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N-sulfonic acid pyridinium-4-carboxylic acid chloride as a novel and efficient catalyst for the condensation reaction of aldehyde with thiobarbituric acid and ammonium acetate

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ABSTRACT

N-sulfonic acid pyridinium -4-carboxylic acid chloride {[Pyridine-*N*-SO_3H-4-COOH]Cl} was synthesized, identified and applied as an effective catalyst for the preparation of pyrido[2,3-*d*:6,5-*d'*]dipyrimidines by the condensation reaction of various aldehyde with 2-thiobarbituric acid and ammonium acetate under solvent-free conditions. The structure of catalyst was studied by various techniques including FT-IR, ^{1}HNMR, ^{13}CNMR, SEM, EDS, TEM, TGA, DTG and XRD analyses.

***Keywords**: N-sulfonic acid pyridinium-4-carboxylic acid chloride, Pyrido[2,3-d:6,5-d']dipyrimidine, 2-Thiobarbituric acid, Ammonium acetate, Aldehyde, Ionic liquid, Solvent-free.*

1. Introduction

The structure of pyridopyrimidine derivatives was seen in many natural heterocyclic compounds containing biological properties which could be used in drug discovery programs. The mentioned properties of pyridopyrimidine depend on the position of the nitrogen in the fused ring in the structure of these compounds [1,2]. Pyridopyrimidine derivatives have a wide range of pharmacological properties and biological activities, including dihydrofolate reductase inhibitory activity [3], antimicrobial activity [4], antitumor activity [5], anti-inflammatory [6], tyrosine kinase inhibition [7,8], calcium channel antagonists [9] and fibroblast growth factor receptor inhibition [3, 10-12].

Various methods for the synthesis of 1,4-DHPs and pyrimidine-fused heterocycles were reported by the reaction of cyclic diketone, aromatic aldehydes and amines or ammonium acetate using different catalysts and reaction conditions [13-17].

Recently, *N*-sulfonic acid heterocyclic compounds as new salts were designed and introduced as effective organo-catalyst and organo-reagent for the preparation of some important organic compounds including bis(indolyl)methans [18], *β*-azido alcohols [19], nitro aromatic compounds [20,21], 1-amidoalkyl-2-naphthols [22], benzimidazoles [23], xanthenes [24], 1-carbamatoalkyl-2-naphthols [25], 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol)s [26], t-butyl aryl carbamates [27], 1,2,4,5-tetrasubstituted imidazoles [28], bis-coumarins [29], hexahydroquinolines [30] and amidoalkyl phenols and bis amidoalkyl phenols [31].

Organocatalysts are generally cheap, stable and available. They have low toxicity and low sensitivity to air humidity. Also, the lack of use of metals in the structure of these compounds is another advantage of using these compounds [32]. One of the superiorities of organocatalysts is their desirable surface to volume ratio which modified the connection between reactants and catalyst and grows the catalytic activity of them [33].

Herein, in continuation of past studies associated with the design and expansion of modern acidic functionalized solid salts and ionic liquids in organic synthesis [18-31, 34-36], we have prepared and identified a novel organo-salt namely *N*-sulfonic acid pyridinium-4-carboxylic acid chloride {[Pyridine-*N*-SO_3H-4-COOH]Cl} (Scheme 1) and successfully tested in the synthesis of various pyrido[2,3-*d*:6,5-*d'*]dipyrimidine derivatives exhibiting various significant properties (Schemes 1 & 2).

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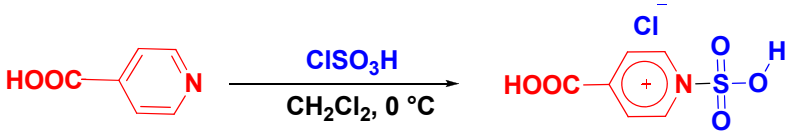


Scheme 1. The preparation of *N*-sulfonic acid pyridinium-4-carboxylic acid chloride.

