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Fast one-pot synthesis of 1,8-dioxo-decahydroacridine derivatives using sulfonic acid functionalized LUS-1 and the study on their antimicrobial activities

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ABSTRACT

Mesoporous silica LUS-1 (Laval University Silica) was successfully functionalized by propyl sulfonic acid and was used as a recyclable catalyst for the synthesis of acridine-1,8-diones via a pseudo four component reaction of aromatic aldehydes, dimedone and ammonium acetate (or anilines). The excellent yields, short reaction time, simple work-up procedure, and environmentally friendly conditions are advantages of this method. All synthesized compounds were screened for antimicrobial and antifungal activities. Only compound **3e** exhibited activity against *Bacillus subtilis*.

***Keywords**: Sulfonic acid functionalized LUS-1; Acridine-1,8-dione; Dimedone; Nanoporous acid catalyst.*

1. Introduction

Mesoporous silica materials have received significant attention in recent years because of their potential applications as supports for catalysis. Functionalizations of the mesoporous silica materials pore walls by organo-acidic groups (e.g. propyl sulfonic acid) create various heterogeneous acid catalysts for organic reactions [1-6]. The LUS-1 is ordered-mesoporous silica with a high surface area (800 cm^2g^{-1}), long range ordered pores (average pore diameter 2-3 nm) and hydrothermal stability [7], which has the potential to be used as a support material for heterogeneous catalysts. In comparison to other ordered mesoporous silica, one of the most specific advantages of this material is different dispersion of hydroxyl groups on the silica surface, which results in higher hydrothermal stability. Furthermore, due to the presence of more hydroxyl groups on the silica surface of LUS-1, it will be able to react with more surface modifying groups [8].

Acridine-1,8-diones have structural similarity to 1,4-dihydropyridines (1,4-DHPs), which are valuable drugs for the treatment of cardiovascular disorders [9] and congestive heart failure [10]. Acridine diones have attracted many interests in view of the unique photochemical and electrochemical behavior of heterocyclic compounds. In particular, they can act as both electron donors and acceptors in the excited state because of their bichromophoric groups [11], and they have also been used in the photoinitiated polymerization of methacrylates and acrylates [12]. In addition, it has been reported that acridine diones can be used as laser dyes with very high-lasing efficiencies [13]. Recently, many methods have become available for the synthesis of these tricycle compounds containing the 1,4-dihydropyridines, from aldehydes, dimedone and ammonium acetate (or anilines) using different catalysts such as Ceric ammonium nitrate [14], Bronsted acid imidazolium salt [15], Amberlyst-15 [2], [Hmim]TFA [16], Silica-bonded *S*-sulfonic acid (SBSSA) [17], *p*-dodecylbenezenesulfonic acid (DBSA) [18] and Proline [19]. However, many of these reported methods suffer from drawbacks such as long reaction times, formation of side products, multistep synthesis, using organic solvents, using large amounts of catalysts, using toxic or expensive catalysts and using catalysts that cannot be recycled. In continuation of our previous works on the application of heterogeneous solid catalysts in organic synthesis

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[20-27], herein, we would like to report a highly efficient method in the synthesis of acridine-1,8-diones using LUS-Pr-SO_3H under solvent-free conditions.

2. Experimental

Melting points were measured using the capillary tube method with an electro thermal 9200 apparatus. IR spectra were obtained as KBr pellets on a FT-IR Bruker Tensor 27 instrument. ^{1}H NMR were recorded in $CDCl_3$ solvent on a Bruker, 300-500 MHz spectrometer with tetramethylsilane as internal reference. The N_2 adsorption/desorption measurements were conducted at liquid nitrogen temperature (77 K) using BELSORP-mini II**.** Scanning electron microscopy (SEM) was carried out on a LEO 1445V microscope.

2.1. Synthesis of LUS-1

Colloidal silica Ludox (15.5 g, 0.26 mol) was added to sodium hydroxide (2 g, 5×10^{-2} mol) in distilled water (50 mL) and the mixture was stirred at 70°C until a clear solution was obtained. A second solution of Cetyltrimethylammonium *p*-toluene sulfonate (2.5 g, 5.5×10^{-3} mol) in distilled water (90 mL) was stirred at 40°C during 1 h. The first solution was added dropwise to the second one and then the mixture was stirred at 40°C for 2 h. The resulting sol-gel was heated in an autoclave at 130°C for 20 h. The surfactant was removed by treatment with HCl 0.1 M in ethanol for 2 h. After filtration and washing with distilled water, the synthesized solid was dried under vacuum at 100°C [28].

