

Nickel Oxide Nanoparticles: Synthesis, Characterization and Recyclable Catalyst

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Abstract— Solid state nanomaterials are of current interesting endeavor for researcher. One of them is catalysis, it is an important field of application for metal and metal oxide nanoparticles (NPs). With this perspective views in the present article, we have summarized synthesis and characterization of nickel oxides nanoparticles by using effective protective agent. The synthesized NiO NPs were characterized by using spectroscopic techniques viz. FTIR spectroscopy, Powder X-ray Diffraction pattern, Ultraviolet-visible Spectroscopy (UV), Energy Dispersive X-ray Spectroscopy (EDS), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Atomic Force Microscopy (AFM) and Thermogravimetry (TG/DTG). The size and morphology behavior of NPs was studied with the help of PXRD, SEM, TEM and AFM which shows particle of varying shapes and sizes. Furthermore, a convenient, alternative and efficient one-pot method was developed for solvent free green synthesis of amidoalkyl naphthol derivatives by using nano catalyst through condensation reaction of β -naphthol, aldehydes and urea with high speed stirring. Present protocol is favored because it offers advantages of high yields, short reaction times, simplicity and easy workup. The efficacy of NPs catalyst exhibited excellent recyclability and reusability up to four times without any additional treatment.

Keywords: Nickel Oxide Nanoparticles; TEM; AFM; Recyclable catalyst; β -Naphthols; Amidoalkyl naphthol derivatives

1 INTRODUCTION

Nowadays the uses of nano catalyst are emerging trends in the field of nanoscience and nanotechnologies which are leading to unprecedented understanding and control over the bulky matter. Instances, an immediately synthesis of organic compound by using nano catalyst are forthcoming towards short reaction time and chemical sustainability. However, the utilization of more benign nano catalysts are most cost-effective and environmentally beneficial [1]. Particularly, metal oxide nanoparticles catalyzed one-pot, multi-component reaction which have achieved significant attention in economic and environment as they generates molecules in a single step synthetic operation [2]. Notwithstanding, metal oxide nanoparticles have emerged as a new generation's catalysts owing to their large surface area, easy dispersion in several solvents, and their efficient recovery from the reaction medium [3-4]. The use of metal oxide nanoparticles as heterogeneous catalysts for a synthesis of various pharmaceutical drugs, organic and inorganic compounds have reached at momentous levels, not only for the possibility to perform environmentally benign, but also for the good yields [5]. The literature data also reveals that the numerous catalysts were used for the synthesis of amidoalkyl naphthols [6-8]. However, it was found that few catalysts have some disadvantage especially in aspect of green chemistry including prolonged reaction times, low yields, and toxicity of the catalyst. Therefore, it was a challenge to all the researchers to introduce a clean process, and eco-friendly and recyclable nano catalyst for synthesis of organic/inorganic

compounds. With this background we have effectively synthesized heterogeneous and recyclable nano catalyst for the transformation of known organic compound via alternative benign procedure.

Moreover, a variety of heterogeneous nano catalysts has shown promising potential in sustainable applications, and catalysts were recovered by easy filtration and reused for the next catalytic cycles with the minimum wastes [9-12]. Among the various nano heterogeneous catalysts, the nano metal oxides have received much more attention because of their large surface area, and high thermal stability. Especially, NiO nanoparticles (NiO NPs) as a nano catalyst for the synthesis of organic derivatives have emerged a considerable interest due to their inherently large surface-to-volume ratio and tunable morphology as compared with the bulk metals. Literature data also reveals that a few organic transformations were carried out by using capping agent free nano NiO [13-15]. On the other hand, the stability and activity of nano catalyst can be increased by using surfactant, protecting agent, capping agent, and stabilizing agent [16-17]. With keeping this in mind we have successfully synthesized NiO nano catalyst using capping agent for carry out the synthesis of amidoalkyl naphthol derivatives. Herein, to the best of our knowledge, an efficient synthesis of 1-amidoalkyl-2-naphthols by the reaction of 2-naphthol, aldehydes and amides (12 mol %) under solvent-free conditions (Scheme 1) has been reported for the first time by using NiO nano catalyst in one-pot condensation method.

2 Experimental

All the chemicals and solvents were used without further purifications. They includes Nickel(II) nitrate hexahydrate (Sigma-Aldrich), Ethylene glycol (Merck), Sodium dodecyl sulphate (Merck), Hydrazine hydrate 80% (Sd Fine), Aromatic aldehydes (Merck), β -naphthol (Merck), Acetamide (Merck) and Urea (Merck).

