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# An efficient synthesis of pyrimido[4,5-*b*]quinoline and indenopyrido[2,3-*d*]pyrimidine derivatives in the presence of $Fe_3O_4$@nano-cellulose/Sb (V) as a bio-based magnetic nano-catalyst

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**KEYWORDS**
$Fe_3O_4$@nano-cellulose/Sb (V); Bio-based catalyst; Pyrimido[4,5-*b*]quinoline; Indenopyrido[2,3-*d*]pyrimidine; Solvent-free; Multi component reaction.

**Abstract.** In this study, an eco-friendly approach was introduced to synthetize pyrimido[4,5-*b*]quinolones and indenopyrido[2,3-*d*]pyrimidines. This synthesis was done via three-component coupling of: 6-amino-2-(methylthio) pyrimidin-4(3*H*)-one, 1,3-indanedione/dimedone, and aromatic aldehydes using $Fe_3O_4$@nano-cellulose/Sb (V) as a catalyst under the solvent-free condition at 70°C by electrical mortar-heater. The catalyst was separated from the reaction mixture by an external magnet and reused for subsequent reactions. The present procedure offers many advantages such as high yield, easy work-up, simple isolation of catalyst by external magnet, and high reusability. The structures of the obtained pyrimido[4,5-*b*]quinolones and indenopyrido[2,3-*d*]pyrimidines products were studied by FT-IR, $^1$H-NMR, and $^{13}$C-NMR spectroscopic data.



## 1. Introduction

Indenopyrido[2,3-*d*]pyrimidine (IPP) and pyrimido[4,5-*b*]quinolone (PQ) exhibit pharmacologic properties and are used in medicinal chemistry. Some of the pharmacological activities of these compounds are anticancer agents inhibiting tyrosine kinases [1–3], antitumor [4,5], antihistaminic [6], anti-inflammatory [7], and antibacterial [8–12]. Therefore, these heterocyclic compounds are highly regarded in research.

Numerous methods and various catalysts such as *p*-TSA [13], acetic acid [14], $InCl_3$ [15], 1,2-dimethyl-*N*-butanesulfonic acid imidazolium hydrogen sulfate ((DMBSI)$HSO_4$) [16], nano-$Fe_3O_4$@$SiO_2$-$SO_3H$ [17], ethylene glycol under sonication condition [18], $Fe_3O_4$@ urea/HITh-$SO_3H$ MNPs [19], $CoFe_2O_4$@$SiO_2$@$Si(CH_2)_3$ $NHCOOCH_2COOH$ [20], [$H_2$-DABCO] $[ClO_4]_2$ [21], nano-[$Fe_3O_4$@-$SiO_2$@R-$NHMe_2$] [$H_2PO_4$] [22], SBA-15/PrN($CH_2PO_3H_2$)$_2$ [23], En/ MIL-100 (Cr) [24], and $Fe_3O_4$@ NCs/Cu(II) [25] have been reported for the synthesis of IPP and PQ. Since the last few decades, green chemistry has been gaining much attention for chemists. Therefore, the preparation of environmentally friendly catalysts is still considered as an interesting challenge.

Previously, we have synthesized and characterized $Fe_3O_4$@nano-cellulose/Sb (V) (FNC-Sb (V)) as a new magnetic and bio-based nano-catalyst [26]. This study attempts to report an efficient and ecofriendly procedure for the synthesis of IPP and PQ derivatives *via* one-pot three-component condensation of 6-amino-2-(methylthio)pyrimidin-4(3*H*)-one (AMP), 1,3 indane-

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dione/dimedone, and various aldehydes in the presence of FNC-Sb (V).

## 2. Experimental

### 2.1. Materials and methods

All solvents and chemical materials were prepared from Merck, Aldrich, and Fluka chemical companies. FT-IR spectra were run on a Bruker, Equinox 55 spectrometer. A Bruker (DRX-400 Avanes) NMR was applied to record $^{1}$H-NMR and $^{13}$C-NMR spectra. Melting points were determined using a Buchi melting point B-540 B. V. CHI apparatus. Electrical mortar-heater was employed for grinding the reaction mixture at 70°C, purchased from Borna-Kherad Co., Iran, Yazd.

