Iranian Journal of Organic Chemistry Vol. 12, No. 2 (2020) 2831-2837

Synthesis and characterization of $SbCl_5$/nano-sawdust as a novel nano catalyst for synthesis of pyrano[2,3-*d*]pyrimidines

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Received: February 2020; Revised: March 2020; Accepted: March 2020

Abstract: SbCl5/nano-sawdust has been prepared from sawdust and SbCl5 in ethanol as solvent. SbCl5/nano-sawdust has been characterized by TEM, TGA and FT-IR and then SbCl5/nano-sawdust has been introduced as a novel andefficient catalyst for thethree component synthesis of pyrano[2,3-d]pyrimidinesfrom the simple one-pot reaction betweenbarbituric acid, ethylcyanoacetate and aromatic aldehydes in EtOH/H_2O(1:1) as solvent atroom temperature. The products are been characterized by melting points, IR,1H and 13C NMR spectroscopy.This method was compared with other catalysts reported in literature cleanliness,an eco-friendly catalyst, simple methodology, short time, and excellent yields of products are some advantages of this method. Sawdust is arenewable, natural, cheap and readily available source of cellulose with OH groups. So in this work, we have investigated the synthesis of bio-based catalyst by bonding Lewis acids to OH groups of D-glucose units in sawdust. And we have examined its catalytic behaviour for the synthesis ofpyrano[2,3-d]pyrimidines.

Keywords: SbCl5, Sawdust, Pyrano [2,3-d]pyrimidines, Nanocatalyst.

Introduction

Multicomponent reaction (MCR) is a synthetic methodology in which three or more reactants come together to form a new product [1]. The synthesis of pyrano [2,3-d]pyrimidines have received considerable attention due to their interesting pharmacological properties [2].They are commonly synthesized via a reaction of barbituric acids, aromatic aldehydes and malononitrile in the presence of a catalyst such as ZnO nano particles[3] , Mn/ZrO2[4], Cu(I)[4], ZnFe2O4 nanoparticles[5], Zn[(L)proline]2[6], H14[NaP5W30O110] [7] and Al-HMS-20[8]. Performing the organic reactions under green conditions such as use recyclable catalysts and non-toxic solvents in reactions has become critically important [9].

Sawdust is an available source of cellulose that composed of a long chain of glucose molecules. Antimony pentachloride (SbCl5) is a Lewis acid that used as catalyst in organic synthesis [10]. SbCl5 is stored away from heat and moisture, since in the presence of moisture, it releases hydrogen chloride gas. But SbCl5/nano-sawdust is stable and non-hydrolysable. In this work SbCl5/nano-sawdust has been synthesized and this catalyst has been used for the synthesis of pyrano[2,3-d]pyrimidines.

Result and Discussion

In this work, we have examined the catalytic activity of SbCl5/nano sawdust in the synthesis of pyrano [2,3-d] pyrimidine derivatives. The one pot, three component reactions of benzaldehyde1a, ethylcyanoacetate **2** and barbituric acid **3** was selected as model reaction for evaluation of catalytic activity and obtaining optimal conditions. This reaction proceeded smoothly in the presence of low loading of catalyst (0.02 g)

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inEtOH/H_2O (1:1) as solvent system of choice to give the expected product **4a** in high yield, in a relatively short reaction time Table **1**.

The size and morphology of SbCl5/nano-sawdust was determined by TEM (Figure **1**). The results show the size of SbCl5/nano-sawdust catalysis is less than30nm.

Table 1. Optimization of the reaction conditions for synthesis of 4a.

Entry	Catalyst (amount)	Solvent/Condition	Time(min)	Yield
1	$SbCl_5$/nano-sawdust (0.02 g)	CH_2Cl_2/ r.t	45	Trace
2	$SbCl_5$/nano-sawdust (0.02 g)	H_2O:EtOH/ r.t	45	92
3	$SbCl_5$/nano-sawdust (0.02 g)	CH_3CN/ r.t	45	38
4	$SbCl_5$/nano-sawdust (0.02 g)	DMF/ r.t	45	47
5	$SbCl_5$/nano-sawdust (0.02 g)	H_2O/ r.t	45	83
6	$SbCl_5$/nano-sawdust (0.01 g)	H_2O:EtOH/ r.t	45	73
7	$SbCl_5$/nano-sawdust (0.03 g)	H_2O:EtOH/ r.t	45	93
8	$SbCl_5$/nano-sawdust (0.02 g) 2^{nd} run	H_2O:EtOH/ r.t	45	90
9	$SbCl_5$/nano-sawdust (0.02 g) 3^{rd} run	H_2O:EtOH/ r.t	45	87
10	-	H_2O:EtOH/ r.t	45	-