2. Experimental

2.1. General procedure for the preparation of [Pyridine-N-SO₃H-4-COOH]Cl

A mixture of 4-pyridinecarboxylic acid (0.615 g, 5 mmol) in CH_2Cl_2 (15 mL) was added dropwise to a 50 mL round-bottomed flask containing a solution of chlorosulfonic acid (0.58 g, 5 mmol) in dichloromethane (10 mL) in a period of 5 minutes and stirred at 0 ℃ for 40 minutes and then the solvent was removed. The residue was washed with dry CH_2Cl_2 (3×30 mL), to remove the starting materials from the product, and then it was dried under vacuum to prepare pyridinium-*N*-sulfonic acid-4-carboxylic acid chloride {[Pyridine-*N*-SO_3H-4-COOH]Cl} as a white precipitate in 95% yield.

Spectral data for N-sulfonic acid pyridinium-4-carboxylic acid chloride:

White solid. IR (KBr): $\bar{\nu}$= 754, 1090, 1192, 1242, 1329, 1508, 1602, 1733, 2602, 2650–3186 cm^{-1}. ^{1}HNMR (400 MHz, DMSO-d_6): δ= 7.68 (d, *J* = 8.00 Hz, 1H), 7.85 (d, *J* = 8.40 Hz, 1H),13.21 (s, 2H, acidic hydrogens) ppm. ^{13}CNMR (100 MHz, DMSO-d_6): δ= 126.8, 129.9, 131.2, 166.5 ppm.

2.2. General procedure for the synthesis of pyrido[2,3-d:6,5-d']dipyrimidines

A mixture of aldehyde (1 mmol), 2-thiobarbituric acid (2 mmol) and ammonium acetate (1.2 mmol) and {[Pyridine-*N*-SO_3H-4-COOH]Cl (0.0238 g, 10 mol%) in a 25 mL round-bottomed flask connected to a reflux condenser, was stirred in an oil-bath at 80 ℃ for appropriate time. Then, the reaction mixture was cooled to room temperature. Afterward, the product was purified by washing with ethanol and hot water.

Spectral data for new compounds

5-(2, 4-Dimethoxyphenyl)-2, 8-dithioxo-2, 3, 7, 8, 9, 10-hexahydropyrido [2, 3-d:6, 5-d'] dipyrimidine-4, 6 (1H,5H)-dione (1a):

Yellow solid. m.p.= 281-285 ℃. IR (KBr): $\bar{\nu}$= 3426, 3146, 2937, 1610, 1508, 1455 cm^{-1}. ^{1}HNMR (400 MHz, DMSO-d_6): δ= 3.70 (s, 6H, $2CH_3$), 5.84 (s, 1H, CH), 6.33 (d, *J* =6.4 Hz, 1H, ArH), 6.92 (d, *J*=8.4 Hz, 1H, ArH), 7.11 (s, 1H, ArH), 11.34 (s, 2H, NH), 11.47 (s, 2H, NH), 16.92 (s, 1H, NH)ppm. ^{13}CNMR (DMSO-d_6, 100 MHz): δ= 19.0, 27.0, 55.4, 55.8, 64.9, 96.4, 98.8, 103.6, 124.0, 129.2, 158.2, 158.7, 163.8, 172.8 ppm.

