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A highly efficient synthesis of 2,4-diamino-6-arylpyrimidine-5-carbonitrile derivatives using $NiCo_2O_4$@Ni(BDC) metal-organic frameworks as a novel and bifunctional catalyst

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ABSTRACT

The $NiCo_2O_4$@Ni(BDC) metal-organic frameworks as a suitable and green catalyst have been developed for the one-pot synthesis of 2,4-diamino-6-arylpyrimidine-5-carbonitrile derivatives. The multi-component reactions of aldehydes, malononitrile and guanidine hydrochloride were efficiently catalyzed using a novel metal-organic framework under reflux conditions. Simple procedures, high yields, short reaction times, and reusability of the catalyst are advantages of this protocol. The catalyst was fully characterized by FT-IR, SEM, XRD, EDX, TGA, VSM and TEM analysis.

1. Introduction

Recent developments in nanoscience and nanotechnology have led to new research interest in employingnanometer-sized particles as an alternative matrix for catalytic reactions. Many reports show asurprising level of performance of nanoparticles as catalysts in terms of reactivity, selectivity and improved yields of products [1].

Nanoporous metal-organic frameworks (MOFs), which are made of inorganic building units and organic linkers, have been taken into consideration in the last decade [2]. This new hybrid material has a crystalline three or two and one-dimensional open framework [3]. MOF-based structures have unique properties and features, including structural diversity, high pore volume, high porosity [4], and high capacity for guest molecular acceptance due to these specific features. MOFs have been used in a variety of fields such as storage and separation of gases and as catalyst [5]. Compared to purely inorganic porous materials, MOFs show higher flexibility for the design of catalytic centers [6]. This flexibility has made them attractive materials for heterogeneous catalysts. Because of the considerable variety of organic linkers and metal ions, MOFs can be modulated in several ways. The size and appearance of the framework can be changed by using various linkers, also changing their connectivity via adding different functional groups on the linkers [7,8]. MOFs as green catalysts were used to increase the speed a number of organic reactions [9]. For example molybdenum (VI) modified Zr-MOF catalysts for epoxidation of olefins [10], postsynthetic modification of IRMOF-3 with a copper iminopyridine complex as heterogeneous catalyst for the synthesis of 2-aminobenzothiazoles [11], basic isoreticular metal–organic framework (IRMOF-3) porous nanomaterial as an appropriate and green catalyst for selective unsymmetrical Hantzsch coupling reaction [12].

Multi component reactions (MCRs) have become very absorbing as tools for the fast preparation of compound libraries of small molecules [13,14]. MCRs are a resource which is time-effective and therefore an economically desirable process in diversity generation. Also, MCRs are effective, environmentally friendly, fast, atom economic and time-saving. They are a suitable method for the preparation of compounds with medicinal and biological properties [15]. In recent years, a lot of attention has been paid to the three-component reaction of aldehydes, malononitrile and guanidine.

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The synthesis of pyrimidine derivatives has attracted much attention due to the therapeutic and biological properties. These features are: antiviral [16–18], antineoplastic [19–21], inflammatory properties [22] and antibiotic [23]. Pyrimidines also exhibit a limited area of pharmacological activities such as anticancer [24,25], cardioprotective effects [26], antibacterial [27–29] and antifungal activities [30,31]. There are many synthetic methods for the synthesis of pyrimidine derivatives which include the condensation of aromatic aldehydes, ethyl cyanoacetate and guanidine nitrate [32], 1,3-indandione, amino uracils and isatins or acenaphthylene-1,2-dione [33], condensation of 6- amino-1,3-dimethyl uracil, 1,3-dicarbonyl compounds and aromatic aldehydes [34]. However, there are only few reports for the synthesis of 2,4-diamino pyrimidine-5-carbonitriles via multi component reactions of aromatic aldehydes, malononitrile and guanidine hydrochloride, which catalyzed by some catalysts such as sulfonic acid functionalized LUS-1 [35], potassium carbonate and tetra butyl ammonium bromide [36] and sodium hydroxid [37].

Herein, with the aim to improve more efficient synthetic procedures, reduce the number of separate reaction steps, minimize by-products and in continuation of our efforts to the preparation of heterocyclic compounds [38–41], we report here a novel and mild method for the preparation of 2,4-diamino-6-arylpyrimidine-5-carbonitriles via multi components reactions of aromatic aldehydes, malononitrile and guanidine hydrochloride in the presence of $NiCo_2O_4$@Ni(BDC) metal-organic framework (Scheme 1).

