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Magnetically Recoverable $Fe_{0.02}Zn_{0.95-x}Cr_{0.05}O$ Iron doped Catalyst for Synthesis of Dihydropyrimidones, Thiones and their Derivatives

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A new technique for the impregnation of magnetic based catalyst component onto the organic molecules are widely discussed. Due to its benefits of high surface area, separation simplicity, recyclability and limited leaching of catalytic active ingredients from the catalyst, in the present work, iron-doped $Fe_{0.02}Zn_{0.95-x}Cr_{0.05}O$ heterogeneous catalyst was applied in the synthesis of dihydropyrimidones, thiones and their derivatives. The synthesized compounds were elucidated by their IR, ^{1}H NMR and ^{13}C NMR and LC-MS analysis.

Keywords: Dihydropyrimidinone, One pot synthesis, Biginelli reaction, Iron doped catalyst, Cyclocondensation.

INTRODUCTION

One of the most prominent heterocyclic compounds, dihydropyrimidin-2(1*H*)-ones/thiones (DHPMs), possesses several biological features [1,2]. Biginelli reaction has drawn increasing attention due to the significance of multicomponent reactions (MCRs) in the combinatorial chemistry and the intriguing the pharmacological properties associated to DHPMs structures. As a result, its scope has been significantly expanded by variation of all three building blocks, and several modified and improved procedures have been reported [3,4]. The pharmacological activities of DHPMs include antiviral, anticancer, antibacterial and anti-inflammatory activities [5-8].

The typical Biginelli synthesis of substituted aromatic and aliphatic aldehydes requires a lengthy reaction time and also the yield is poor [9]. Although one pot techniques for the Biginelli reaction are simpler, multi-step synthesis is another method for the reaction with higher yields [10]. Besides this, there are lots of methods reported in literature for the synthesis of dihydropyrimidinones (DHPMs) [11-15].

The majority of the presented procedures and their standards have advantages, but there are also some disadvantages, like long reaction time and high reagent cost. Multicomponent one-pot synthesis is one such method for the synthesis of DHPMs [16-20]. For green chemistry purposes, chemists also demand catalysts that produce high activity and high efficiency catalysts to separate and recover from reaction mixtures, with low energy consumption, long life and excellent selectivity [21,22]. Magnetic nanocatalysts also have these properties like easy separation, reusability, high catalytic activity and high chemical stability in different solvents [23-26].

This encourage us for synthesis of dihydropyrimidones, thiones and their derivatives by cyclocondensation reaction *via* Biginelli reaction using magnetically recoverable iron doped catalyst $Fe_{0.02}Zn_{0.95-x}Cr_{0.05}O$ (synthesized by sol-gel process) [27]. The structures of the compounds were confirmed by IR and NMR (^{1}H and ^{13}C) and LCMS data.

EXPERIMENTAL

Using silica gel-percolated plates (TLC), the reaction was monitored and the purity of product was also determined. The melting point was determined by using electro-thermal micro melting point equipment and are uncorrected. The IR spectra were recorded using a spectrometer. The ^{1}H NMR was recorded in $CDCl_3$ on a Bruker spectrometer ^{13}C spectra recorded by using a spectrometer.

The catalyst $Fe_{0.02}Zn_{0.95-x}Cr_{0.05}O$ (x = 0.0, 0.02, 0.04, 0.06, 0.08, 0.10) was synthesized by sol-gel method as per literature method [27] and used for the model reaction of benzaldehyde,

TABLE-1
OPTIMIZATION CONDITION FOR MODEL REACTION BETWEEN BENZALDEHYDE, ETHYL ACETO ACETATE AND UREA BY USING CATALYST $Fe_{0.02}Zn_{0.95-x}Cr_{0.05}O$ (WITH x = 0.0, 0.02, 0.04, 0.06, 0.08, 0.10)