### 2.2. Synthesis of AMP

At Step 1, in a 100-ml round bottom vessel, 0.3 g of sodium metal was added to 20 ml of dry ethanol. Then, 1 g of thiourea and 1.38 ml of ethylcyanoacetate were charged to it. The resulting solution was refluxed for 4 hours. The obtained residue was dissolved in 30 ml of water and gradually added to NaOH (0.5 M) to obtain neutral pH. At this stage, the substance 6-amino-2-thioxo-2,3-dihydropyrimidin-4(1$H$)-one was synthesized.

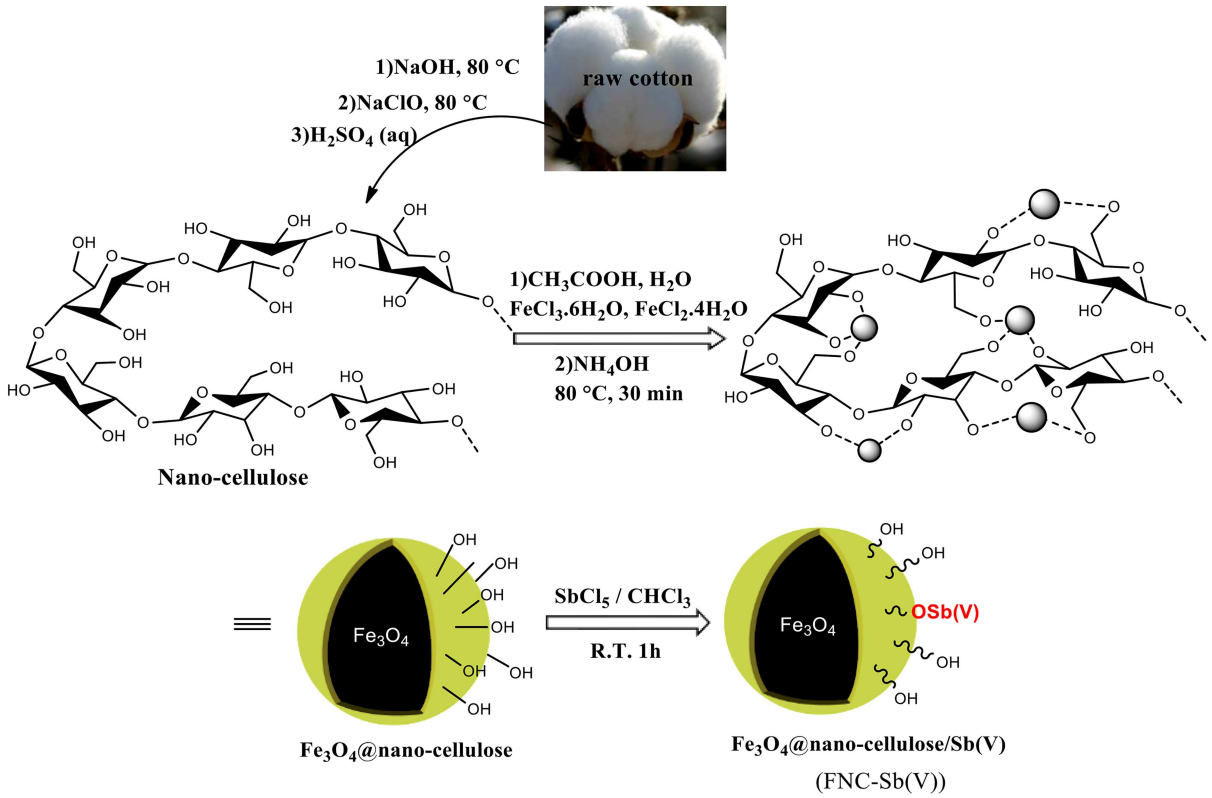
At Step 2, a mixture of 0.5 g of 6-amino-2-thioxo-2,3-dihydropyrimidin-4(1$H$)-o ne, 0.14 g NaOH, 35 ml of dry methanol, and 0.2 ml of methyliodide was heated under reflux condition. Then, the obtained product AMP was washed with water and dried at 110°C.