2. Experimental

2.1. Materials

Chemicals were purchased from the Sigma-Aldrich and Merck in high purity. All of the materials were of commercial reagent grade and were used without further purification. All melting points are uncorrected and were determined in capillary tube on Boetius melting point microscope. ^1H NMR and ^{13}C NMR spectra were obtained on Bruker 400 MHz spectrometer with DMSO-d_6 as solvent using TMS as an internal standard. FT-IR spectra were recorded on Magna-IR, spectrometer 550. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyzer. Powder X-ray diffraction (XRD) was carried out on a Philips diffractometer of X'pert Company with mono chromatized Cu Kα radiation ($\lambda = 1.5406$ Å). Microscopic morphology of products was visualized by SEM (LEO 1455VP). The mass spectra were recorded on a Joel D-30 instrument at an ionization potential of 70 eV. The compositional analysis was done by energy dispersive analysis of X-ray (EDX, Kevex, Delta Class I). Thermogravimetric analysis (TGA) was performed on a Mettler Toledo TGA under argon and heated from room temperature to 825 °C. The approximate sample weight was 10 mg in TG experiment with 10 °C/min heating rate.Magnetic property of the sample was measured with a vibrating sample magnetometer (VSM) instrument MDKFT.Transmission electron microscopy (TEM) was performed with a Jeol JEM-2100UHR, operated at 200 kV.

2.2. Preparation of $NiCo_2O_4$ nanoparticles

Magnetic $NiCo_2O_4$ NPs were prepared by the reported co-precipitation method [42]. In a typical experiment, $Ni(NO_3)_2.6H_2O$ (0.18 g, 1 mmol) and $Co(NO_3)_2.6H_2O$ (0.36 g, 2 mmol) (Ni and Co, molar ratio 1:2) was dissolved in 30 mL of deionized water. Subsequently 12 mmol of urea was added into the mixture and stirred for 30 min. The resulting mixture was transferred into 50 mL teflon-lined autoclave. The autoclave was sealed and heated at 120 °C for 6 h. After cooling slowly to room temperature, the resulting precipitate was collected, washed with deionized water and ethanol for several times, and dried at 60 °C for 24 h. Finally, the dried black powder was further annealed at 400 °C for 3 h.

2.3. Preparation of $NiCo_2O_4$@Ni(BDC) nanocomposite

As shown in Scheme 2, the novel magnetic $NiCo_2O_4$@Ni(BDC) was prepared in a two-step process by using $NiCo_2O_4$ as a magnetic core and terephthalic acid (H_2BDC) as a linker. Firstly, the $NiCo_2O_4$ (0.1 g) was dispersed into 15 mL ethanol solution under sonication for 15 min at room temperature. Then, $Ni(NO_3)_2.6H_2O$ (0.42 g) and terephthalic acid (0.16 g) were added into the mixture and again stirred under sonication for 30 min at room temperature. Next, the above mixture was stirred under reflux conditions for 12 h at 80 °C. When the reaction was completed, the product was separated from the solvent by magnet and washed several times with water and ethanol and then dried under vacuum at 60 °C for 12 h. The prepared $NiCo_2O_4$@Ni(BDC) MNPs have been structurally characterized by FT-IR, XRD, TGA, FE-SEM, EDX, TEM and VSM analysis.

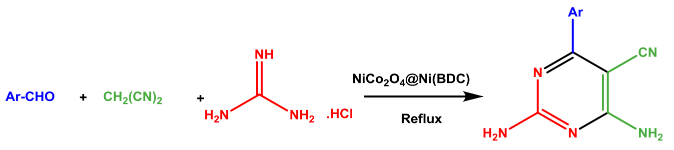
2.4. General procedure for the synthesis of 2,4-diamino-6-arylpyrimidine-5-carbonitriles catalyzed by $NiCo_2O_4$@Ni(BDC) under reflux conditions

A mixture of aromatic aldehyde (1 mmol), malononitrile (1.2 mmol), guanidine hydrochlorid (1 mmol), $NiCo_2O_4$@Ni(BDC) MNPs (0.008 g) in ethanol (5 mL) was heated in the oil bath under reflux conditions at 80 °C for 2–2.5 h. After completion of the reaction as indicated by TLC, the reaction mixture was cooled to room temperature and solid obtained was dissolved in dichloromethane, the catalyst was insoluble in CH_2Cl_2 and separated by a simple filtration. The solvent was evaporated and the residue was recrystallized from ethanol to give pure compounds in high yields. All of the products were characterized and identified with m.p., ^1H NMR, ^{13}C NMR and FT-IR spectroscopy techniques.

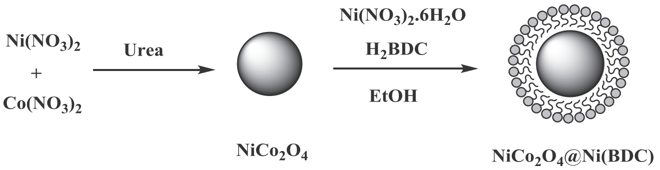
2.5. Spectral data of the new products

2.5.1. 2,4- diamino-6-(4-isopropylphenyl)pyrimidine-5-carbonitrile) **4j)**

Gray solid; m.p 125–128 °C; ^1H NMR (DMSO-d_6, 400 MHz) δ.: 1.19 (d, 6H,2CH_3), 2.9 (m, 1H, CH), 7.4 (s, 2H, NH_2), 3.4 (s, 2H, NH_2), 7.1–7.3 (m, 4H, H- Ar); ^{13}C NMR (DMSO-d_6, 100 MHz) δ: 23.1, 33.0, 79.2, 115.3, 124.3, 127.9, 131.9,148.1, 163.7, 165.5, 169; IR (KBr) ν:



Scheme 1. Synthesis of 2,4- diamino-6-arylpyrimidine-5-carbonitriles using $NiCo_2O_4$@Ni(BDC)



Scheme 2. Preparation of $NiCo_2O_4$@Ni(BDC) MOF.