2.2. Functionalization of LUS-1

The mixture of (3-Mercaptopropyl) trimethoxysilane (12 ml) and LUS-1 (10 g) in dry toluene was refluxed for 24 h and then filtered. The obtained LUS-Pr-SH was washed with acetone and dried LUS-Pr-SH was oxidized with H_2O_2 (50 ml) and one drop of H_2SO_4 in methanol (10 ml) for 24 h at room temperature. The mixture was filtered and washed with H_2O and then acetone to obtain pure LUS-Pr-SO_3H as catalyst (Fig. 1).

2.3. Ion-Exchange pH Analysis

To an aqueous solution of NaCl (1 M, 50 mL), LUS-Pr-SO_3H (0.5g) was added and the resulting mixture was stirred for 2 h.

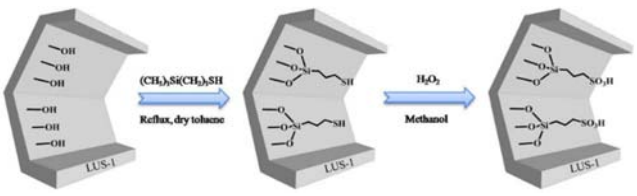


Fig. 1. Preparation of LUS-Pr-SO_3H.

The solution pH dropped virtually instantaneously to approximately pH 2, as ion exchange occurred between protons and sodium ions. This is equal to a loading of approximately 1 mmol SO_3H g^{-1}, which is in good agreement with the results obtained from TGA.

2.4. General Procedure for the synthesis of 1,8-dioxo-decahydroacridine derivatives

The sulfonic acid functionalized LUS-1 (0.02 g) was activated in vacuum at 100°C and then after cooling to room temperature, aromatic aldehyde **1** (1 mmol) and 5,5-dimethylcyclohexane-1,3-dione **2** (2 mmol, 0.28 g) and ammonium acetate **3** or anilines **4** (1.2 mmol) were added to it. The mixture was heated (120°C) in solvent-free conditions for an appropriate time as shown in Table 2. After completion of the reaction which was monitored by TLC, the mixture was dissolved in hot ethanol in order to separate the catalyst and then the filtrate was cooled to afford the pure product.

The catalyst was washed subsequently with diluted acid solution, distilled water and then acetone. Then, it was dried under vacuum and reused for several times without loss of a significant activity. The spectral (^{1}H NMR, MS and IR) and analytical data for selected compounds are given below.

Selected Spectral data

9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione ***(5e):***

Yellow solid. m.p.= 304-305°C. IR (KBr): $\bar{\nu}$ = 3287, 3207, 3047, 2956, 2870, 1644, 1608, 1472, 1391, 1364, 1222, 1168, 1140, 1087, 1012, 934, 885, 845, 818, 775, 730, 661, 567, 517 cm^{-1}. ^{1}HNMR (250 MHz, $CDCl_3$): δ= 7.68 (br s, 1H, NH), 7.28 (d, *J*=7.5 Hz, 2H, 2CH arom.), 7.16 (d, *J*=7.5 Hz, 2H, 2CH arom.), 5.06 (s, 1H, CH), 2.33-2.11 (m, 8H, $4CH_2$), 1.07 (s, 6H, $2CH_3$), 0.95 (s, 6H, $2CH_3$) ppm.