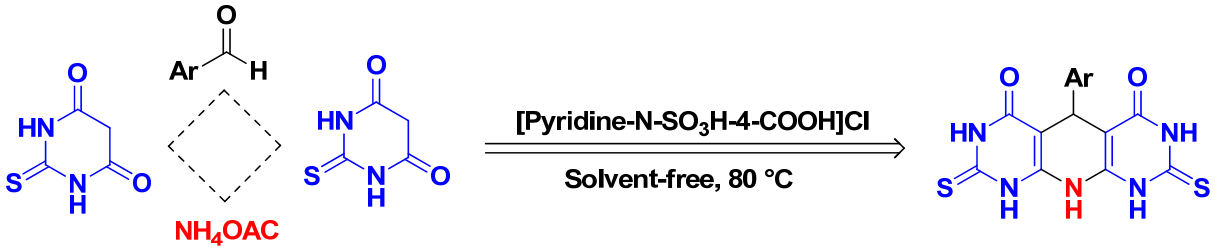
5-(2, 5-Dimethoxyphenyl)-2, 8-dithioxo-2, 3, 7, 8, 9, 10-hexahydropyrido [2, 3-d:6, 5-d'] dipyrimidine-4, 6 (1H,5H)-dione (1b):

White solid. m.p.= 267-268 ℃. IR (KBr): $\bar{\nu}$= 3479, 3083, 2887, 1631, 1532, 1394 cm^{-1}. ^{1}HNMR (400 MHz, DMSO-d_6): δ= 3.36 (s, 6H, $2CH_3$), 5.71 (s, 1H, CH), 6.84 (s, 1H, ArH), 6.99 (d, *J*=7.2 Hz, 1H, ArH), 7.05 (d, *J*=1.2 Hz, 1H, ArH), 10.32 (s, 2H, NH), 11.05 (s, 2H, NH), 16.82 (s, 1H, NH) ppm. ^{13}CNMR (DMSO-d_6, 100 MHz): δ= 27.0, 28.4, 46.9, 50.9, 54.2, 89.0, 101.4, 108.9, 128.2, 134.5, 149.4, 153.4, 163.9, 178.7 ppm.

3. Results and Discussion

3.1. Characterization of the catalyst

[Pyridine-*N*-SO_3H-4-COOH]Cl was prepared by the reaction of 4-pyridinecarboxylic acid with chlorosulfonic acid (Scheme 1) and identified by different techniques. In the IR spectrum of the catalyst, which is given in supporting information, the broad peak at 2650–3186 cm^{-1} is related to O-H stretching of the SO_3H group in [Pyridine-*N*-SO_3H-4-COOH]Cl.



Scheme 2. The preparation of pyrido[2,3-*d*:6,5-*d'*]dipyrimidines.

Also, the two peaks displayed at 1192 cm^{-1} and 1329 cm^{-1} correspond to vibrational modes of N-SO_2 and O-SO_2 bonds. This observation confirms the bond between SO_3H group and nitrogen of pyridine [22, 28].

Energy-dispersive X-ray spectroscopy (EDX) of [Pyridine-*N*-SO_3H-4-COOH]Cl was studied and displayed in the presence of carbon, oxygen, nitrogen, sulfur and chlorine and the structure of the catalyst. Hence, the structure of [Pyridine-*N*-SO_3H-4-COOH]Cl was supported by EDX analysis (Fig. 1).

The scanning electron microscope (SEM) micrograph of [Pyridine-*N*-SO_3H-4-COOH]Cl was also studied and showed that the particle size of the catalyst is on a nano scale and micro scale (Fig. 2).

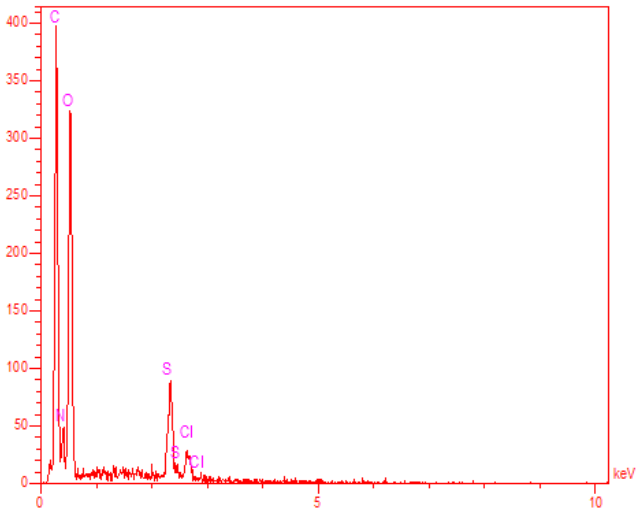


Fig. 1. Energy-dispersive X-ray spectroscopy (EDX) of *N*-sulfonic acid pyridinium-4-carboxylic acid chloride.