3350 (NH_2), 2197 (C≡N), 1485 (C=C), 1383 (CH_3), cm^{-1}; MS (EI) (*m/z*): 253.31; Anal.calcd. for $C_{14}H_{15}N_5$: C 66.38, H 5.97, N 27.65. Found C 65.7, H 5.89, N 27.5.

2.5.2. 2,4- diamino-6-(4-cyanophenyl)pyrimidine-5-carbonitrile (4k)

Yellow solid, m.p. 167–170 °C, ^{1}H NMR (DMSO-d_6, 400 MHz) δ: 1.19 (d, 6H,2CH_3), 2.9 (m, 1H, CH), 7.4 (s, 2H, NH_2), 6.2 (s, 2H, NH_2), 7.2–7.4 (m, 4H, H- Ar); ^{13}C NMR (DMSO-d_6,100 MHz) δ: 79.6, 112.2, 115.3, 118.4, 126.0, 132.5, 139.1,163.5, 166.3, 169.3; IR (KBr) *v*: 3350(NH_2), 2198(C≡N), 1698 (C=N), 1400 (C=C), cm^{-1}; MS (EI) (*m/z*): 236.08; Anal. calcd. for $C_{12}H_8N_6$: C 61.01, H 3.41, N 35.58. Found C 61.0, H 3.43, N 35.0.

3. Results and discussion

Initially, the metal-organic framework $NiCo_2O_4$@Ni(BDC) was synthesized and characterized by different spectroscopic techniques including FE-SEM, EDX, XRD, TGA, TEM, VSM and FT-IR analysis.

Scanning electron microscopy (SEM) is an effective tool for determining the size distribution, particle shape, and porosity. The basis of the operation of a scanning electron microscope is the interaction of electron beam with matter, which carries out the emission of electrons and photons from the material. Fig. 1 shows FE-SEM images of the as-synthesized $NiCo_2O_4$@Ni(BDC) sample. Scanning electron microscopy of $NiCo_2O_4$@Ni(BDC) is obtained to investigate the morphology and the particle size of this organic–inorganic hybrid material. The results revealed that the presence of well-formed crystals.

The composition of $NiCo_2O_4$@Ni(BDC) was further studied by Fourier transform infrared (FT-IR) (Fig. 2). Compared to $NiCo_2O_4$, the $NiCo_2O_4$@Ni(BDC) spectrum shows higher adsorption bands, which are related to the Ni(BDC) structure. As shown in Fig. 2a, the absorption bands at 526 and 440 cm^{-1} are related to Ni–O and Co–O in $NiCo_2O_4$ nanoparticles. For the $NiCo_2O_4$@Ni(BDC) metal organic frameworks (Fig. 2b), characteristics peaks at 1678 and 733 cm^{-1} are assigned to the COO^- groups and absorption bands at 1508, 1423 and 1282 cm^{-1} are assigned to the C–H in the BDC ring, which clearly indicates the presence of Ni(BDC). Moreover, the stretching vibrations of Ni–O and Co–O are appeared at 548 and 428 cm^{-1} $NiCo_2O_4$@Ni(BDC) which these absorption bands represent the maintenance of the catalyst structure.

Fig. 3 shows the thermogravimetric analysis (TGA) curve of $NiCo_2O_4$@Ni(BDC) MOF. This nanocomposite exhibited two-stage weight loss in the TGA curve. The first weight loss of the $NiCo_2O_4$@Ni(BDC) occurred below 350 °C, indicating the removal of the occluded DMF and water from of the coordinating polymer frameworks. Below 350 °C, $NiCo_2O_4$@Ni(BDC) MOF exhibited weight loss of 23%–25% (wt) %. After the first weight loss, the TGA curve for the $NiCo_2O_4$@Ni(BDC) reached a plateau at temperatures of 380–430 °C. This illustrates the stability of the framework of the $NiCo_2O_4$@Ni(BDC) structure at the temperature of guest desorption and framework decomposition. The onset temperature of the second weight loss corresponded to the decomposition of organic species in the framework. The onset decomposition temperature at 450 °C was for $NiCo_2O_4$@Ni(BDC) in the second stage.

An EDX analysis was used to confirm the chemical purity of the $NiCo_2O_4$@Ni(BDC) sample as well as its stoichiometry. The results of Fig. 4 show that typical characteristic peaks of Ni, O, C and Co elements in the survey spectrum, verifying the existence of them in the $NiCo_2O_4$@Ni(BDC).

The XRD pattern of the $NiCo_2O_4$@Ni(BDC) nanocatalyst is shown in Fig. 5. The positions and relative diffraction peaks at around 2θ = 18.89°, 31.20°, 36.88°, 38.4°, 44.47°, 55.4°, 59.16°, 65.12°, and 76.9° for $NiCo_2O_4$@Ni(BDC) nanocrystallites confirm well with its structure [43].

