ethanol several times, dried in air and kept for further characterization**.**

3.3.1. FT-IR spectrum $Ni_{0.9}Zn_{0.1}O$ nanoparticle

The FT-IR spectrum from the complex after calcination at 600 ℃ is shown in **Fig.4** In this spectrum, there is a broad peak in the 3400 cm^{-1} area, which is related to the tensile vibrations of ν(O-H) water. Peak of Zn-O and Ni-O that are overlapping is seen in the 462 cm^{-1} area [29].

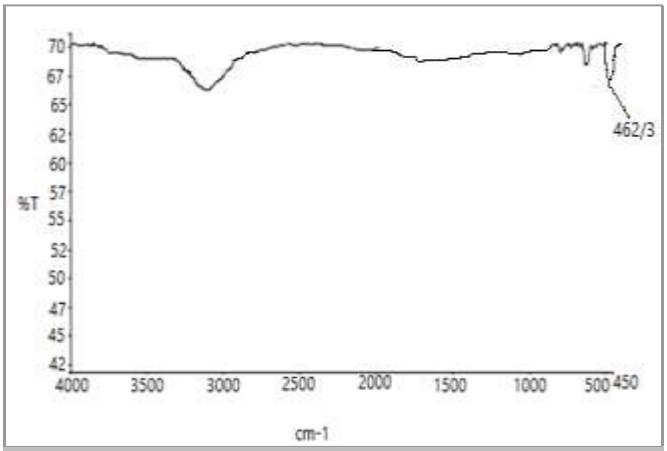


Figure 4. The FT-IR spectrum of $Ni_{0.9}Zn_{0.1}O$ after calcination

3.3.2.X-ray powder diffraction investigation

Fig. 5 shows the XRD pattern of the $Ni_{0.9}Zn_{0.1}O$ nanoparticle. The diffraction peaks can be indexed to pure nanoparticle with cubic phase with space group *Fm-3m* a. The crystallite size of the title nanoparticle, was calculated using the Debye-Scherre formula from the major diffraction peak of the corresponding particle. Specifically, the position and relative intensities of all peaks confirm well with standard specification 0270-075-01. The highest amount of absorption is in 100, which is related to 2θ with the value of 43/16 and as miller indices (200). The average particle size was 27 nm using the Debye- Scherrer equation [34].

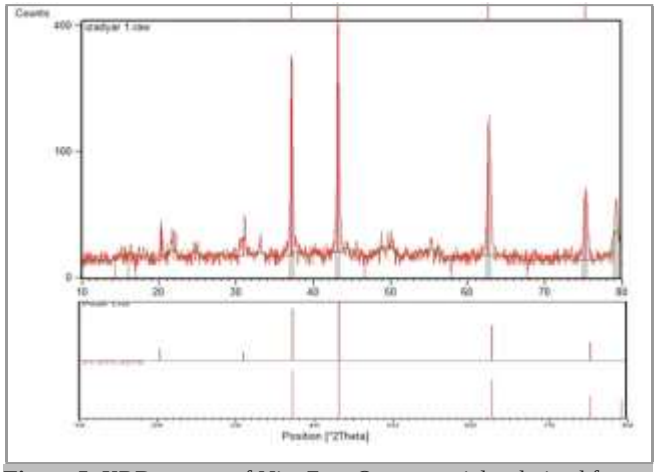


Figure 5. XRD spectra of $Ni_{0.9}Zn_{0.1}O$ nanoparticles derived from the complex **(1)**

3.3.3. SEM Photograph of $Ni_{0.9}Zn_{0.1}O$ nanoparticle

The surface morphology, structure and size of the nanoparticle was carried out using SEM image. The SEM image of title nanoparticle is clear. So provided in the form of nanoparticle and almost the morphology and uniformity in the image of them, relatively spherical and porous and beneath the high porosity for use as a catalyst for much is suitable. **Fig. 6.**