The size and morphology of NiO NPs were examined by JEOL model JSM-690LV, Scanning Electron Microscopy whose

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maximum magnification is 300,000X and resolution is 3nm at the Sophisticated Test and Instrumentation Center, Cochin University Kerala. TEM images were formed using CM200 which can produce magnification details up to 1,000,000X with resolution better than 10 Å at Indian Institute of Technology Pawai, Mumbai. The qualitative elemental analyses of the powder sample were studied by JEOL Model JED-200, Energy Dispersive Spectroscopy (EDS) and thermal analyses (TG/DTG/DTA) at heating rate 10 °C/min under nitrogen atmosphere at the Sophisticated Test and Instrumentation Center, Cochin University Kerala. The crystal structure of the sample was characterized by PXRD, Bruker AXS D8 Advance X-ray diffractometer using CuK α radiation. Infrared spectroscopy was recorded at a 2 cm⁻¹ resolution from 4000 to 400 cm⁻¹ on a Bruker IFS 66v Fourier transform spectrometer using KBr pellets. ¹H-NMR was carried out NMR Spectrometer model Avance-II (Bruker) is the acquisition in the SAIF Chandigarh, India. The instrument is equipped with a cryomagnet of field strength 9.4 T. Its ¹H frequency is 400 Mhz.

2.1 Synthesis of NiO nano catalyst

NiO nano catalyst was prepared by sol gel method. In this method, 0.1 M solution Nickel nitrate hexahydrate in double distilled water was mixed with 0.01 g of Sodium Dodecyl sulphate as a capping agent and titrated with 0.1 M NaOH solution drop by drop with constant stirring until green precipitate of Ni(OH)₂ were sediment. The green precipitates of Ni(OH)₂ were washed with double distilled water, ethanol and finally acetone repeatedly in order to remove the capping agent. The Ni(OH)₂ were dried at 120 °C under vacuum oven for 3 h followed by calcinated at 400 °C to obtain black color NiO NPs.

2.2 General procedure for the synthesis of amidoalkyl naphthol derivative

Nickel oxide nano catalyst (12 mol %) was added into a mixture of aromatic aldehyde (1 mmol), β -naphthol (1 mmol) and urea or acetamide (1.1 mmol), then the reaction mixture was heated to 100 °C and maintained for the appropriate time. After completion of the reaction (monitored by TLC), the reaction mixture was diluted with water, and the resulting solid product was collected by filtration, which was purified by recrystallization from EtOH/H₂O.

2.3 Spectral data

Selected data for typical compounds are given below.

1. Entry (4a): FTIR (KBr) 3410, 3240, 29284, 1642, 1583, 1530, 1437, 1373, 1348, 1270, 1220, 1170, 1039, 997, 930, 875, 839, 808, 745 cm⁻¹. ¹H NMR (400 MHz, DMSO-*d*₆) δ = 9.96 (s, 1H), 8.55–8.42 (d, 1H), 7.89–7.74 (m, 3H), 7.32–7.11 (m, 9H), 1.95 (s, 3H).
2. Entry (4b): FTIR (KBr) 3370, 3212, 2924, 2840, 1649, 1578, 1524, 1438, 1474, 1350, 1319, 1205, 1169, 1110, 1065, 1035, 1001, 925, 807, 741, 710 cm⁻¹. ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.13 (s, 1H), 8.63–8.60 (d, 1H), 8.05–8.001 (m, 2H), 7.83–7.78 (m, 3H), 7.50–7.40 (m, 2H), 7.35–7.25 (m, 1H), 7.19–7.15 (m, 3H), 2.05 (s, 3H).

Entry (4d): FTIR(KBr) 3391, 3072, 1640, 1602, 1524, 1439, 1352, 1281, 1246, 1167, 1066, 1093, 983, 935, 883, 855, 825, 734, 750 cm⁻¹. ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.21 (s, 1H), 8.79–8.66 (d, 1H), 8.16–8.13 (m, 2H), 7.85–7.79 (m, 3H), 7.43–7.38 (m, 3H), 7.28–7.16 (m, 2H), 2.05 (s, 3H).