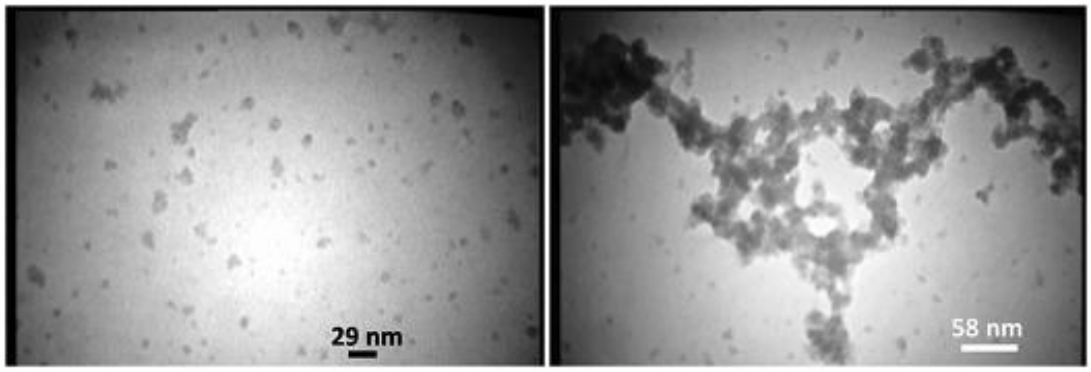


Figure 1: TEM micrograph of $SbCl_5$/nano-sawdust.

The results of EDX analyses (Table **3**) before and after reaction with SbCl5 were reported. The EDX data results showed the high peak of antimony and chlorine in the nanocatalyst after reaction with SbCl5.compared to the gas phase,also increased by 314.5kJ/mol (308.9kJ/mol).

Table 2. EDXs Results of sawdust and $SbCl_5$/nano-sawdust.

	Sawdust	$SbCl_5$/nano-sawdust
Element	W%	W%
C	56.85	12.87
O	39.89	21.09
Cl	-	31.05
Sb	-	34.87

The thermal decomposition of SbCl5/nano-sawdust wasexamined, using athermogravimetry (TGA) and derivative thermogravimetry (DTG)under nitrogen atmosphere over 800˚C. The structural conversion was observed by TG curves. They were supported by the obtained DTG data. Thermal analysiswas found being useful structural elucidation SbCl5/nano-sawdust. The thermal stability and the decomposition modewere carefully controlled under heating rate. In fact, three thermal decomposition mass loss steps were realized for the SbCl5/nano-sawdust (Figure **2**).The first stage of degradation at 142 °C, corresponds to 41.81% mass loss was attributed to the removal of moisture. In the secondstage of decomposition, DTG curve exhibits mass losses at 551°C and 7.79%, most probably due to the common degradation of the cellulose units. The mass loss of 7.70% at the third stage can be well attributed to theDTG peak at 569 °C, It is a common mass for decomposition of lignin. FT-IR spectrum of $SbCl_5$/nano-sawdust catalyst (Figure **3**), showed the hydroxyl bonds of sawdust catalysis at 3443 cm^{-1}. The peak at 2931 cm^{-1} belongs to C-H stretching vibrations of the aliphatic systems for cellulose and hemicelluloses units. The peak at 1616 cm^{-1} is assigned to C=C stretching vibrations of present in phenyl rings of lignin unit. The Sb-O stretching vibration observed in the 1011 cm^{-1}. The FT-IR data's and EDX approved the actual event of chemical interaction of antimony pentachloride with the surface area of sawdust.