The magnetic properties of $NiCo_2O_4$ and $NiCo_2O_4$@Ni(BDC) were studied by using vibration sample magnetometry (VSM) at room temperature. As shown in Fig. 6, the magnetic saturation (MS) values of the $NiCo_2O_4$ NPs is 5 emug^{-1}, and for the $NiCo_2O_4$@Ni(BDC) composite is 2 emug^{-1}. The slightly lower MS value of $NiCo_2O_4$@Ni(BDC) mainly results from the introduction of Ni(BDC). The presence of suitable magnetic properties allows $NiCo_2O_4$@Ni(BDC) to be completely, efficiently and quickly separated from the reaction mixture by an external magnet.

The structure of $NiCo_2O_4$@Ni(BDC) was studied by using the transmission electron microscopy (TEM) (Fig. 7). These images are an appropriate tool for determining the size and structure of particles. The TEM images show that small particles of 30–50 nm which confirmed by the FE-SEM images.

To optimize the reaction conditions, the condensation reaction of 4-chlorobenzaldehyde, malononitrile and guanidine hydrochloride was selected as a model study to examine the effect of the catalyst, solvent and reactants for the synthesis of 2,4-diamino pyrimidine-5-carbonitrile derivatives (Scheme 3).

To explore the effect of the solvent in the preparation of 2,4-diamino-6-(4-chlorophenyl)pyrimidine-5-carbonitrile, we preformed the model reaction using $NiCo_2O_4$@Ni(BDC) metal-organic framework under various solvents and also solvent-free conditions in different temperatures. The best results were obtained under reflux conditions. Fig. 8 shows that, the solvent has a great

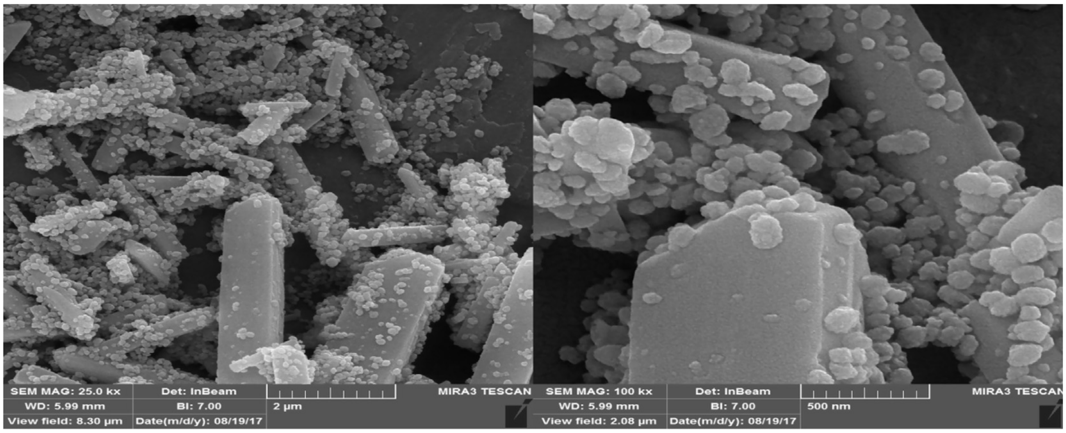


Fig. 1. FE-SEM images of $NiCo_2O_4$@Ni(BDC)

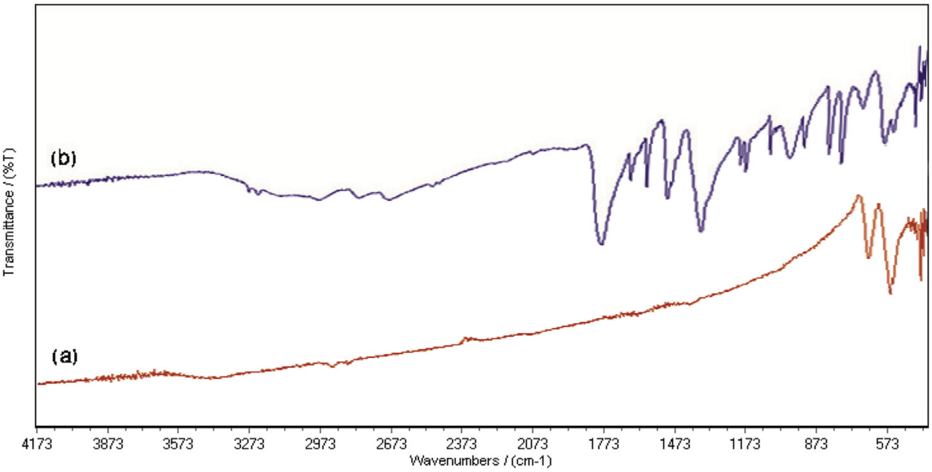


Fig. 2. FT-IR spectra of the $NiCo_2O_4$ (a) and $NiCo_2O_4$@Ni(BDC) (b).