Entry (4f): FTIR(KBr) 3399, 3068, 3022, 2969, 2839, 2797, 2714, 2614, 1629, 1589, 1515, 1458, 1370, 1334, 1304, 1279, 1255, 1177, 1088, 1065, 1043, 983, 930, 886, 848, 822, 814, 802, 745 cm⁻¹. ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.3 (s, 1H), 8.4 (d, 1H), 7.80–7.73 (m, 4H), 7.30–7.04 (m, 6H), 2.55 (s, 3H), 1.95 (s, 3H).

Entry (4i): FTIR (KBr) 3439, 3390, 3370, 3193, 2920, 2849, 1726, 1655, 1601, 1520, 1476, 1439, 1355, 1315, 1267, 1206, 1142, 1095, 1040, 926, 812, 740 cm⁻¹. ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.11 (s, 1H), 8.05–8.03 (m, 2H), 7.89–7.79 (m, 3H), 7.54–7.46 (m, 3H), 7.31–7.09 (m, 3H), 5.84 (s, 2H).

3 Result and discussion

3.1 Microscopic characterization of nano catalyst

The PXRD pattern of synthesized NiO nano catalyst is match well with Bragg reflections of the standard face-centered cubic structure [ICDD: 04-0835], diffraction peaks at 37.11 °, 43.18 °, 62.70 °, 75.20 ° and 79.21 ° can be indexed as (111), (200), (220), (311), and (222) crystal planes, respectively [18]. The average crystallite size is measured by X-ray diffraction line broadening using the Debye–Scherrer formula. The average crystallite size of NiO was found to be 18 nm.

$$D = \frac{K\lambda}{\beta \cos\theta}$$

In above equation, D represents the average crystallite size, K = 0.89 is the Scherrer constant, λ = 1.5406 Å is the wavelength of the X-ray (Cu K α ₁ radiation), θ is the diffraction angle of the peak and β represents the full width at half maximum of the peaks. XRD data clearly shows uniform size and well dispersion in the bulk state with diameter around 30-50 nm. This indicates that sodium lauryl sulphate (SDS) as a surfactant played a key role in the formation of well-dispersed nanoparticles by avoiding their aggregation and leads to the formation of uniform sized NiO during heat treatment in air [19].

Additionally, scanning electron microscopy (Fig. 1) and transmission electron micrographs were recorded using a copper grid dipped in a solution containing NiO NPs dispersed in ethanol by ultrasonication (Fig. 2a-c). From Fig. 2a-b it is revealed the presence of a large number of NiO particles with uniform spherical shape and size around 20-30 nm and well dispersed in the bulk material. Also from this figure (Fig. 2c) clearly confirmed that an electron diffraction pattern of the selected area of nanoparticles appeared strong diffraction spots rather than diffraction rings confirmed the formation of single crystalline cubic nickel oxide [20].

Moreover, the 2D and 3D-topological AFM images of NiO NPs revealing both surface roughness and particle agglomeration, and it can be easily seen on the surface images. Also, a NiO NPs histogram measurement exposes the particles size in the range 20 to 60 nm which was supported by SEM and TEM.

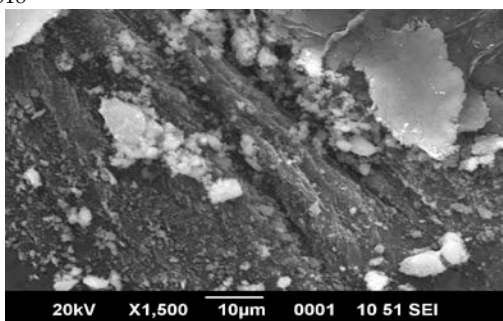


Fig. 1: SEM image of NiO NPs

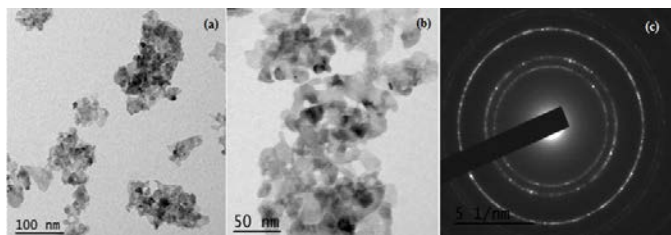


Fig. 2: TEM images of NiO NPs; (a) lower magnification, (b) higher magnification (c) selected area electron diffraction pattern (SAED).