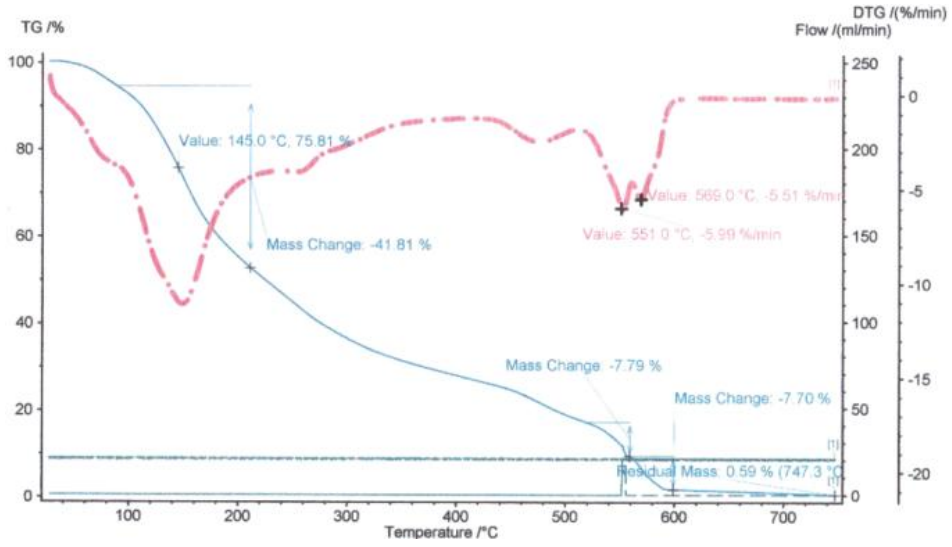


Figure 2: The TGA/DTG curves of SbCl5/nano-sawdust.

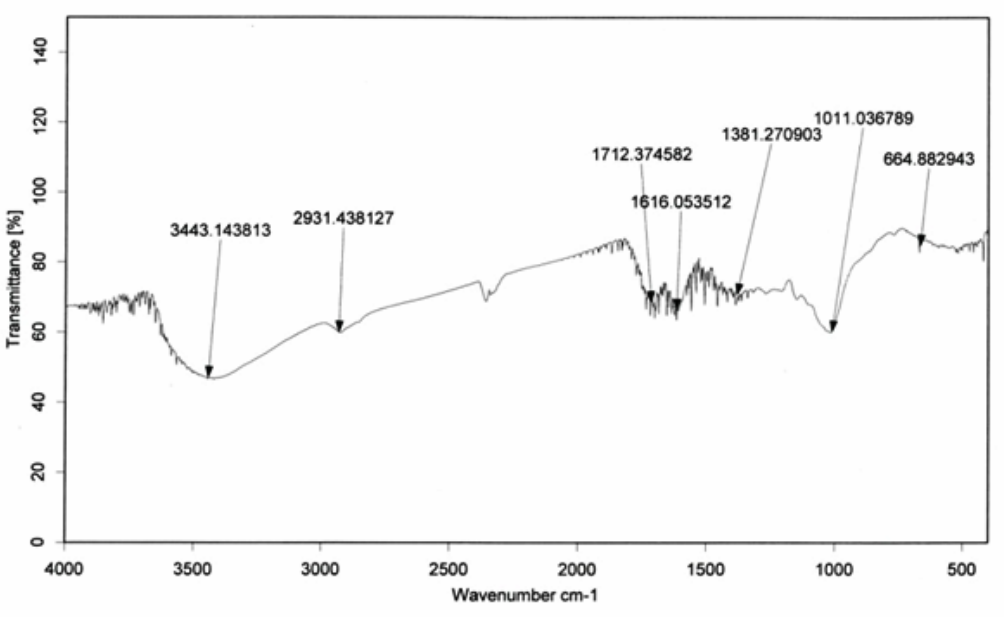
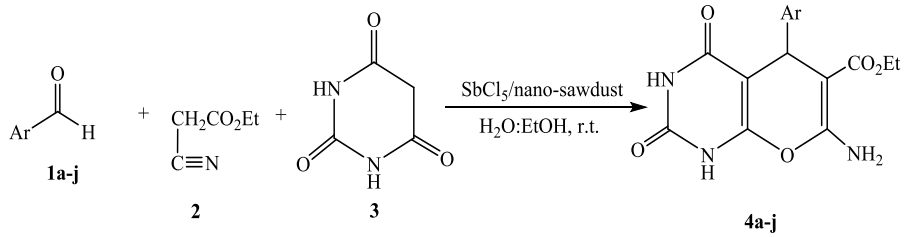


Figure 3: FT IR spectrum of $SbCl_5$/nano-sawdust.