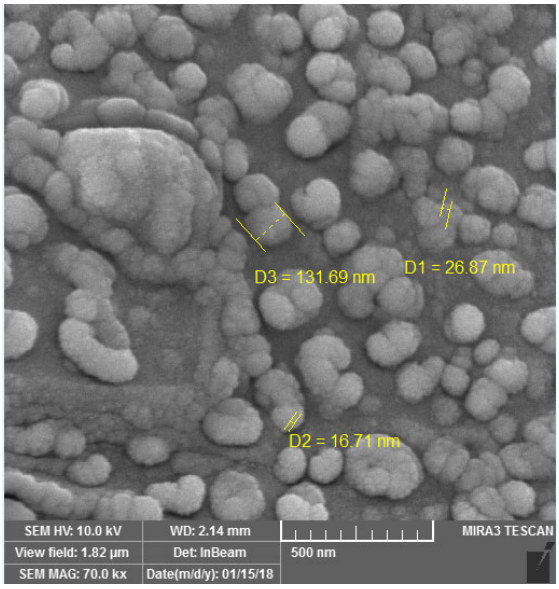


Fig. 2. SEM micrograph of *N*-sulfonic acid pyridinium-4-carboxylic acid chloride.

Thermal gravimetric analysis (TGA) and differential thermal gravimetric (DTG) of [Pyridine-*N*-SO_3H-4-COOH]Cl were studied and indicated that the catalyst can be used up to 200 °C (Fig. 3).

X-ray diffraction analysis (XRD) pattern of [Pyridine-*N*-SO_3H-4-COOH]Cl was also investigated in a domain of 0-90 degree (Fig. 4). XRD patterns depicted diffraction lines of a high crystalline nature at about 2Θ = 15.4°, 20.3°, 21.1°, 24.7°, 26.8°, 28.0° and 29.9°.

To prove the activity of [Pyridine-*N*-SO_3H-4-COOH]Cl as new catalyst, we have tested it in the synthesis of pyrido[2,3-*d*:6,5-*d'*]dipyrimidines. To optimize the reaction conditions, the condensation of 2-thiobarbituric acid (2 mmol), 4-chlorobenzaldehyde (1 mmol), and ammonium acetate (1.2 mmol), was studied using different amounts of [Pyridine-*N*-SO_3H-4-COOH]Cl at range of 50 to 100 °C under solvent-free conditions (Table 1). The results show that 10 mol% of [Pyridine-*N*-SO_3H-4-COOH]Cl was enough to catalyze the reaction at 80 °C. In these reaction conditions, the expected pyrido[2,3-*d*:6,5-*d'*]dipyrimidine was prepared in 90% of yield within 10 min (Table 1, entry 2).

To investigate the effect of solvent, we tested the model reaction in different solvents, including $CHCl_3$, H_2O, CH_2Cl_2, EtOAC, EtOH, acetone and n-hexane using 10 mol% of the catalyst under reflux condition. The results of these experiments showed that the reaction in a solvent led to a significant decrease in the yield of the desired product in comparison with the obtained yield under solvent-free conditions (Table 2, entries 1–7).

To investigate the extent and efficiency of the proposed method, 2-thiobarbituric acid and ammonium acetate was reacted with various aromatic aldehydes containing electron withdrawing groups, electron-donating groups and halogens, under the optimized reaction conditions (Table 3).