Infrared spectrum of nano catalyst was taken after calcined at 400 °C. but the broad absorptions bands at 3326 and 1,606 cm^{-1} are assigned due to O-H stretching and bending modes of water, respectively [21]. The peaks were observed at 1410 cm^{-1} , 1301 cm^{-1} and 1032 cm^{-1} which assigned to the O-C=O symmetric and asymmetric stretching vibrations and the C-O stretching vibration originating from the adsorption of atmospheric CO_2 or ethanol respectively. But the intensity of the band has weakened, which indicates that the ultrafine powers tend to strong physically absorption to H_2O and CO_2 . Further, the peaks were found at 445 and 1000 cm^{-1} may be due to the Ni-O stretching vibration which was reported previously by other researchers [22]. Some peaks were observed in the range 2000 to 2500 cm^{-1} which might be due to the presence of capping agent (Sodium Dodecyl sulphate).

The UV-visible absorption spectrum of the NiO NPs shows strong absorption at wavelength about 345 nm in the UV region which may be due the blue shifted from the absorption edge of nano catalyst. Further, the band-gap energy was calculated from UV-absorption data is 3.53eV which is close to the value (3.55 eV) reported by Alagiri M. *et. al* [21].

3.2 Thermal analysis of nano catalyst

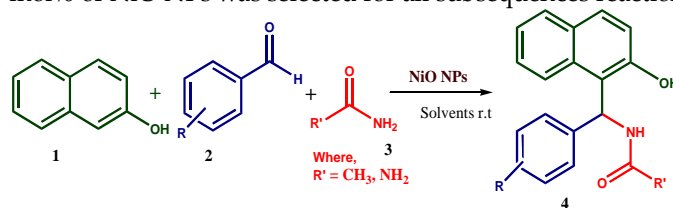
Besides, the thermogravimetric analysis is always used to evaluate the thermal degradation behaviour and thermal stability of inorganic compound [23-26]. The typical TG-DTG/DTA curves of the nano catalyst were clearly demonstrated the weight loss occurs in two temperature stages: first step decomposition at 40–205 °C with supported one endothermic T_{DTA} peak at 100 °C and one T_{DTG} peak at 64.77 °C, while second step decomposition at 206–720 °C with supported one exothermic T_{DTA} peak at 398 °C and one weak T_{DTG} peak at 510 °C and one sharp T_{DTG} peak at 719 °C. The first stage decompositions were attributed due to desorption of absorbed water molecules. And the second stage decomposition occurs

due to thermal decomposition of attached organic moiety (capping agent). Finally, the last part of the curve suggested formation of nickel oxide nanoparticles.

3.3 Nano NiO-catalyzed amidoalkyl naphthols derivatives

After successfully synthesis of spectral and thermal characterization of nano catalyst, we applied this nano catalyst in the synthesis of previously reported compounds i.e. amidoalkyl naphthols derivatives. These compounds were already synthesized by different techniques using simple catalyst. To the best of our knowledge the green synthesis of amidoalkyl naphthols derivatives through the reaction of different aromatic aldehydes, β -naphthol, and amide or urea using NiO NPs as catalyst is reported for the first time. In the present article our main goal is to develop green cost-effective solvent free synthesis, benign environmentally, high yields, short reaction times, simplicity easy workup, and reusable-recyclable nano catalyst. Further, we also demonstrated comparatives yields which were obtained by using NiO nano catalyst under solvent free condition with reported normal synthesis reaction. Fortunately, we have noteworthy obtained a better efficiency and reusability of nano catalyst up to five cycles. The synthesis of biologically active amidoalkyl naphthols derivatives were achieved by the one-pot, three-component condensation of an aromatic aldehydes, β -naphthol, and amide or urea in the presence of freshly prepared NiO NPs using capping agent under solvent free condition at 80 °C achieved excellent products yields (Table 1).

In our initial study, for optimization of reaction conditions, the reaction of benzaldehyde, β -naphthol and urea was used as a model reaction to optimize the reaction conditions. First the reaction was conducted in various solvents using nano catalyst under refluxing conditions and also under solvent free conditions (Table 1). As it can be seen from the Table 1 (Table 1, entry 6), the best results are obtained under solvent free conditions. Also, we check out the catalytic activity with amount of catalyst is shown in Fig. 3a. The reaction were carried out with various amount of nano catalyst (3 mol% to 18 mol%) for the synthesis of amidoalkyl naphthol. Significantly, the yield of products increased remarkably from 32% to 94 % with increases the concentration of catalyst amount from 3 mol % to 12 mol%. However, it was observed that when the catalyst amount increased from 12 mol % to 18 mol% no further increase in the yield of product. Therefore the amount of 12 mol% of NiO NPs was selected for all subsequences reaction.