9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione ***(5h)****:*

Yellow solid. m.p.= 301-303°C; IR (KBr): $\bar{\nu}$ = 3275, 3204, 3071, 2957, 2870, 2839, 1645, 1607, 1508, 1482, 1423, 1394, 1366, 1299, 1261, 1223, 1169, 1144, 1031, 1006, 987, 834, 667, 610, 567, 528 cm^{-1}. ^{1}HNMR (250 MHz, $CDCl_3$): δ= 8.22 (s, 1H, NH), 7.25 (d, *J*=8.7 Hz, 2CH arom.), 6.71 (d, *J*=8.5 Hz, 2CH arom.), 5.04 (s, 1H, CH), 3.66 (s, 3H, OCH_3), 2.21-2.09 (m, 8H, $4CH_2$), 1.05 (s, 6H, $2CH_3$), 0.94 (s, 6H, $2CH_3$) ppm. EIMS: m/z (rel. int.)= 381 [M+1] (5), 379 [M^+] (15), 272 (100), 216 (13), 188 (10), 92 (15), 77 (20), 41 (22).

9-(2,3-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione ***(5i):***

Yellow solid. m.p.= 326-328°C; IR (KBr) $\bar{\nu}$ = 3458, 3281, 3213, 3061, 3027, 2955, 2929, 2874, 1639, 1625, 1605, 1480, 1424, 1393, 1367, 1249, 1217, 1167, 1140,

1120, 1072, 1029, 1001, 980, 941, 913, 885, 836, 817, 700, 516 cm^{-1}. ^{1}HNMR (250 MHz, DMSO-d_6): δ= 9.19 (s, 1H, NH), 6.78-6.64 (m, 3H, 3CH arom.), 5.03 (s, 1H, CH), 3.81 (s, 3H, OCH_3), 3.69 (s, 3H, OCH_3), 2.39 (d, *J*=17.0 Hz, 2H, 2CH), 2.25 (d, *J*=17.0 Hz, 2H, 2CH), 2.09 (d, *J*=16.0 Hz, 2H, 2CH), 1.90 (d, *J*=16.0 Hz, 2H, 2CH), 0.96 (s, 6H, $2CH_3$), 0.84 (s, 6H, $2CH_3$) ppm.

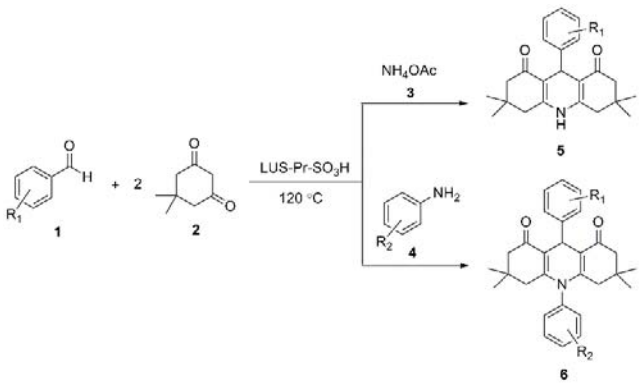
10-(4-bromophenyl)-3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione ***(6c):***

Yellow solid. m.p.= 301-303°C; IR (KBr): $\bar{\nu}$ = 3084, 3053, 3028, 3003, 2955, 2934, 2886, 2868, 1642, 1577, 1489, 1365, 1300, 1276, 1260, 1222, 1176, 1143, 1122, 1068, 1012, 1001, 978, 945, 919, 886, 849, 747, 698, 569, 515 cm^{-1}. ^{1}HNMR (300 MHz, DMSO-d_6): δ= 7.80 (d, *J*=7.6 Hz, 2H, 2CH arom.), 7.19-7.38 (m, 1H, 6CH arom.), 7.08 (t, *J*=6.5 Hz, 1H, 1CH arom.), 5.02 (s, 1H, CH), 2.17 (d, *J*=17.5 Hz, 2H, 2CH), 2.16 (d, *J*=15.9 Hz, 2H, 2CH), 1.98 (d, *J*=16.0 Hz, 2H, 2CH), 1.76 (d, *J*=17.4 Hz, 2H, 2CH), 0.87 (s, 6H, $2CH_3$), 0.70 (s, 6H, $2CH_3$) ppm. EIMS: m/z (rel. int.)= 505 [M+2] (7), 503 [M^+] (8), 428 (70), 426 (72), 347 (10), 155 (13), 77 (100).