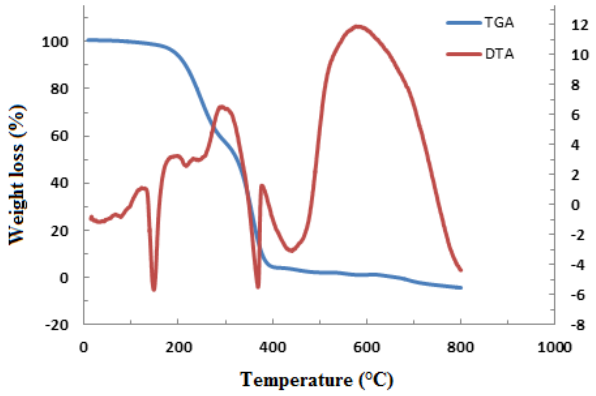


Fig. 3. Thermal gravimetric analysis (TGA) and differential thermal gravimetric (DTA) analysis of *N*-sulfonic acid pyridinium-4-carboxylic acid chloride at range of 0–800 °C, with a temperature increase rate of 10 °C per minute.

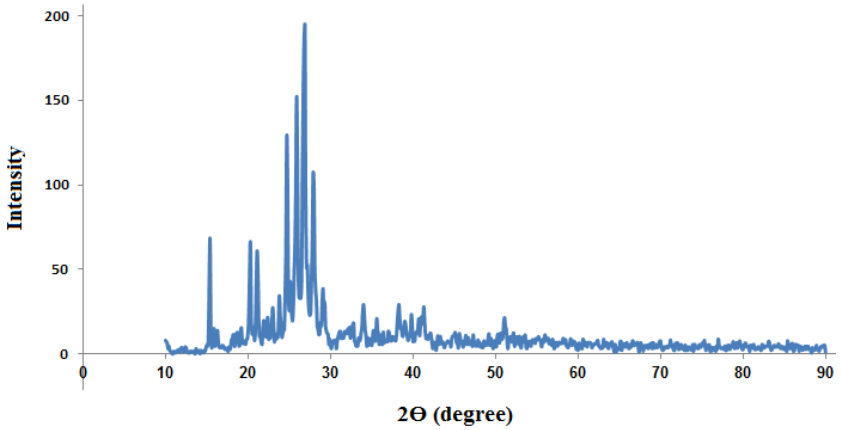


Fig. 4. XRD diagram of *N*-sulfonic acid pyridinium-4 -carboxylic acid chloride.

Table 1. Effect of the catalyst amount and temperature on the reaction between 2-thiobarbituric acid, 4-chlorobenzaldehyde and ammonium acetate.

Entry	Catalyst	Catalyst amount (mol%)	Temp. (ºC)	Time (min)	Yield (%)[a]
1	[Pyridine-*N*-SO_3H-4-COOH]Cl	10	50	25	80
2	[Pyridine-*N*-SO_3H-4-COOH]Cl	10	80	10	90
3	[Pyridine-*N*-SO_3H-4-COOH]Cl	10	100	10	90
4	[Pyridine-*N*-SO_3H-4-COOH]Cl	5	80	30	75
5	[Pyridine-*N*-SO_3H-4-COOH]Cl	7	80	20	85
6	[Pyridine-*N*-SO_3H-4-COOH]Cl	15	80	10	90

[a]Isolated yield.

Table 2. Effect of various solvents on the reaction of 2-thiobarbituric acid (2 mmol), 4-chlorobenzaldehyde (1 mmol), and ammonium acetate (1.2 mmol) in the presence of [Pyridine-*N*-SO_3H-4-COOH]Cl (0.0238 g, 10 mol %).

Entry[a]	Solvent	Temp. (°C)	Time (min)	Yield[a] (%)
1	$CHCl_3$	Reflux	10	45
2	EtOAc	Reflux	10	70
3	CH_2Cl_2	Reflux	10	55
4	H_2O	80	10	trace
5	Acetone	Reflux	10	65
6	n-hexane	Reflux	10	25
7[b]	-	80	10	90

[a]Isolated yield.
[b]The reaction proceeded in the absence of solvent.