### 2.3. General procedure for synthesis of PQ and IPP derivatives

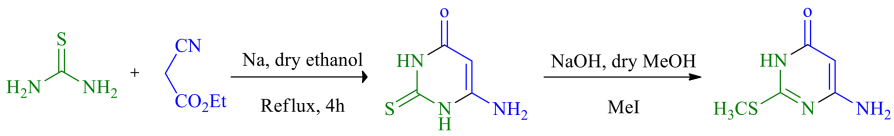
A mixture of AMP (0.156 g), indanedione/dimedone (0.146/0.140 g), aldehyde (0.106 g), and FNC-Sb (V) (0.03 g) was ground by an electrical mortar-heater at 70°C. The progress of reaction was monitored by TLC ($n$-Hexan:EtOAc, 8:2). After the completion of reaction, 5 ml of ethanol was added to the reaction mixture and the catalyst was separated by an external magnet. Through the cooling of the mixture, the product appeared as solid which was crystallized from EtOH:$H_2O$ (1:1).

## 3. Results and discussion

In this work, an efficient and environmentally benign protocol was developed for the synthesis of PQ and IPP derivatives using three-component reaction of AMP, 1,3 indanedione/dimedone, and various aromatic aldehydes in the presence of FNC-Sb (V). The steps of the synthesis of FNC-Sb (V) catalyst are shown in Scheme 1. The resulting catalyst was characterized by FT-IR, XRD, VSM, EDS, and TGA.




**Scheme 1.** Preparation of FNC-Sb (V).




**Scheme 2.** Synthesis of AMP (6-Amino-2-thioxo-2,3-dihydropyrimidine-4(1H)-one).

AMP as a very expensive substrate for preparation of PQ and IPP was synthesized through two stages (Scheme 2).

To investigate the catalytic activity of FNC-Sb (V), the reaction of AMP (0.156 g), dimedone (0.140 g), and 4-chlorobenzaldehyde (0.28 g) was performed as a model reaction under various conditions (Table 1). According to the results, the best condition is characterized by 0.03 g of FNC-Sb (V) under a solvent-free condition at 70°C by using electrical mortar-heater (Table 1, entry 8). In a reaction, without catalyst, a low yield of the product was achieved after a long reaction time (Table 1, entry 13), indicating the high efficiency of catalyst for this reaction.

According to the results of the model reaction, we have decided to synthesize PQ and IPP derivatives, results of which are shown in Table 2. The aromatic aldehydes with electron-withdrawing groups are more active than others. The structure of products was characterized by their melting points and spectral analyses such as FTIR and NMR.

In order to examine the reusability of FNC-Sb (V), it was separated by an external magnet, washed with chloroform, and dried at room temperature. The separated FNC-Sb (V) was reused four times without the considerable decrease of catalytic activity (Figure 1). The slight decrease in the catalytic activity may result from the obstruction of the active sites of the catalyst or partial secretion of antimony from it.

The catalytic activity of FNC-Sb (V) in the model reaction was compared with other reported catalysts (Table 3). According to the obtained data, use of FNC-

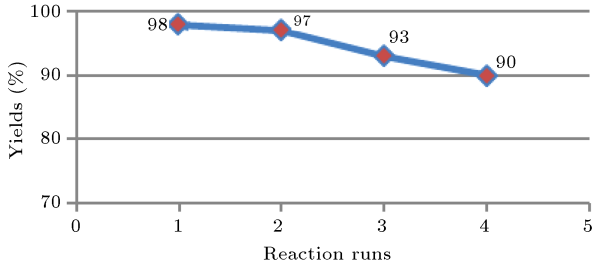
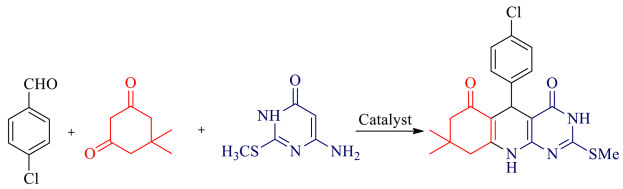



**Figure 1.** Catalyst reusability experiments.

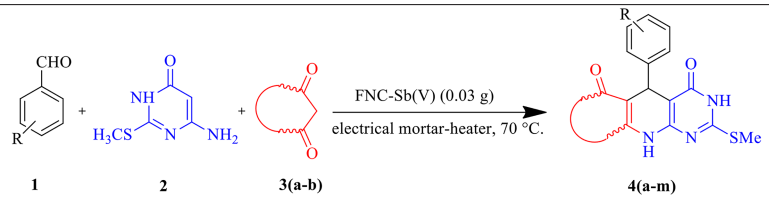
**Table 1.** The reaction of AMP (1 mmol), dimedone (1 mmol), and 4-chlorobenzaldehyde (1 mmol) under various conditions.