Scheme 1: Synthesis of amidoalkyl naphthols derivatives under solvent-free conditions.

Additionally, the effect of temperature in solvent free conditions was studied by carrying out the reaction at 60 °C, 70

°C, 80 °C, 90 °C, 100 °C, 110 °C and 120 °C. The results showed that 80 °C would be the best temperature for all reactions. Under the optimized reaction conditions, a series of amidoalkyl naphthols derivatives (Table 2, entries 1-12) were synthesized. Interestingly, in all the cases, aromatic aldehydes substituted

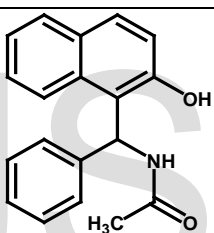
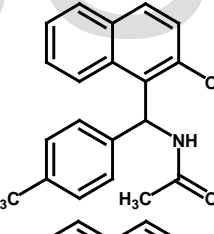
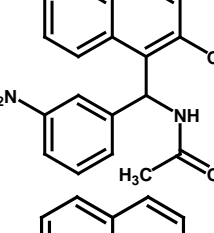
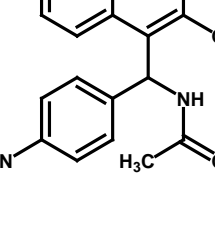
with either electron-donating or electron-withdrawing groups underwent the reaction smoothly and gave the expected products in good to high yields (Table 2, entries 4b, 4g, 4f and 4l) under the same reaction condition.

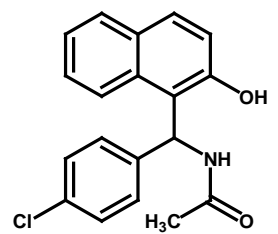
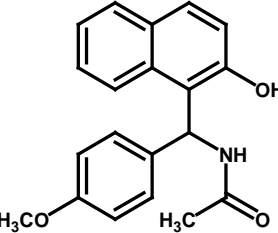
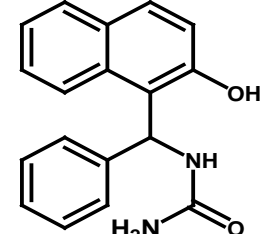
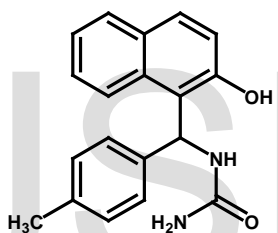
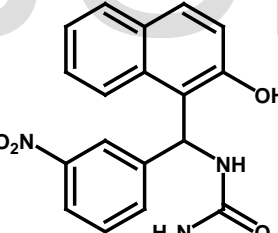
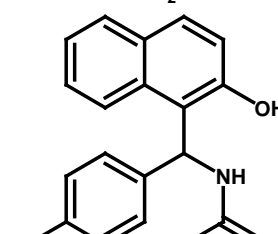
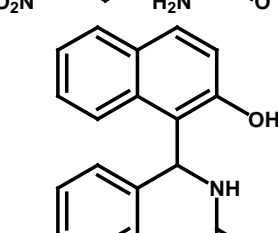
Table 1: Synthesis of amidoalkyl naphthol derivative in various solvents using NiO NPs and solvent free condition

Entry	Solvent	Time(min)	Yield(%) ^a
1	Ethanol	80	60
2	Methanol	80	59
3	CH ₃ CN	80	58
4	CHCl ₃	80	66
5	Toluene	80	45
6 ^b	Solvent-free Condition	60	94,93,91,90

Isolated Yield, Catalyzed recycled four times

Table 2: Amidoalkyl naphthols catalyzed by NiO NPs under solvent free condition

Entry	R at 2	R'at 3	Product 4	Time (h)	Yield (%) ^b	Mp (°C)	
						Found	Litearure [27-30]
1	H	CH ₃		7	76	244	245-246
2	4-CH ₃	CH ₃		7.5	73	220	221-222
3	3-NO ₂	CH ₃		7	79	242	241-242
4	4-NO ₂	CH ₃		5.5	79	249	249-250