Entry	Catalyst	Catalyst loading (g)	Solvent	Time (h)	Yield (%)
1	$Fe_{0.02}Zn_{0.95}Cr_{0.05}O$	0.0631	Water	–	–
			Ethanol	10	17
			Heptane-toluene	6.5	32
2	$Fe_{0.02}Zn_{0.93}Cr_{0.05}O$	0.0630	Water	–	–
			Ethanol	10	21
			Heptane-toluene	6	37
3	$Fe_{0.02}Zn_{0.91}Cr_{0.05}O$	0.0630	Water	15	21
			Ethanol	9	19
			Heptane-toluene	5	45
4	$Fe_{0.02}Zn_{0.89}Cr_{0.05}O$	0.0628	Water	13	25
			Ethanol	7	26
			Heptane-toluene	4.5	54
5	$Fe_{0.02}Zn_{0.87}Cr_{0.05}O$	0.0628	Water	12	27
			Ethanol	6.5	34
			Heptane-toluene	4.2	65
6	$Fe_{0.02}Zn_{0.85}Cr_{0.05}O$	0.0627	Water	10	36
			Ethanol	6.2	42
			Heptane- toluene	4	85

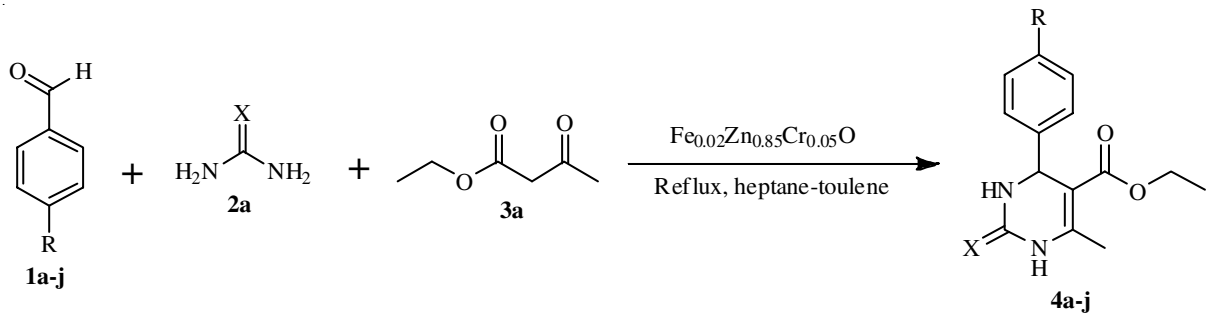
ethyl acetoacetate and urea in different solvents. It was clear from Table-1 that using iron doped catalyst, the Biginelli reaction proceeds in heptane-toluene medium [28] and gave the best results with catalyst $Fe_{0.02}Zn_{0.85}Cr_{0.05}O$ in accordance to its yield and time requirements.

General synthesis procedure: After obtaining the optimized conditions, the reaction for the synthesis of dihydropyrimidinone, thione and its derivatives (**4a-j**) was carried out in heptane-toluene solvent under reflux conditions. A reaction of aromatic aldehyde (1 mmol), ethyl acetoacetate (1.5 mmol), urea/thiourea (1.5 mmol) and $Fe_{0.02}Zn_{0.93}Cr_{0.05}O$ catalyst (1 mmol) was carried out by refluxing the mixture in heptane-toluene medium (5 mL, 1:1) with magnetic stirring for 3 h (**Scheme-I**). TLC confirmed the completion of the reaction. The obtained product was cooled to room temperature and poured into 15 mL of cold water. The resulting solid was filtered and successively washed with H_2O and petroleum ether-ethyl acetate (10:2:20 mL). The crude product was then purified by crystallization from ethanol. The structures of obtained **4a-j** products were confirmed by using IR, ^{1}H NMR and ^{13}C NMR and LCMS data with standards.