As it is displayed in Table 3, all of the reactions proceeded efficiently to afford the expected pyrido[2,3-*d*:6,5-*d′*]dipyrimidine derivatives in high yields and in short reaction times.

To show the suitability of this method, an evaluation of this work with few formerly introduced methods for the synthesis of pyrido[2,3-*d*:6,5-*d′*]dipyrimidine (1g) was given in Table 4. It can be concluded that this method is superior to some of the previously reported methods in terms of the amount of catalyst used, the temperature and reaction time to afford the expected product.

Based on the proposed mechanism in the previously reported methods [37-39], the formation of the pyrimidine derivatives is expected to proceed by the reaction of aldehyde, which is activated by the catalyst, with 2-thiobarbituric acid to give intermediate **I** as a

Table 3. The solvent-free synthesis of pyrido[2,3-*d*:6,5-*d'*]dipyrimidines from 2-thiobarbituric acid, arylaldehydes and ammonium acetate catalyzed by [Pyridine-*N*-SO_3H-4-COOH]Cl (10 mol%) at 80 ℃.

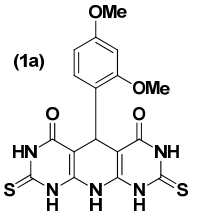
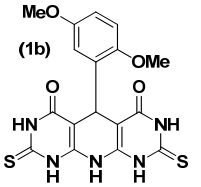
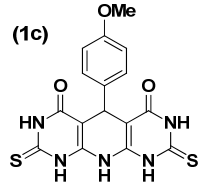
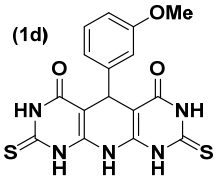
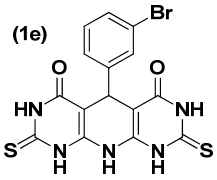
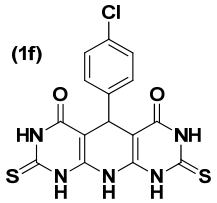
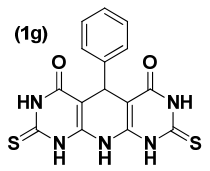
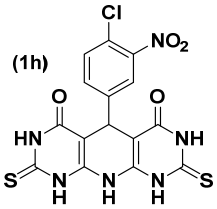
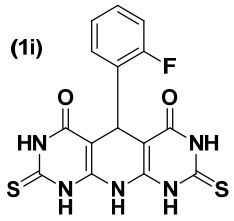
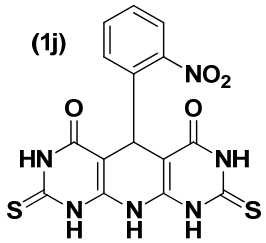
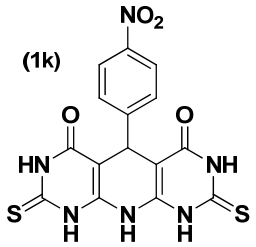
Entry	Product	Time (min)	Yield (%)[a]	m.p. (°C)		Ref.
				Found	Reported	
1		10	93	281-285	-	New
2		7	90	267-268	-	New
3		15	92	278-280	280	[17]
4		20	85	238-241	242	[17]
5		20	92	249-253	246-248	[39]
6		15	85	245-249	257	[17]
7		10	90	214-217	218	[17]
8		15	85	248-249	249	[16]

Table 3. (*Continued*).

9		5	95	239-240	240	[16]
10		5	93	232-234	230	[16]
11		5	95	327-329	330	[16]

[a]Isolated yield.

Michael acceptor. In the next step, another molecule of 2-thiobarbituric acid is reacted with ammonium acetate to prepare intermediate **II. III** is formed by the Michael reaction of **II** with **I** and finally, by the internal nucleophilic attack of intermediate **III**, the desired product was produced (Scheme 3).