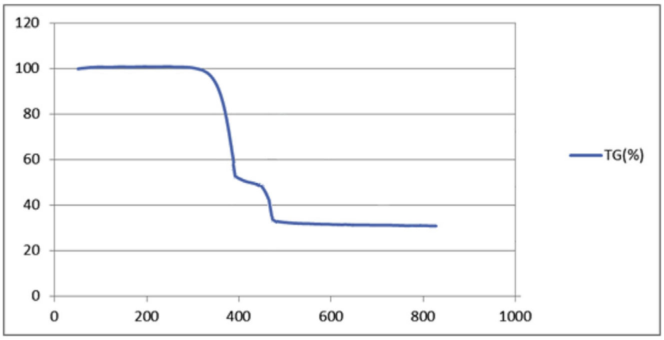


Fig. 3. TGA profile of the $NiCo_2O_4$@Ni(BDC)

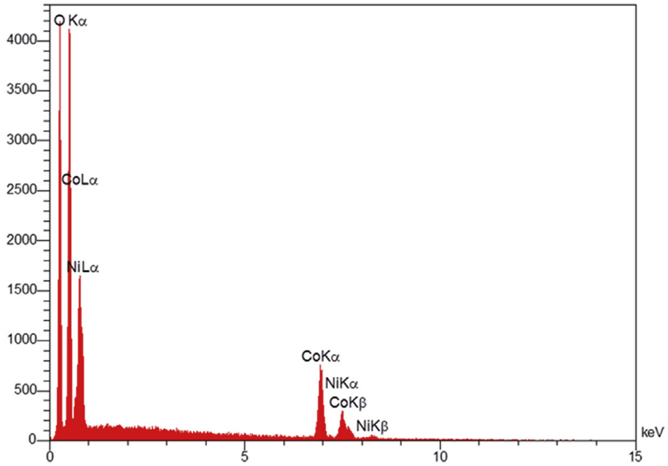


Fig. 4. The EDX spectrum of $NiCo_2O_4$@Ni(BDC)

effect on the accelerating of the reaction. The best results (93% Yield) were obtained in ethanol for this multi component reaction.

Then, to demonstrate the importance of the present research in compared to other catalysts, the model reaction was performed in the presence of various catalysts such as KOH, MgO, ZnO, CuI, NaOH, Ni(BDC) and $NiCo_2O_4$@Ni(BDC) using ethanol as solvent under reflux conditions. As shown in Fig. 9, the nanoporous metal-organic framework was the best catalysts with respect to reaction time and yield of the obtained product.

In continuation of our research, we ran the study model using different amounts of $NiCo_2O_4$@Ni(BDC) nanocatalyst under reflux conditions. As shown in Fig. 10, the optimum concentration of $NiCo_2O_4$@Ni(BDC) MNPs was obtained at 0.008 g. A value greater than 0.008 g does not change the time and yield of the reaction.

The scope and limitation of this research were examined when we obtained the optimized reaction conditions. A diversity of aldehydes were used in this study in presence of $NiCo_2O_4$@Ni(BDC) MNPs under reflux conditions. We observed that various aryl aldehydes could be introduced in high efficiency and produced high yields of products in high purity. The summarized results of Table 1 show that sterically hindered aromatic aldehydes need to longer reaction times in comparison with *p*-substituted aryl aldehydes. In addition, aromatic aldehydes bearing electron-withdrawing groups such as Br, and Cl in the *p*-position reacted very smoothly, in short reaction times and high yields while reactants with electron-releasing groups such as methoxy decreased both the rate of the reaction and the yield of the corresponding product.

A plausible mechanism for the preparation of 2,4-diamino-6-arylpyrimidine-5-carbonitriles in the presence of $NiCo_2O_4$@Ni(BDC) nanostructure as catalyst is depicted in Scheme 4 based on the obtained results together with previous published paper [35]. It is supposed that catalytically active site of $NiCo_2O_4$@Ni(BDC) NPs is Ni^{2+} that behaves as a Lewis acid and coordinates to the carbonyl groups of the aldehyde and double and triple bonds of the reactants and intermediates. Firstly, intermediate I is formed through the Knoevenagel reaction between malononitrile and aldehyde, and subsequently olefin II is produced by dehydration. Subsequent condensation of the guanidine 3 and olefin II followed by intramolecular cyclization and the oxidation which affords the corresponding products **4** (**a-k**).