3. Results and Discussion

3.1. Synthesis of acridine-1,8-dione derivatives

The synthesis of acridine-1,8-dione derivatives **5** or **6** were achieved via the four component condensation reaction of aromatic aldehydes **1**, two equivalent dimedone **2** and ammonium acetate **3** (or anilines **4**) using LUS-Pr-SO_3H as a heterogeneous acid catalyst (Scheme 1). We first studied a reaction between 3-methoxybenzaldehyde, dimedone and ammonium acetate in the presence of LUS-Pr-SO_3H by the screen of reaction conditions. In order to determine the optimized conditions, we examined the influence of different conditions, as the results show in Table 1. Among the tested solvents such as H_2O, and solvent-free system, the best result was obtained after 2 min at 120°C in solvent-free condition in excellent yield.



Scheme 1. Synthesis of acridine-1,8-dione derivatives in the presence of LUS-Pr-SO_3H.

Table 1. The Optimization of reaction conditions in the synthesis of **5a** using LUS-Pr-SO_3H.

Entry	Solvent	Condition	Time (min)	Yield (%)
1	H_2O	Reflux	4 h	-
2	EtOH	Reflux	4 h	83
3	Neat	140°C	2	98
4	Neat	120°C	2	97
5	Neat	100°C	30	80

After optimizing of reaction conditions, we developed the solvent free condition at 120°C for the synthesis of other derivatives using several aromatic aldehydes and different amine sources, as the results show in Table 2. By these conditions, the reactions were carried out easily to produce acridine-1,8-diones in excellent yields. The experimental procedure is very simple, convenient and has the ability to tolerate a variety of functional groups such as methyl, methoxy, nitro and halides under the reaction conditions. For all substrates, the reaction could be completed in 0.5-3 min with high yields. After completion of the reaction (monitored by TLC), the crude product was dissolved in hot ethanol, the heterogeneous solid catalyst was removed easily by simple filtration, and after cooling of the filtrate, the pure crystals of products were obtained. The products were characterized by melting points, ^{1}HNMR and IR spectroscopic analyses. Melting points are compared with reported values in literature as shown in Table 2.

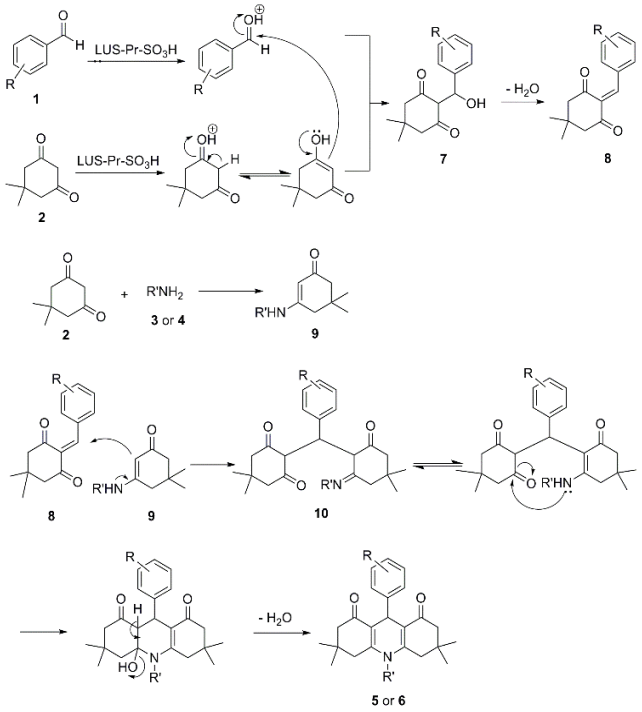
The acid catalyst can be reactivated by simple washing, subsequently with diluted acid solution, water and acetone, and then reused without noticeable loss of reactivity. The reusability of the catalyst was investigated under optimized conditions for the synthesis of the model compound **3a**. The recycling process was completed four times with no significant decrease in the activity of the catalyst. The yields for the four runs were found to be 97%, 92%, 89%, and 85%, respectively.

In these processes, LUS-Pr-SO_3H plays an important role in the accelerating of reaction. A suggested mechanism for this transformation is proposed in Scheme 2. LUS-Pr-SO_3H acts as a source of H^+, which can protonate the carbonyl group to create a more reactive species. In this reaction, intermediate **7** is formed through the Knoevenagel reaction between dimedone and aldehyde, and subsequently olefin **8** is produced by dehydration. Subsequent condensation of the second dimedone **2** and the amine **3** or **4** yielded enamine **9**. Michael addition of enamine **9** to olefin **8** was followed by cyclization and dehydration affords the corresponding products **5** or **6 (a-j)**. The high yields of reactions are attributed to the nano pore effect of solid acid catalyst, which could act as nano-reactor.