Model reaction were performed at ambient temperature in ethanol/water using different amounts of $SbCl_5$/nano-sawdust for determination of the optimum quantity of $SbCl_5$/nano-sawdust.As illustrated in Table **2**, 0.02 g of $SbCl_5$/nano-sawdust catalysis affords an excellent yield(92%). To establish the generality of this method, differently substituted aromatic aldehydes were employed, under optimized conditions, leading to the formation of the corresponding pyrano[2,3-*d*]pyrimidines in satisfactory yields (Scheme **1**).The results are given in Table **3.**



Scheme 1: Synthesis of pyrano [2,3-*d*] pyrimidine derivatives in the presence of $SbCl_5$/nano-sawdust as catalyst.

Table 3. Synthesis of pyrano [2,3-*d*] pyrimidines.

Entry	Ar	Product	Time(min)	Yield	M.P.(˚C) Ref.
1	C_6H_5	**4a**	45	92	206-208(206-210) [13]
2	*m*-Cl-C_6H_4	**4b**	45	87	282-284(283-284) [14]

3	*p*-Cl-C_6H_4	**4c**	40	89	297-299(>300) [14]
4	*m*-NO_2-C_6H_4	**4d**	40	90	262-264(237-240) [13]
5	*p*-NO_2-C_6H_4	**4e**	40	92	290-292(289-293) [13]
6	*p*-CH_3-C_6H_4	**4f**	50	88	295-298(296-298) [13]
7	*p*-CH_3O-C_6H_4	**4g**	50	89	293-295(297-298) [14]
8	*3,4*-CH_3O-C_6H_3	**4h**	55	91	>300 (303-306) [13]
9	*m*-OH-C_6H_4	**4i**	40	92	172-174(170-174) [14]
10	*p*-OH-C_6H_4	**4j**	40	93	169-170(163-167) [13]

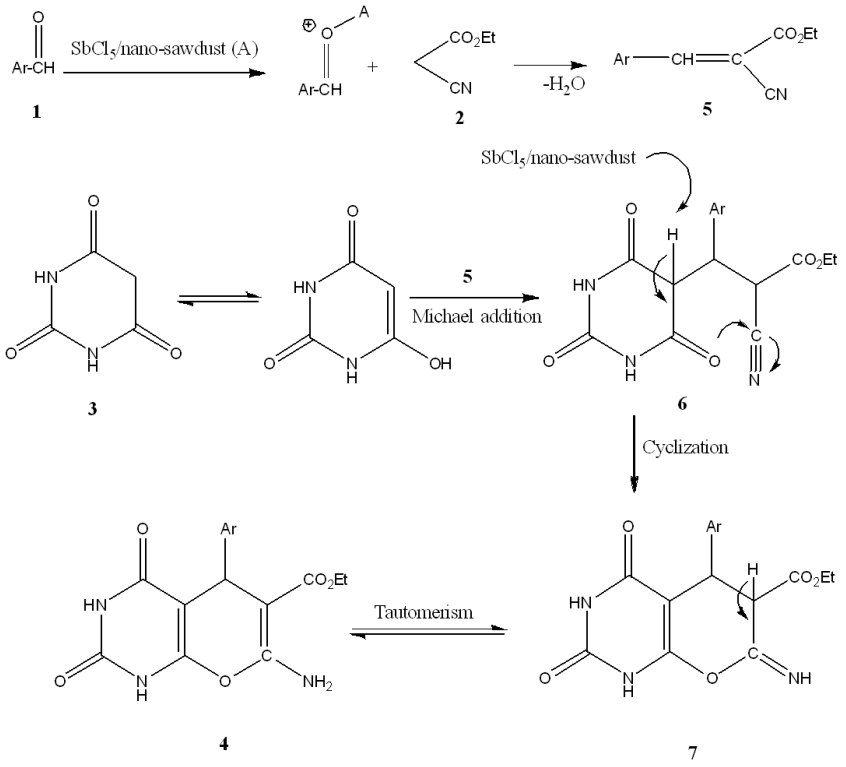
Comparison of SbCl5/nano-sawdust and various catalyst in the synthesis of 4a was tested in Table **4**. Literature survey revealed some other effective catalysts such as DABCO[13],CaCl2[14] and Glycerol [15]which have been used in the catalytic synthesis ofpyrano[2,3-d]pyrimidines, each has its own merits and drawbacks. Some of disadvantages in these methods, realized are harsh reaction conditions, long reaction times, tedious work up procedure and difficulty in purification, high amount of catalyst loading and moderate yields. Thus, the development of a simple, mild and efficient method was still much in demand. After successful synthesis of pyrano[2,3-d]pyrimidine derivatives in the presence of our novel catalytic system, SbCl5/nano-sawdust, we thought, it is rational to compare our results with those of previously reportedfrom different points of view. As shown in Table **4**, the synthesis of pyrano[2,3-d]pyrimidines in the presence of SbCl5/nano-sawdust in H2O:EtOH needsreasonable reaction time and yields and occurs under milder conditions relative to the three selected previously reported protocols.Practically, when SbCl5 was supported with sawdust was not fuming at all, and showed no change as an easy to handle solid with no tendency to hydrolyze to hydrochloric acid.