3.1. Catalyst recovery

After completion of the reaction, the reaction mixture was dissolved in dichloromethane and then the catalyst was separated magnetically. The $NiCo_2O_4$@Ni(BDC) MNPs were washed three to four times with water and ethanol and dried at 60 °C for 12 h. The separated catalyst was used for six cycles with a slightly decreased in its activity (Fig. 11). In conclusion, we were able to express that a series of 2,4-diamino-6-arylpyrimidine-5-carbonitrile derivatives could be obtained by the catalytic application of the $NiCo_2O_4$@Ni(BDC) metal-organic frameworks under reflux conditions. In addition, to prove the stability and performance of the recovered

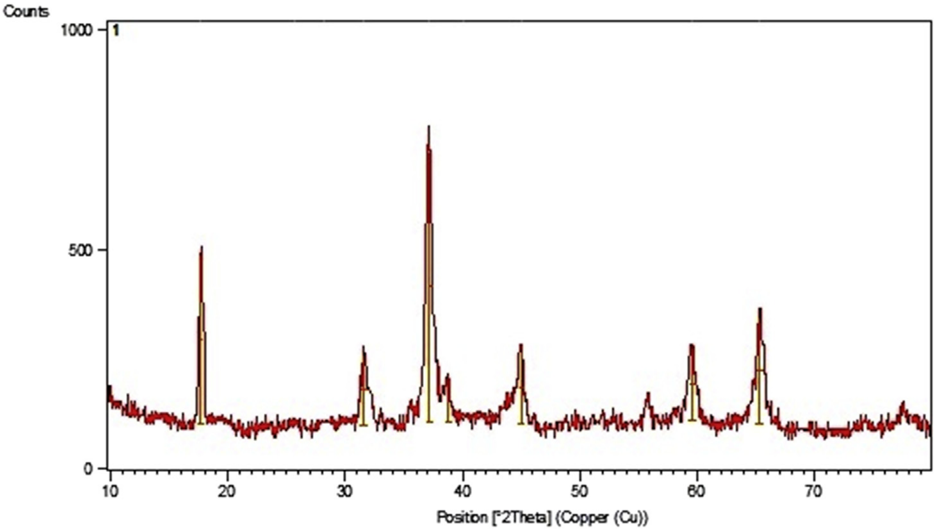


Fig. 5. XRD pattern of $NiCo_2O_4$@Ni(BDC)

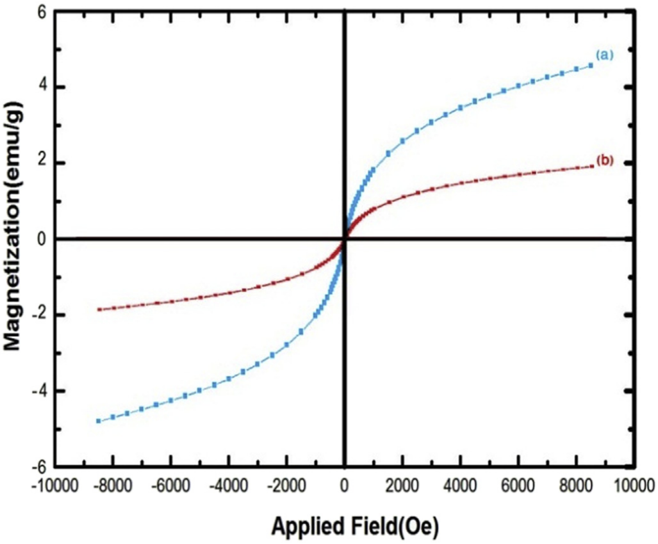
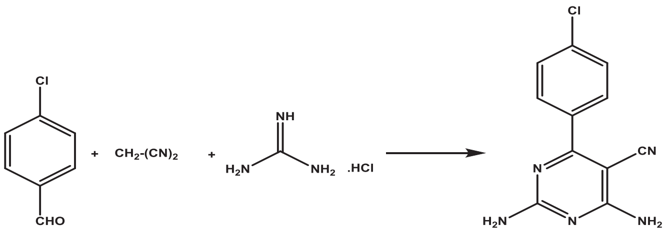


Fig. 6. Magnetization curves of the $NiCo_2O_4$(a) and $NiCo_2O_4$@Ni(BDC) (b).



Scheme 3. The model reaction for the synthesis of 2,4-diamino-6-(4-chlorophenyl) pyrimidine-5-carbonitrile.

nanocatalyst, the FT-IR spectrum and the XRD pattern of the recovered $NiCo_2O_4$@Ni(BDC) is given after six runs uses (Fig. 12). As seen, there is no significant difference between the XRD and FT-IR of fresh and recovered nanocomposite. These facts prove that the appearance and structure of $NiCo_2O_4$@Ni(BDC) catalyst remained intact in recycles and there was no considerable deformation or leaching after 6 runs.

In this research to confirm that we used a heterogeneous catalyst, hot filtration experiment was conducted using 4-chlorobenzaldehyde, malononitrile and guanidine hydrochloride

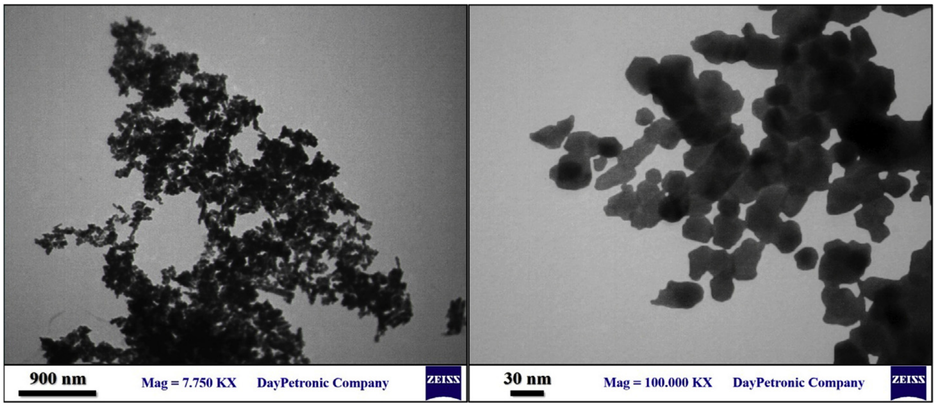


Fig. 7. TEM images of $NiCo_2O_4$@Ni(BDC)