4. Conclusions

In conclusion, we have synthesized and characterized [Pyridine-*N*-SO_3H-4-COOH]Cl as an efficient organo-catalyst with nano structure for the one-pot four-component reaction of various aldehydes, 2-thiobarbituric acid and ammonium acetate at 80 ℃ under solvent-free conditions to afford pyrido[2,3-*d*:6,5-*d'*]dipyrimidines.

Acknowledgments

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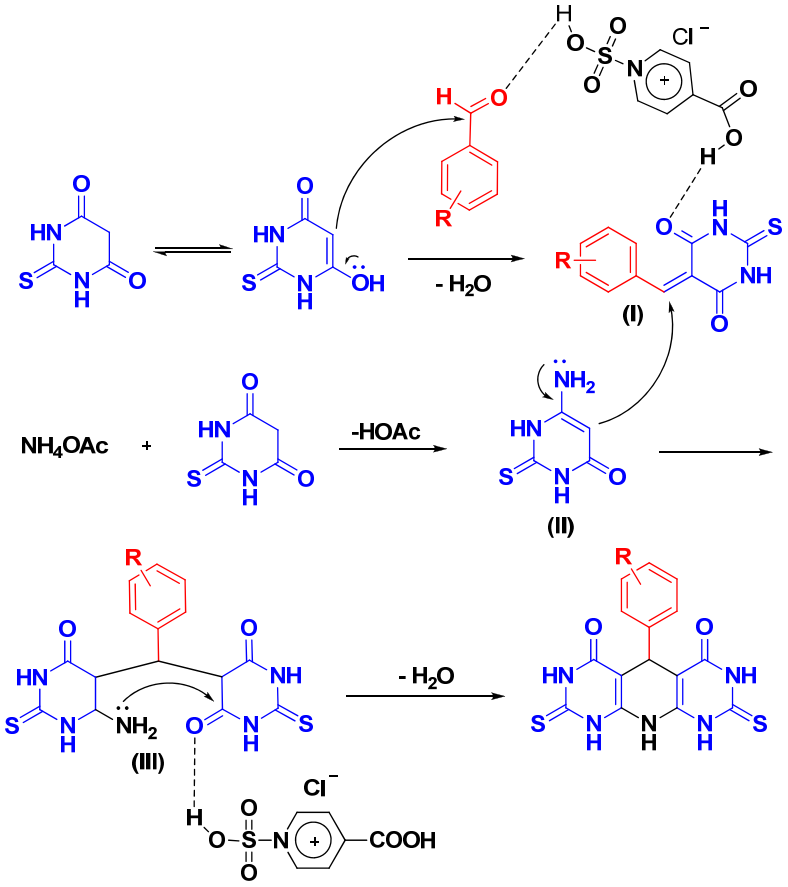
Table 4. Comparison of the efficiency of [Pyridine-*N*-SO_3H-4-COOH]Cl with various reported catalysts on the reaction of 2-thiobarbituric acid, benzaldehyde and ammonium acetate.

Entry	Catalyst /Reaction Conditions	Time	Yield (%)	Ref.
1	$[HNMP]^+[HSO_4]^-/H_2O$, US	8 min	95	[17]
2	Nano $CuFe_2O_4$/ H_2O, US	10 min	95	[16]
3	Al_2O_3 (solid phase)/ MW, 800w	6h	86	[38]
4	Al_2O_3 (liquid phase)/Δ	48 h	68	[38]
5	Fe-MCM-41-IL/H_2O, RT	40 min	89	[37]
6	[Pyridine-*N*-SO_3H-4-COOH]Cl/Solvent-free, 80 °C	10 min	90	This work



Scheme 3. Proposed mechanism for the synthesis of pyrido[2,3-*d*:6,5-*d'*]dipyrimidines.

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