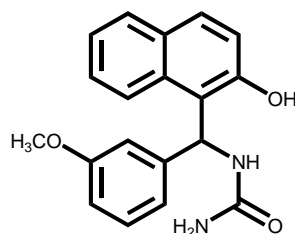
5	4-Cl	CH ₃		6	81	224	224-225
6	4-OCH ₃	CH ₃		6	80	186	186-187
7	H	NH ₂		5	81	182	180-181
8	4-CH ₃	NH ₂		5	79	117	117-118
9	3-NO ₂	NH ₂		6	80	187	187-188
10	4-NO ₂	NH ₂		4.5	79	163	162-163
11	4-Cl	NH ₂		5	78	171	170-171

12 3-OCH₃ NH₂

6.5

79

242 242-245



^aReaction Condition: aldehyde (1.0 mmol), β - Naphthol (1.0mmol), Urea/amide (1.5 mmol), NiO NPs 12 mol %, solvent free stirring at room temperature

^bIsolated Yields

3.4 Reusability of nano catalyst

We have also investigated the possibility of recycling the nano catalyst catalyst (NiO NPs). The nano catalyst recovered by filtration from the model reaction was checked in the subsequent runs without further purification (Fig. 3). The activity of the nano catalyst did not get much affected in terms of yields after five successive runs for the model reaction, no fresh catalyst was added. It exposed that the nano catalyst displayed very good reusability. Notwithstanding, the tentative mechanism for this reaction has been presented (Scheme 2) and to best of our knowledge this plausible mechanism do not produced earlier. In the first step, β -naphthol reacted with aromatic aldehydes in the presence of NiO nano catalyst which produced *ortho*-quinone methides (*o*-QMs) [31-32]. After that, in the second step, the *o*-QMs were reacted with urea (amides) *via* conjugated addition to develop amidoalkyl naphthol as a final product (amidoalkyl naphthols derivatives).

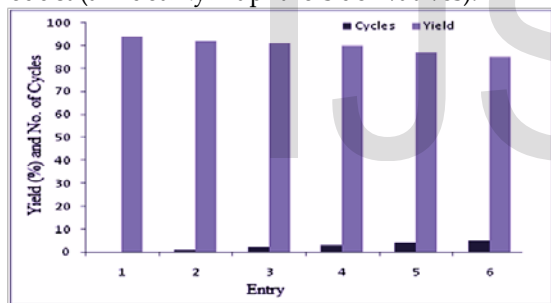
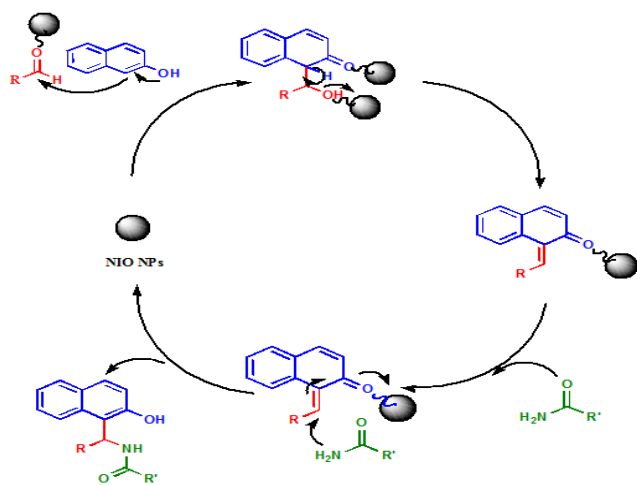


Fig. 3: Recyclability of nano catalyst



Scheme 2: Tentative mechanism for synthesis of Amidoalkyl naphthols derivative catalyzed by NiO NPs

4 Conclusion

In conclusion, here we have synthesized NiO NPs with average crystalline size 40 nm with sol-gel method by using sodium dodecyl sulphate as surfactant. Furthermore, NiO NPs was used as nano catalyst for an efficient and very simple in the solvent free green synthesis of amidoalkyl naphthols derivative by stirring at 80 °C. This catalyst is expected to contribute to the development of more environment-benign methods and forms part of nano-metal chemistry. The mildness of the conversion, experimental simplicity, compatibility with various functional groups, excellent yields, shorter reaction time, and the easy work-up procedure makes this procedure more attractive in synthesizing a variety of these derivatives.

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