5-(Ethoxycarbonyl)-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1*H*)-thione (4a): Yield: 85%, time: 4 h, m.p.: 209 ºC [Lit. 207-209] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3325, 3207 (N-H *str.*), 1710 (C=O *str.*) 1205 (C=S *str.*); ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.06 (t, 3H, OCH_2CH_3), 2.23 (s, 3H, CH_3), 3.95 (q, 2H, OCH_2CH_3), 5.17 (s, 1H, -CH), 7.18 (m, 5H, Ar-H), 7.71 (s, 1H, NH), 9.31 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 14.29, 17.65, 54.74, 59.81, 101.33, 126.84, 127.76, 128.55, 143.88, 145.05, 165.54, 174.69 ppm; ESI-MS: m/z = 277.44 $[M+H]^+$; found 276.35.

5-(Ethoxycarbonyl)-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1*H*)-one (4b): Yield: 90%, time: 3.8 h, m.p.: 203 ºC [Lit. 202-203] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3319, 3209 (N-H *str.*), 1698 and 1706 (C=O *str.*); ^{1}H NMR (400 MHz, $CDCl_3$-d_6): δ 1.08 (t, 3H, OCH_2CH_3), 2.44 (s, 3H, CH_3), 3.99-4.04 (q, 2H, OCH_2CH_3), 5.08 (s, 1H, -CH), 7.20-7.26 (m, 5H, Ar-H), 7.39 (s, 1H, NH), 8.09 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 13.90, 18.40, 52.16, 60, 98.93, 127.54, 129.30, 129.81, 132.59, 139.43, 148.29, 152.86, 165.29 ppm.

5-(Ethoxycarbonyl)-6-methyl-4-(4-methylphenyl)-3,4-dihydropyrimidin-2(1*H*)-one (4c): Yield: 93%, time: 3.4 h, m.p.: 221 ºC [Lit. 221] [Ref. 30]. IR (KBr, ν_{max}, cm^{-1}): 3157 and 3212 (N-H *str.*), 1719 and 1645 (C=O *str.*); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.19 (t, 3H CH_3), 2.33 (s, 6H CH_3), 4.10 (q, 2H CH_2), 5.36 (s, 1H CH), 5.64 (s, 1H, NH) 7.10-7.26 (m, 4H



4a: R = H, X = S; **4b:** R = H, X = O; **4c:** R = 4-CH_3, X = O; **4d:** R = 2-Cl, X = O; **4e:** R = 2-Cl, X = S; **4f:** R = 4-NO_2, X = S; **4g:** R = 3-OH, X = S; **4h:** R = 4-CH_3O, X = O; **4i:** R = 4-CH_3O, X = S; **4j:** R = 4NO_2, X = O

Scheme-I

ArH), 7.89 (s, 1H NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 14.13, 18.54, 21.06, 55.21, 59.84, 101.16, 126.49, 129.20, 137.40, 141.13, 146.57, 153.26, 165.78 ppm.

5-(Ethoxycarbonyl)-6-methyl-4-(2-chlorophenyl)-3,4-dihydropyrimidin-2(1*H*)-one (4d): Yield: 86%, time: 3.2 h, m.p.: 216 ºC [Lit. 215-217] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3320, 3211 (N-H *str.*), 1690 and 1660 (C=O stretch.); ^{1}H NMR ($CDCl_3$) 1.07 (t, 3H, OCH_2CH_3), 2.49 (s, 3H, CH_3), 4.08 (q, 2H, OCH2), 5.58 (s, 1H), 7.27-7.42 (m, 4H, Ar-H), 8.08 (s, 1H, NH), 9.35 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): 13.96, 18.32, 52.23, 60.01, 98.96, 123.64, 127.99, 129.32, 132.59, 139.42, 148.44, 153.01, 165.22 ppm.

5-(Ethoxycarbonyl)-6-methyl-4-(2-chlorophenyl)-3,4-dihydropyrimidin-2(1*H*)-thione (4e): Yield: 82%, time: 3.6 h, m.p.: 164 ºC [Lit. 164-165] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3210 and 3118 (N-H *str.*), 1710 (C=) *str.*), 1213 (C=S *str.*); ^{1}H NMR ($CDCl_3$) 1.10 (t, 3H, *J* = 7.1 Hz, OCH_2CH_3), 2.57 (s, 3H, CH_3), 4.10 (q, 2H, *J* = 7.1 Hz, OCH_2), 5.98 (s, 1H), 5.88 (s, 1H), 7.27-7.42 (m, 4H, Ar-H), 8.10 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): 13.99, 18.00, 52.63, 60.12, 100.68, 127.58, 128.34, 129.82, 129.99, 132.72, 138.44, 144.32, 164.53, 173.98 ppm.