Table 4. Comparison of $SbCl_5$/nano-sawdust and various catalyst in the synthesis of pyrano [2,3-*d*] pyrimidine derivatives.

Entry	Catalyst	Solvent	Condition	Time (min)	Yield	Ref.
1	DABCO, 10 mol%	H_2O:EtOH	r.t	30-40	82-94	[13]
2	$CaCl_2$, 20 mol%	EtOH	US	10	90-93	[14]
3	$CaCl_2$, 20 mol%	EtOH	r.t	120-170	90-92	[14]
4	Glycerol, 1 mL	-	80 °C	100-150	90-92	[15]
5	$SbCl_5$/nano-sawdust	H_2O:EtOH	r.t	40-55	87-95	This work

A plausible mechanism of this reaction was proposed in Scheme **2**. The initiation step of this chain process was begun with the interaction of aldehyde **1** and $SbCl_5$/nano-sawdustas a solid acid catalyst. The subsequent step was Knoevenagel condensation between the activated aldehyde and ethylcyanoacetate **2** to form intermediate **5**. Then the Michael addition of barbituric acid **3** to intermediate **5** would furnish intermediate **6**. Finally, the product **4** was obtained by an intramolecular cyclization and tautomerism.

After the completion of the reaction, the mixture was filtered to remove the catalyst. The catalyst was washed well with ethanol and dried at room temperature for 7 h.



Scheme 2: Plausible mechanism for the formation of pyrano [2,3-d] pyrimidine derivatives.

The reusability of $SbCl_5$/nano-sawdust was tested by repeating the model study in the presence of $SbCl_5$/nano-sawdust under optimized conditions. The results of these experiments showed that $SbCl_5$/nano-sawdust can be regenerated at the end of the reaction and can be used 3 times without losing too much activity (Table **5**).

Table 5. Recoverability of $SbCl_5$/nano-sawdust.

Yield (%)		
First	Second	Third
93	90	87

Conclusions

We have demonstrated a rapid and an efficient synthetic route for SbCl5/nano-sawdust catalyzed one-pot three component synthesis of pyrano[2,3-d]pyrimidines in EtOH/H2O as solvent. This method was compared with other catalysts reported in literature, indicating cleanliness, simple methodology and short time are some advantages of this method.

Experimental Section

Nano sawdust was prepared by the method reported previously by our research group[11, 12]. All other chemicals were purchased from Fluka chemical companies and used as they received. An Electrothermal 9100 apparatus measured melting points. IR spectra were recorded from KBr disk on the Shimadzu IR-470. The 1H NMR spectra were obtained from Bruker DRX-300 Advancedspectrometer using TMS as internal standard. The size and morphologies of the nanoparticles were measured by obtaining TEM (Philips CM10) and respectively. Thermogravimetric analysis was conducted by using a TG 209 F1 (Netzsch Germany). The EDX analysis was performed using a SAMx-analyser.