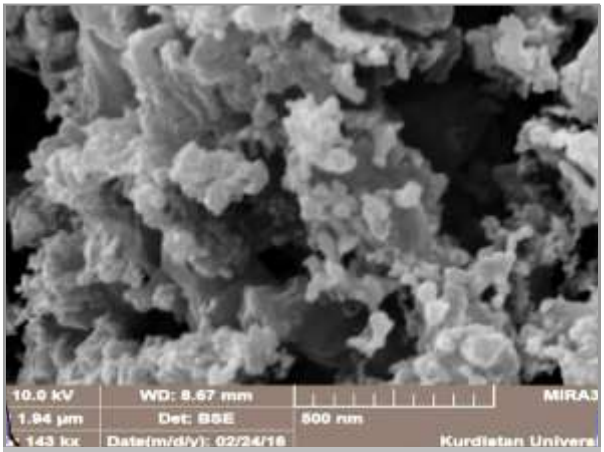


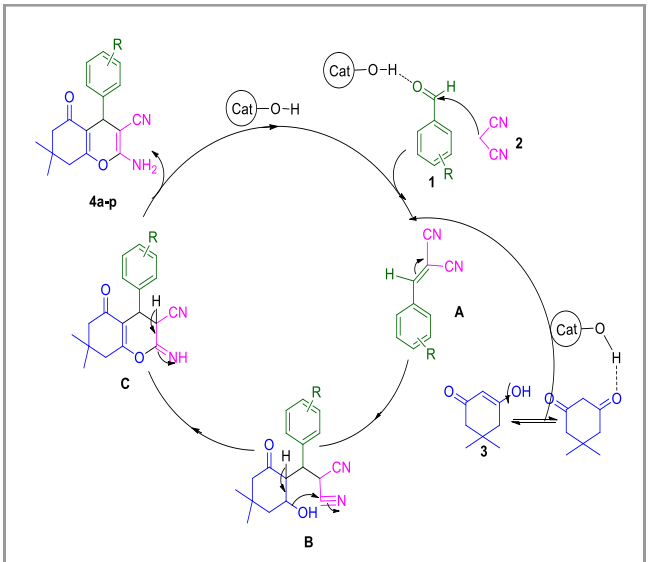
Figure 6. spectrum SEM of complex

4.preparation of tetrahydrobenzo[*b*]pyran

In the next step, $Ni_{0.9}Zn_{0.1}O$ nanocatalyst was used to prepare tetrahydrobenzo[b]pyran. The reaction between malononitrile (1.0 mmol), benzaldehyde (1.0 mmol) and dimedone (1.0 mmol) was considered as a model and the optimization study was carried out. At first, different amounts of catalyst were studied. The final results are summarized in Table 1 The best result was achieved in the presence of 5 mol % of $Ni_{0.9}Zn_{0.1}O$. Further increases to the amounts of catalyst did not have considerable effect on the reaction progress. In addition,to optimize the reaction temperature, the model reaction was performed by using 5 mol % of the catalyst at various temperatures in EtOH (Table 1, entries 6-8) and the best result was acquired at 60 °C (Table 1, entry 6). Finally, the effects of solvent were measured and it was obtained that the rate 2:1 EtOH/Water is better than other rates (As shown in Table 2, entry 3). The best datas were achieved when the reaction was carried out at 60 °C in the presence of $Ni_{0.9}Zn_{0.1}O$ (5 mol %) in 2:1 $EtOH/H_2O$, while the relative ratio of the substrate/benzaldehyde/malonitrile/dimedone was 1.0 mmol/1.0 mmol/1.0 mmol respectively.

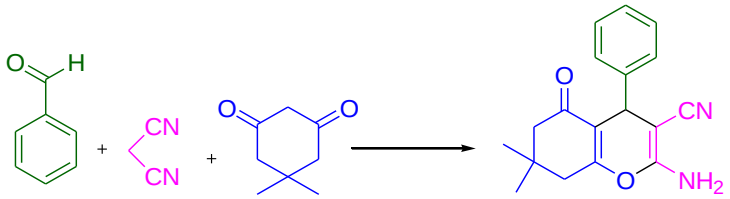
After the reaction states were optimized, we measured the range and feasibility of reactions by different aryl aldehydes. As shown in Table 3, the reaction of aryl aldehydes either with electron-withdrawing groups or electron-donating groups gave the corresponding products with good to excellent yields.