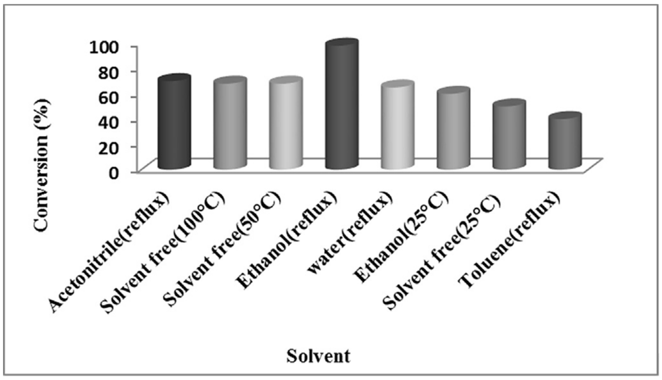


Fig. 8. Preparation of corresponding pyrimidine in different solvents.

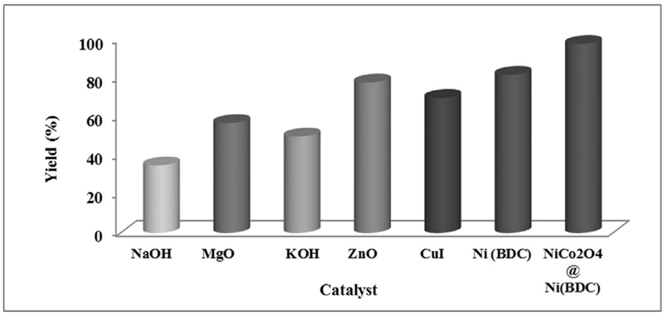


Fig. 9. The model reaction was carried out by various catalysts.

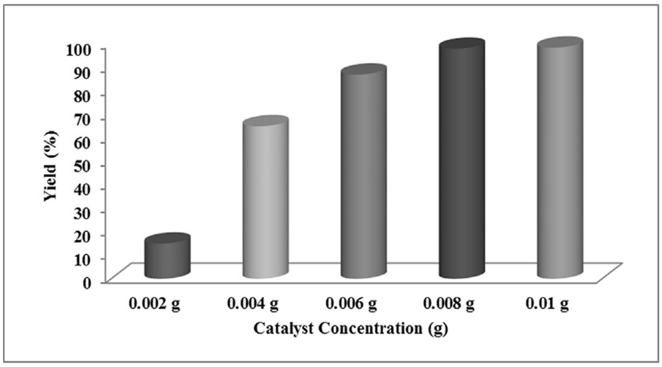
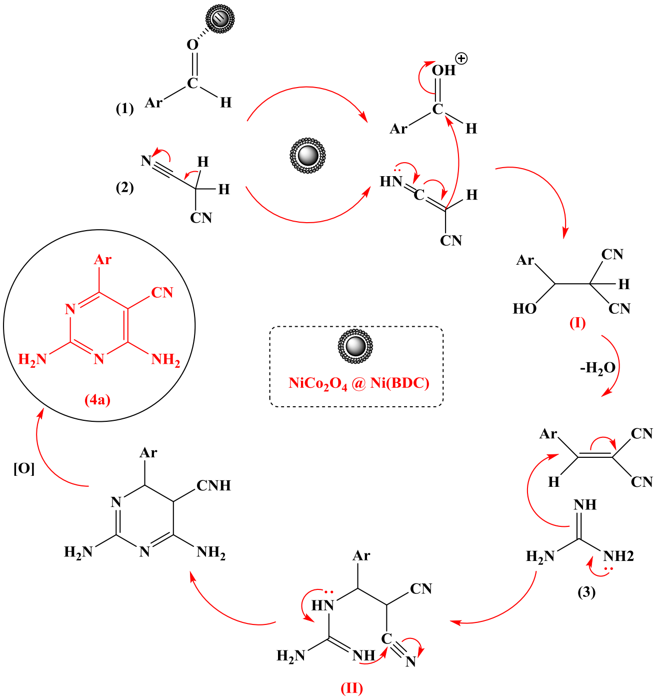


Fig. 10. The effect of different amounts of $NiCo_2O_4$@Ni(BDC) on the model reaction.



Scheme 4. Proposed reaction mechanism for preparation of 2,4-diamino-6-arylpyrimidine-5-carbonitriles using $NiCo_2O_4$@Ni(BDC) MNPs.