Synthesis of SbCl5/nano-sawdust

The SbCl5/nano-sawdust catalysis was prepared by stirring a mixture of SbCl5 (1 ml) and 1 g of sawdust in 5 ml of ethanol for 1h at room temperature. The slurry was filtered and washed with ethanol. The solidwas dried in an oven at 60°C for 4h and then was milled at the mortar.

Synthesis of pyrano[2,3-d]pyrimidines

SbCl5/nano-sawdust catalysis (0.02 g) was added to a stirred mixture of anaromatic aldehyde (1 mmol),

ethylcyanoacetate (1 mmol) and barbituric acid (1 mmol) in EtOH:H2O(5 mL). This mixture was magnetically stirred atroom temperature for the indicated time (Table **1**). TLC monitored the progress of the reaction (n-hexane:ethylacetate 3:1). Upon the completion of the reaction, the reaction mixture was conventionally filtered off for the removal the heterogeneous catalyst. The filtrate was evaporated under reduced pressure and the crude product was crystallized from hot ethanol to obtain the pure corresponding desired compounds. The melting points and yields of products are given in table **1**. For recovery of the catalyst, after completion of the reaction, the mixture was filtered and catalyst was washed with ethanol and the solid residue was then dried. This recovered catalyst was employed in the reactions (Table **1**).

Selected spectral data

Ethyl 7-amino-2,4-dioxo-5-phenyl-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4a):

IR (KBr, cm^{-1}): 3387, 3164, 1712; ^{1}H NMR (300 MHz, DMSO-d_6): δ 3.6 (s, 3H, CH_3), 4.18 (s, 1H), 4.31 (s, 2H, CH_2), 6.09 (s, 1H, ArH), 7.09 (s, 2H, NH_2), 6.51-8.13 (m, 4H, ArH), 11.12 (s, 1H, NH), 12.14 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 30.0, 60.2, 69.2, 128.6, 129.8, 130.9, 135.4, 146.4, 148.6, 156.3, 152.8, 159.2, 160.7, 161.0 ppm.

Ethyl 7-amino-5-(3-chlorophenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4b):

IR (KBr, cm^{-1}): 3376, 3343, 3192, 1727; ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.17 (s, 3H, CH_3), 3.94 (s, 2H, CH_2), 4.73 (s, 1H), 6.99 (s, 2H, ArH), 7.11-7.25 (m, 2H, ArH), 7.21 (s, 2H, NH_2), 9.10 (s, 1H, NH), 11.17 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 35.7, 52.8, 76.9, 78.8, 124.8, 125.2, 127.9, 129.6, 133.5, 137.4, 143.0, 150.2, 160.1, 160.3, 163.2, 165.4 ppm.

Ethyl 7-amino-5-(4-chlorophenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4c):

IR (KBr, cm^{-1}): 3327, 3187, 1705; ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.29 (s, 3H, CH_3), 4.11 (s, 2H, CH_2), 5.28 (s, 1H), 7.28 (m, 2H, ArH), 7.37 (m, 2H, ArH), 7.75 (s, 2H, NH_2), 10.99 (s, 1H, NH), 11.55 (r s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 37.6, 98.5, 114.8, 126.9, 128.8, 129.9, 130.5, 135.9, 150.1, 155.4, 155.8, 159.7, 160.9 ppm.

Ethyl 7-amino-5-(3-nitrophenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4d):

IR (KBr, cm^{-1}): 3385, 3327, 1730; ^{1}H NMR (300 MHz, DMSO-d_6): δ 3.63 (s, 3H, CH_3), 4.01 (s, 2H, CH_2), 4.82 (s, 1H), 7.26 (s, 2H, NH_2), 7.52 (m, 2H, ArH), 8.13 (m, 2H, ArH), 11.12 (s, 1H, NH), 12.18 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 35.7, 57.5, 87.5, 119.0, 124.4, 130.7, 130.9, 146.4, 149.6, 151.9, 152.7, 157.8, 159.2, 161.7, 162.6 ppm.