5-Ethoxycarbonyl-6-methyl-4-(4-chlorophenyl)-3,4-dihydropyrimidin-2(1*H*)-one (4f): Yield: 78%, time: 3.8 h, m.p.: 110 ºC [Lit. 109-111] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3227 and 3298 (N-H *str.*), 1620 and 1720 (C=O *str.*); ^{1}H NMR (400 MHz, DMSO-d_6) δ: 1.23 (t, 3H, OCH_2CH_3), 2.23 (s, 3H, CH_3), 3.92 (q, 2H, OCH_2CH_3), 5.72 (s, 1H, CH), 7.18 (d, 2H, ArH), 7.58 (d, 2H, Ar-H), 7.93 (s, 1H, NH), 8.89 (s, 1H, NH), ppm; ^{13}C NMR (100 MHz, DMSO-d_6) δ: 14.18, 18.62, 53.99, 63.10, 100.11, 127.62, 128.35, 129.92, 132.65, 138.33, 143.9, 164.70, 175.98 ppm.

5-Ethoxycarbonyl-6-methyl-4-(4-hydroxyphenyl)-3,4-dihydropyrimidin-2(1*H*)-one (4g): Yield: 77%, time: 3.1 h, m.p.: 185 ºC [Lit. 184-185] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3312 and 3206 (N-H *str.*), 1658 and 1698 (C=O *str.*); ^{1}H NMR (400 MHz, DMSO-d_6) δ: 1.09 (t, 3H, OCH_2CH_3), 2.19 (s, 3H, CH_3), 3.88 (q, 2H, OCH_2CH_3), 5.13 (s, 1H, CH), 7.18 (d, 2H, ArH), 7.38 (d, 2H, Ar-H), 7.91 (s, 1H, NH), 9.05 (s, 1H, NH), 9.32 (s, 1H, OH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6) δ: 13.9, 17.1, 53.99, 60.0, 100.12, 121.9, 125.99, 130.1, 143.3, 145.9, 154.99, 165.0 ppm.

5-(Ethoxycarbonyl)-6-methyl-4-(4-methoxyphenyl)-3,4-dihydropyrimidin-2(1*H*)-one (4h): Yield: 81%, time: 3 h, m.p.: 200 ºC [Lit. 200-201] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3323 and 3218 (N-H *str.*), 1702 and 1682 (C=O *str.*); ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.17 (t, 3H, OCH_2CH_3), 2.34 (s, 3H, CH_3), 3.98 (s, 3H, -OCH_3), 4.12 (q, 2H, OCH_2CH_3), 5.41 (d, 1H, *J* = 2.15 -CH), 6.98 (d, 2H, Ar-H), 7.31 (d, 2H, Ar-H), 7.88 (s, 1H, NH), 9.35 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 14.41, 18.76, 54.98, 55.44, 60.25, 100.51, 114.26, 127.68, 136.98, 146.30, 153.55, 159.30, 165.44 ppm.

5-(Ethoxycarbonyl)-4-(4-methoxyphenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-thione (4i): Yield: 83%, time: 3 h, m.p.: 150 ºC [Lit. 150-151] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3329 and 3206 (N-H *str.*), 1710 (C=O *str.*), 1207 (C=S *str.*); ^{1}H NMR (DMSO-d_6): 1.08 (t, 3H, CH_3), 2.28 (s, 3H, CH_3), 4.12 (s, 3H, CH_3), 4.14 (q, 2H, –OCH_2CH_3), 5.38 (d 1H, -CH), 6.7 (d, 2H, Ar), 7.12 (d, 2H, Ar), 7.33 (s, 1H), 7.88 (s, 1H NH) 9.42 (s, 1NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 14.38, 18.10, 55.20, 55.48, 60.38, 101.23, 114.32, 127.69, 137.12, 147.10, 159.40, 165.58, 182.42 ppm.