A proposed mechanism, demonstrating the task of $Ni_{0.9}Zn_{0.1}O$ in the prepares derivatives of tetrahydrobenzo [*b*] pyran has been showed in **scheme 1.** Knoevenagal concentration between at first 2-benzylidenemalononitrile A would be formed in via situ Knoevenagel condensation active aldehyes**1** and malononitrile**2**, and then Michael addition of A with dimedone**3** which undergoes intramolecular cyclization and tautomerization to afford the corresponding products (**4a-p**).



Scheme 1. The suggested mechanism for the prepare of tetrahydrobenzo[b]pyrans

Table 1. Effect of the catalyst level and temperature for the synthesis of tetrahydrobenzo[*b*]pyran



Entry	Temperature (°C)	Catalyst (mol %)	Time (min)	Isolated Yield (%)
1	70	-	12h	71
2	70	3	30	80
3	70	5	15	91
4	70	10	15	92
5	70	15	15	89
6	**60**	**5**	**15**	**90**
7	50	5	30	85
8	r.t	5	24h	Trace

Table 2. Effects of solvents for the prepare of tetrahydrobenzo [b] pyrans in the presence of $Ni_{0.9}Zn_{0.1}O$ (5 mol %) at 60 °C.

Entry	Solvent	Isolated Yield (%)
1	H_2O	65
2	H_2O/EtOH (1:1)	87
3	**H_2O/ EtOH (2:1)**	**86**
4	H_2O/ EtOH (1:2)	90
5	EtOH	85

The reusability of the catalyst from an economical and biologicallyhas been transformed as important factors in pathway of chemical synthesis. Thus, the reusability of catalyst was investigated in the prepare of **4a** as anpattern. In this method, after ending of reaction, hot ethanol was added and the catalyst was washed with ethanol, dried and reused. The results are showed in **Fig .7.** It was found that the reused catalyst works with the same performance up to the 2nd run, while in the 3rd, 4th and 5th runs product yield gets reduced slightly.

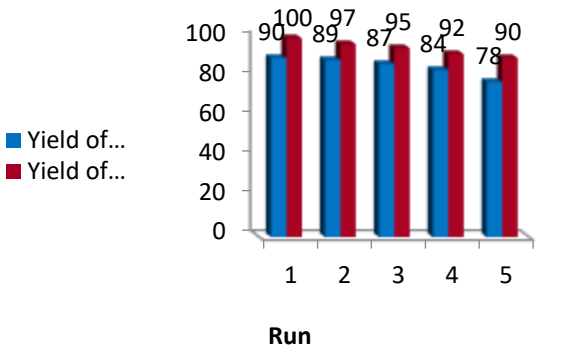


Figure 7. The reusability of the catalyst in the prepares of **4a** over 5 runs.

4.1. General method for preparation of tetrahydrobenzo[b]pyran derivatives

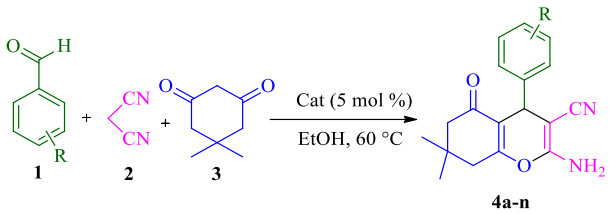
A mixture of aromatic aldehyde (1 mmol), dimedone (1 mmol), malononitrile (1 mmol) and $Ni_{0.9}Zn_{0.1}O$ (5 mol %) was added and stirred at 60 °C in EtOH/H_2O (3 mL) for an suitable time. The evolution of reaction was monitored by using TLC or thin layer chromatography. After finish of the reaction, hot ethanol was added to the mixture of reaction to dissolve the product and the catalyst was segregated through centrifugation. After recrystallization, pure crystals were created.