Ethyl 7-amino-5-(4-nitrophenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4e):

IR (KBr, cm^{-1}): 3422, 3374, 3106, 1730; ^{1}H NMR (300 MHz, DMSO-d_6): δ 3.09 (s, 3H, CH_3), 4.12 (s, 2H, CH_2), 4.92 (s, 1H), 7.26 (s, 2H, NH_2), 7.32 (m, 2H, ArH), 8.09 (m, 2H, ArH), 9.67 (s, 1H, NH), 10.15 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 37.2, 61.7, 79.5, 121.0, 128.0, 130.1, 131.2, 145.4, 148.3, 150.5, 160.3, 162.3, 163.8, 167.2 ppm.

Ethyl 7-amino-5-(4-methylphenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4f):

IR (KBr, cm^{-1}): 3395, 3367, 3106, 1723; ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.36 (s, 3H, CH_3), 2.60 (s, 3H, CH_3), 4.13 (s, 1H), 5.21 (s, 2H, CH_2), 7.12 (m, 2H, ArH), 7.20 (m, 2H, ArH), 7.60 (s, 2H, NH_2), 10.89 (s, 1H, NH), 11.43 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 20.9, 35.2, 88.7, 98.2, 115.5, 127.5, 128.1, 133.7,137.4, 150.1, 155.5, 155.9, 159.1, 159.9, 160.8 ppm.

Ethyl 7-amino-5-(4-methoxyphenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4g):

IR (KBr, cm^{-1}): 3413, 3389, 3106, 1732; ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.49 (s, 3H, CH_3), 3.32 (s, 3H, OCH_3), 3.71 (s, 2H, CH_2), 4.41 (s, 1H), 6.93 (m, 2H, ArH), 7.65 (m, 2H, ArH), 9.07 (s, 2H, NH_2), 10.03 (s, 1H, NH), 11.09 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 33.0, 37.2, 55.8, 75.6, 114.2, 126.0, 128.4, 130.1, 134.2, 143.9, 150.5, 157.2, 162.4, 167.3 ppm.

Ethyl 7-amino-5-(3,4-dimethoxy phenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4h):

IR (KBr, cm^{-1}): 3493, 3303, 3106, 3123, 1722; ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.06 (s, 3H, CH_3), 3.50 (s, 2H, CH_2), 3.78 (s, 3H, OCH_3), 4.02 (s, 3H, OCH_3), 4.2 (s, 1H), 7.1 (s, 2H, NH_2), 8.27 (m, 1H, ArH), 8.47 (m, 2H, ArH), 11.1 (s, 1H, NH), 11.4 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 37.9, 56.3, 57.4, 79.7, 114.2, 124.3, 127.5, 128.7, 135.8, 138.0, 143.8, 146.9, 149.4, 150.1, 157.3, 157.9, 163.9 ppm.

Ethyl 7-amino-5-(3-hydroxyphenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4i):

IR (KBr, cm^{-1}): 3493, 3337, 3106, 1723; ^{1}H NMR (300 MHz, DMSO-d_6): δ 3.6 (s, 3H, CH_3), 3.91 (s, 2H, CH_2), 4.10 (s, 1H), 6.56 (s, 2H, NH_2), 6.59 (m, 1H, ArH), 7.04-7.10 (m, 3H, ArH), 9.33 (s, 1H, OH), 11.1 (s, 1H, NH), 12.1 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 35.6, 59.9, 89.5, 114.7, 114.9, 118.8, 120.1, 127.4, 128.0, 130.1, 146.5, 150.4, 153.1, 158.1, 158.5, 163.3 ppm.

Ethyl 7-amino-5-(4-hydroxyphenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carboxylate (4j):

IR (KBr, cm^{-1}): 3343, 3185, 1719; ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.43 (s, 3H, CH_3), 3.17 (s, 2H, CH_2), 3.68 (s, 1H), 6.07 (s, 1H, OH), 6.67 (m, 2H, ArH), 6.74 (m, 2H, ArH), 7.31 (s, 2H, NH_2), 10.47 (s, 1H, NH), 11.03 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 29.03, 37.2, 61.5, 75.2, 79.9, 115.3, 123.7, 129.0, 134.4, 142.3, 150.4, 155.5, 160.1, 163.5 ppm.

Acknowledgements

Authors sincerely thank the Islamic Azad University of Yazd and Payam Noor University for providing financial support of this work.

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