| Entry | Solvent | Catalyst (g)[a] | Condition | Time (min) | Yield (%)[b] |
|---|---|---|---|---|---|
| 1 | $H_2O$ | 0.03 | Reflux | 18 | 73 |
| 2 | $C_2H_5OH$ | 0.03 | Reflux | 12 | 80 |
| 3 | $CH_3OH$ | 0.03 | Reflux | 20 | 69 |
| 4 | $CH_3CN$ | 0.03 | Reflux | 20 | 72 |
| 5 | $C_2H_5OH$ | — | r.t.[c] | 30 | 25 |
| 6 | — | 0.03 | 90°C [d] | 6 | 93 |
| 7 | — | 0.03 | 80°C [d] | 6 | 98 |
| 8 | — | **0.03** | **70°C** [d] | **6** | **98** |
| 9 | — | 0.03 | 60°C [d] | 6 | 82 |
| 10 | — | 0.03 | 50°C [d] | 6 | 75 |
| 11 | — | 0.02 | 70°C [d] | 6 | 94 |
| 12 | — | 0.04 | 70°C [d] | 6 | 98 |
| 13 | — | — | 70°C [d] | 50 | 47 |

[a]: FNC-Sb (V); [b]: Isolated yield; [c]: Room temperature; [d]: Electrical mortar-heater.

**Table 2.** Synthesis of PQ and IPP derivatives **(4a-m)** in the presence of FNC-Sb (V) under the solvent-free condition at 70°C in electrical mortar-heater.




1 2 3(a-b) 4(a-m)

| Entry | *R* | 3 a or b | Product | Time (min) | Yield (%) | M.P.°C [Ref.] Found |
|---|---|---|---|---|---|---|
| 1 | 4-Cl- | a | 4a | 6 | 98 | > 300 [17] |
| 2 | 4-$NO_2$- | a | 4b | 4 | 98 | > 300 [27] |
| 3 | H- | a | 4c | 6 | 94 | > 300 [27] |
| 4 | 2,4-$(OMe)_2$- | a | 4d | 6 | 95 | > 300 [27] |
| 5 | 3-$NO_2$- | a | 4e | 5 | 98 | > 300 [27] |
| 6 | 2,4-$(Cl)_2$- | a | 4f | 4 | 92 | > 300 [27] |
| 7 | 4-OMe- | a | 4g | 5 | 92 | > 300 [17] |
| 8 | 3,4-$(OH)_2$- | a | 4h | 5 | 95 | > 300 [27] |
| 9 | 4-OMe- | b | 4i | 6 | 90 | > 300 [27] |
| 10 | 4-Cl- | b | 4j | 7 | 98 | > 300 [27] |
| 11 | 2,4-$(Cl)_2$- | b | 4k | 5 | 92 | > 300 [27] |
| 12 | 4-Me- | b | 4l | 6 | 80 | > 300 [27] |
| 13 | 4-OH-3-OMe- | b | 4m | 6 | 89 | > 300 [27] |