4.1.1.Spectral data for the selected samples:

2-Amino-5,6,7,8-tetrahydro-4-(4-nitrophenyl)-7,7-dimethyl-5-oxo-4H-chromene-3 carbonitrile (4g). IR (KBr) (νmax, cm^{-1}): 3285, 3160, 2960,2185, 1675, 1209; ^{1}H NMR (300 MHz, DMSO-*d*6): δ 0.95 (s, 3H, CH_3), 1.027 (s, 3H, CH_3), 2.12 (d, 1H, CH_2, J = 16.0 Hz), 2.27 (d, 1H, CH_2, J = 16.0 Hz), 2.51 (d, 2H, CH_2, J = 12.0 Hz), 4.36 (s, 1H,CH), 7.19 (s,br, NH_2), 7.44 (d, 2H, Ar, J =8.0 Hz), 8.16 (d, 2H, Ar, J = 8.0 Hz).

2-Amino-5,6,7,8-tetrahydro-4-(4-methoxyphenyl)-7,7-dimethyl-5-oxo-4H-chromene-3-carbonitrile (4l). IR (KBr) (νmax, cm^{-1}): 3465, 3320, 2955, 2190, 1676, 1247; ^{1}H NMR (300 MHz, DMSO-*d*6): δ 0.95 (s, 3H,CH_3), 1.04 (s, 3H,CH_3),2.09 (d, 1H, CH_2, J = 18.0 Hz), 2.25 (d, 1H, CH_2, J = 18.0 Hz), 3.37 (s, 1H,CH_2), 3.72 (s, 3H,OCH_3), 4.13(s, 1H,CH), 6.85 (d, 2H.Ar, J = 8.0 Hz), 6.97 (br,NH_2), 7.06 (d, 2H, Ar, J = 8.0 Hz).

Table 3. One-pot, three-component preparesof tetrahydrobenzo[*b*]pyrans in the presence of 5 mol % of catalyst at 60 °C.



Entry	R	Product	Time (min)	Yield (%)	Mp (°C)	
					Found	Reported[Ref]
1	H	**4a**	15	90	231-234	**233-234 [35]**
2	4-Cl	**4b**	10	92	209-211	**215-217 [36]**
3	2-Cl	**4c**	20	88	217-219	**214-215[37]**
4	2,4-diCl	**4d**	15	85	120-122	**115-117[38]**
5	4-F	**4e**	15	89	219-221	**221-223[39]**
6	4-Br	**4f**	10	90	230-232	**229-230[40]**
7	4-NO_2	**4g**	10	93	172-175	**169-171 [41]**
8	3-NO_2	**4h**	15	91	205-208	**208-211[41]**
9	2-NO_2	**4i**	15	90	218-220	**224-226[36]**
10	4-$N(Me)_2$	**4j**	45	87	202-204	**198-200 [35]**
11	4-Me	**4k**	30	84	214-216	**214-216[42]**
12	4-OMe	**4l**	20	88	203-205	**199-201[42]**
13	4-OH	**4m**	40	83	208-211	**210-212 [43]**
14	2,3-diOMe	**4n**	25	85	213-215	**217-219 [44]**
15	2-Furyl	**4o**	25	86	217-220	**217-219[45]**
16	Thiophene-2-	**4p**	35	85	211-214	**210-212 [46]**

5.Conclusion

In summary, we have successfully synthesized precursor Zn(II)-Ni(II) complex with phen=1-10-phenanthroline, 2-aminobenzoate and thiosyanate ligands. $Ni_{0.9}Zn_{0.1}O$ nano-particle prepared using thermal decomposition method from title complex. The size of the nano-particle was measured using XRD and SEM. The results were in good agreement with each other. XRD reveals the pure phase formation of title nano-particle, which is further confirmed by FT-IR spectra. All results reveal that thermal decomposition method can be employed successfully as a simple, efficient, low cost, environmentally friendly and very promising method for the synthesis of nanoscale materials without special conditions, such as long reaction times, and high pressure. This method may be extended to synthesize other metal oxide nanoparticles, nanowires, nanodisks and even nanotubes. Finally, the nano-particle has been used as a catalyst for the preparation of tetrahydrobenzo[b]pyran. The best datas were achieved when the reaction was carried out at 60 °C in the presence of $Ni_{0.9}Zn_{0.1}O$ (5 mol %) in 2:1 EtOH/H_2O.

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