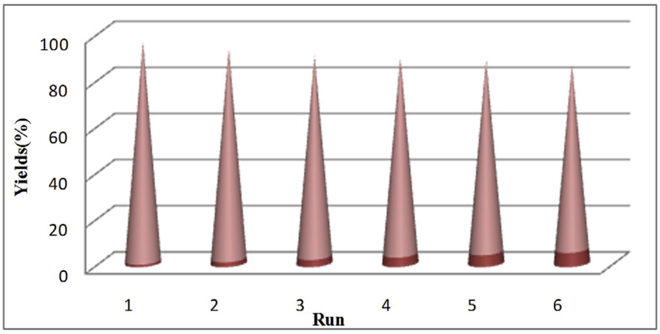


Fig. 11. Reusability of the $NiCo_2O_4$@Ni(BDC) nanocomposite.

Table 1
$NiCo_2O_4$@Ni(BDC)NPs catalyzed one-pot synthesis of 2,4-diamino-6-arylpyrimidine-5-carbonitrile derivatives[a].

Number	Ar	Product	T (min)	Yield[a] (%)	mp, °C	Mp.[Ref][b]
1	4-Cl-C_6H_4	4a	120	93	265–267	265–266[37]
2	4-MeO-C_6H_4	4b	150	88	235–237	236–238[37]
3	2-OH-C_6H_4	4c	180	82	170–172	172[36]
4	C_6H_5	4d	150	90	236–239	237–239[37]
5	4-F-C_6H_4	4e	120	90	237–239	235–237[37]
6	4- Br–C_6H	4f	120	90	258–260	260–262[37]
7	4-OH-C_6H_4	4g	150	91	258–260	260[36]
8	3-MeO-C_6H_4	4h	150	93	262–264	261–263[35]
9	4-Me-C_6H_4	4i	150	87	256–258	255–257[35]
10	4-CH$(CH_3)_2$–C_6H_4	4j	160	85	125–128	…. [c]
11	4-CN-C_6H_4	4k	150	91	127–130	…. [c]

[a] Reaction conditions: aldehyde (1 mmol), malononitrile (1.2 mmol), guanidine hydrochlorid(1 mmol) and $NiCo_2O_4$@Ni(BDC) NPs (0.008 g).
[b] Isolated yield.
[c] New compounds.

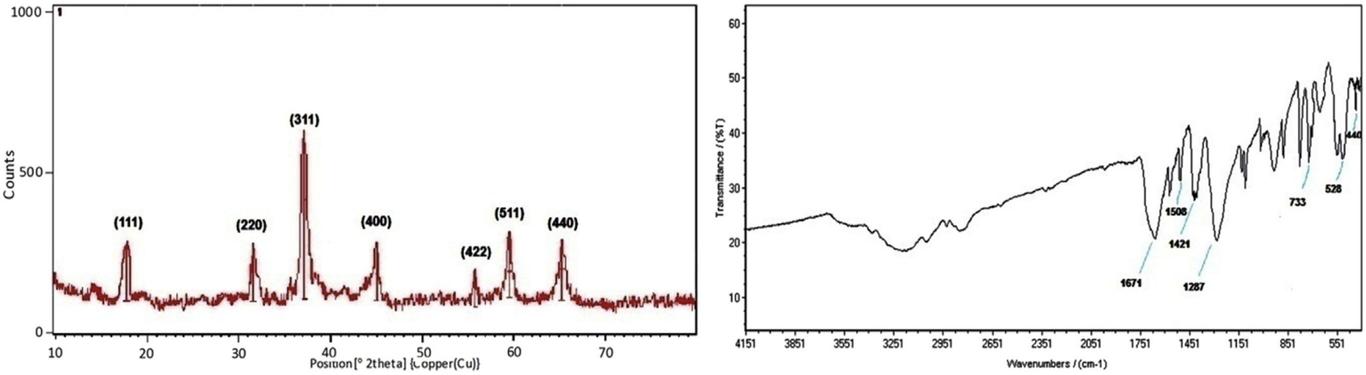


Fig. 12. The XRD pattern and FT-IR spectrum of the recovered $NiCo_2O_4$@Ni(BDC)

as a standard leaching experiment. The reaction was continued for about 20 min in the presence of $NiCo_2O_4$@Ni(BDC) MNPs in ethanol under reflux conditions. Then, the filtered reaction media was continued under stirring without a catalyst for 8 h; no product formation was observed, indicating that no homogeneous catalyst was involved. Furthermore, energy-dispersive X-ray spectroscopy (EDX) analysis of the filtrate (hot) discloses the absence of Ni and Co species in the filtrate.

4. Conclusion

We have developed a green and straightforward protocol for the synthesis of 2,4-diamino-6-arylpyrimidine-5-carbonitrile derivatives via a multi component condensation reactions in the presence of $NiCo_2O_4$@Ni(BDC) as a novel nanocatalyst under reflux conditions. The attractive features of this protocol are the simple procedure, cleaner reaction conditions, and the use of recyclable nanocatalyst. Satisfactory yields of products and easy workup make this a useful protocol for green synthesis of this class of compounds.

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