Note: a: Dimidone; b: 1,3-indanedione

**Table 3.** Comparison of catalytic performances of FNC-Sb (V) versus some other catalysts for the synthesis of PQ.

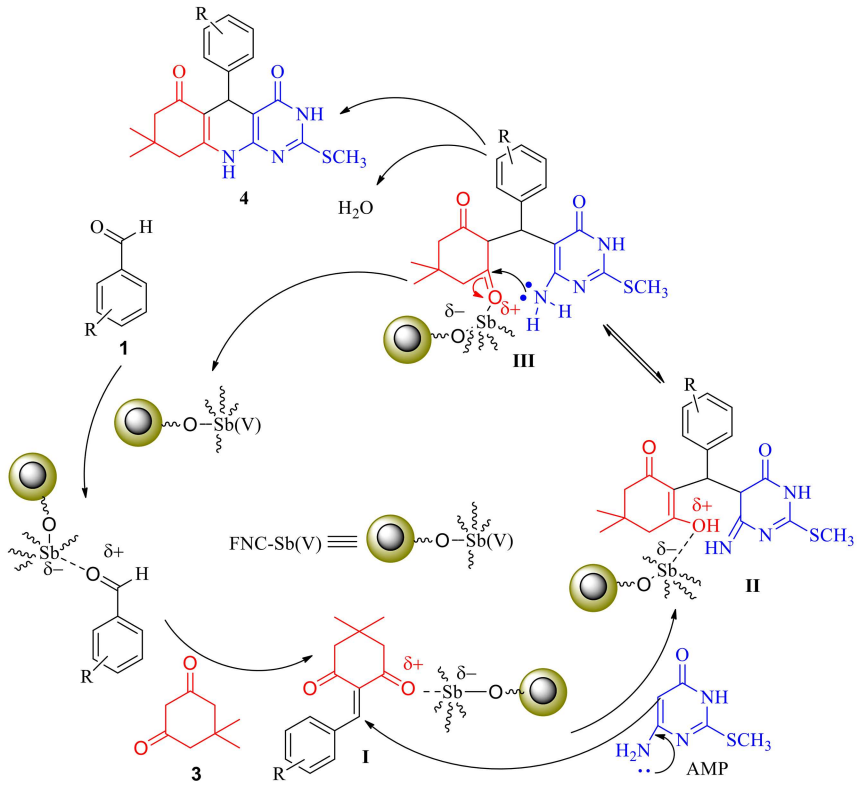
| Entry | Solvent | Catalyst | Tem. (°C) | Time (min) | Yield (%)[a] | [Ref.] |
|---|---|---|---|---|---|---|
| 1 | $H_2O$:EtOH | SBA-Pr-$SO_3H$[b] | 90 | 60 | 85 | [28] |
| 2 | $H_2O$ | $InCl_3$ | 90 | 60 | 91 | [15] |
| 3 | $H_2O$ | P-TSA[c] | 90 | 150 | 89 | [13] |
| 4 | $H_2O$ | $Fe_3O_4$@Cellulose-$SO_3H$ | 80 | 20 | 90 | [29] |
| 5 | $H_2O$ | $Fe_3O_4$@$SiO_2$-$SO_3H$ | 70 | 25 | 92 | [17] |
| 6 | $H_2O$ | [Bmim]Br[d] | 95 | 210 | 90 | [30] |
| 7 | — | $Fe_3O_4$@NCs[e] | 70 | 24 | 51 | — |
| **8** | — | **FNC-Sb (V)** | **70**[f] | **6** | **98** | **[This work]** |

[a]: Isolated yield, [b]: Sulfonic acid functionalized SBA-15, [c]: *p*-Toluenesulfonic acid (PTSA),

[d]: Ionic liquid 1-*n*-butyl-3-methylimidazoliumbromide, [e]: $Fe_3O_4$@Nano-cellulose, [f]: By Electrical Mortar-Heater

Sb (V) promoted the reaction in a shorter reaction time with higher yields.

The proposed mechanism for the synthesis of PQ **(4)** is shown in Scheme 3. The Lewis acid moiety of catalyst (Sb (V)) increases the electrophilic activity of carbonyl group in aldehyde and dimedone. In an acceptable mechanism, it is assumed that the reaction may continue at first through the Knoevenagel condensation between aldehydes and dimedone to form intermediate **I**. Next, Michael addition of AMP to




**Scheme 3.** Proposed mechanism for the synthesis of PQ derivatives.

intermediate **I** affords **II**. Intermediate **II** converts into **III** after tautomerization. Then, Intermediate **III** converts via cyclization to Product **4**.

## 4. Conclusion

In summary, a simple multi-component procedure was introduced for the facile synthesis of PQ and IPP derivatives using FNC-Sb (V) as a bio-based magnetic nano-catalyst with high efficiency. PQ and IPP derivatives were prepared through the one-pot three-component reaction of AMP, 1,3-indanedione/dimedone, and various aldehydes under the solvent-free condition at 70°C by electrical mortar-heater. This protocol includes many advantages such as high atom-economy, mild reaction conditions, and use of inexpensive reusable heterogeneous catalyst.

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