5-(Ethoxycarbonyl)-4-(4-nitrophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4j): Yield: 85%, time: 3.3 h, m.p.: 208 ºC [Lit. 208-209] [Ref. 29]. IR (KBr, ν_{max}, cm^{-1}): 3229 (N-H *str.*), 1738 and 1668 (C=O *str.*); ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.20 (t, 3H, OCH_2CH_3), 2.32 (s, 3H, CH_3), 4.12 (q, 2H, OCH_2CH_3), 5.69 (d, 1H, -CH), 7.45 (d, 2H, Ar-H), 7.63 (d, 2H, Ar-H), 7.88 (s, 1H, NH), 9.12 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 14.18, 18.42, 54.98, 60.21, 100.01, 120.12, 127.98, 138.28, 152.34, 153.32, 158.98, 165.76 ppm.

RESULTS AND DISCUSSION

The magnetically recoverable and reusable (1 mmol) $Fe_{0.02}Zn_{0.95-x}Cr_{0.05}O$ (x = 0.0, 0.02, 0.04, 0.06, 0.08, 0.10) ferrite nanocatalysts were used for model reaction between (1 mmol) benzaldehyde, (1.5 mmol) thiourea and (1.5 mmol) ethyl acetoacetate to synthesize dihydropyrimidinone (**4a**) in different solvents (Table-1). It was clear that the reaction will proceed in heptane-toulene medium gave better results with catalyst $Fe_{0.02}Zn_{0.85}Cr_{0.05}O$ in accordance to its yield and time requirements. As soon as the reaction was completed, the reaction mixture was removed from the flask. The catalyst was repeatedly cleaned with acetone after being drawn by a magnet to the bottom of flask. The reaction was then repeated for the following cycle with the addition of fresh substrate to the flask. $Fe_{0.02}Zn_{0.85}Cr_{0.05}O$ can be used as a catalyst four to five times without significantly decreasing catalytic activity. The number of cycles and **4a** product yield are listed in Table-2.

TABLE-2
RECYCLABILITY OF CATALYST

Catalyst	Number of cycles	Yield (%)
$Fe_{0.02}Zn_{0.85}Cr_{0.05}O$	First	85
$Fe_{0.02}Zn_{0.85}Cr_{0.05}O$	Second	83
$Fe_{0.02}Zn_{0.85}Cr_{0.05}O$	Third	80
$Fe_{0.02}Zn_{0.85}Cr_{0.05}O$	Fourth	79
$Fe_{0.02}Zn_{0.85}Cr_{0.05}O$	Fifth	78

Spectral analysis of the obtained product (**4a**) shows IR spectrum of absorptions at 1710cm^{-1} for carbonyl group and broad absorption band at 3325 and 3207 cm^{-1} for N-H groups. The ^{1}H NMR spectrum registered two singlet's at δ 7.71 and 9.31 for two N-H groups and another singlet at δ 2.23 cm^{-1} of three protons for methyl group and one singlet of one proton for (-CH) group. One triplet of three protons at δ 1.06 ppm and one quartet of two protons at δ 3.95 ppm for (-OCH_2CH_3) group. Multiplate in region of δ 7.18 ppm shows five aromatic protons. All these spectral data confirms that compound **4a** is 5-(ethoxycarbonyl)-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1*H*)-thione.

Conclusion

The used catalyst $Fe_{0.02}Zn_{0.95-x}Cr_{0.05}O$ proved as an efficient catalyst for the synthesis of dihydropyrimidones, thiones and

their derivatives. This procedure offers advantages like short reaction time, recyclability of catalyst and also high product yield. So, this method is easy, useful and efficient for synthesis of dihydropyrimidones and its derivatives.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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