Table 2. LUS-Pr-SO_3H catalyzed the synthesis of acridine derivatives **5** and **6** under solvent-free condition.

Entry	R_1	R_2	Product	Time (min)	Yield (%)	m.p. (°C)		Ref.
						Found	Reported	
1	3-CH_3O	-	**5a**	2	97	303-305	287-289	[29]
2	H	-	**5b**	1	95	290-292	290-292	[30]
3	2,4-Cl_2	-	**5c**	0.5	96	321-323	321	[31]
4	3-NO_2	-	**5d**	1.5	96	305-307	307-310	[32]
5	4-Cl	-	**5e**	1	90	304-305	303-305	[31]
6	4-CH_3	-	**5f**	2.5	92	310-312	318-320	[33]
7	2-CH_3O	-	**5g**	2	95	291-293	293-295	[29]
8	4-CH_3O	-	**5h**	3	94	301-303	298-300	[34]
9	2,3-$(CH_3O)_2$	-	**5i**	3	92	326-328	324-326	[32]
10	H	H	**6a**	3	98	248-250	252-254	[29]
11	3-NO_2	H	**6b**	2	93	295-297	297-299	[29]
12	H	4-Br	**6c**	2	88	304-306	269-272	[29]
13	4-CH_3O	4-F	**6d**	3	91	244-246	242-244	[35]
14	H	4-CH_3	**6e**	2.5	89	260-262	262-263	[36]

The syntheses of acridine derivatives have been studied with several conditions in the literature as shown in Table 3. In comparison with other supported sulfonic acids (Entries 8, 9 and 10), the present methodology has very short reaction times, which attributed to the mesoporous silica structure of LUS-1, that could act as nano-reactor.



Scheme 2. Proposed mechanism for the synthesis of acridine derivatives **5** and **6**.

3.2. Antimicrobial activities of 1,8-dioxo-decahydroacridine derivatives

Antimicrobial activities of synthesized 1,8-dioxo-decahydroacridine derivatives were determined using the disc diffusion method. In this research, five microorganisms were used such as gram positive bacteria *Bacillus subtilis* (ATCC 465) and *Staphylococcus aureus* (ATCC 25923), gram negative bacteria *Escherichia coli* (ATCC 25922) and *Pseudomonas aeruginosa* (ATCC 85327) and fungus *Candida albicans* (ATCC 10231). At first 1,8-dioxo-decahydroacridines were dissolved in DMSO (100 µg ml^{-1}), and 25 µl of each solution was loaded onto 6-mm paper discs. On the other hand, 100 µl of a suspension of the microorganisms (109 cell ml^{-1}) was spread on sterile Mueller–Hinton agar plates, and the discs were placed on the surface of these culture plates. The inhibition zone of compounds around disc was compared with three commercial antibiotics such as Chloramphenicol, Gentamicin and Nystatin. Only compound **5e** displayed activity against *B. subtilis* (12 mm), which is lower than those reported for Chloramphenicol (26 mm) and Gentamicin (28 mm).

The minimum inhibitory concentration (MIC) of the synthesized compounds which showed antibiotic activity in disc diffusion tests were also determined by microdillution method [21] to compare with three commercial antibiotics such as Chloramphenicol, Gentamicin and Nystatin. The MIC value of compounds **5e** for the *B. subtilis* (256 µg ml^{-1}) was higher than the range of those reported for Chloramphenicol (4 µg ml^{-1}) and Gentamicin (0.125 µg ml^{-1}) to National Committee for Clinical Laboratory Standards (NCCLS) (2000).

Table 3. Comparison of different conditions in the synthesis of acridine derivatives **5** and **6**.

Entry	Catalyst	Solvent	Condition	Time	Yield (%)	Ref.
1	HY-zeolite	EtOH	Reflux	2.5-3.5 h	70-90	[37]
2	-	2,2,2-Trifluoroethanol	70°C	3 h	95-98	[38]
3	NH_4Cl	H_2O	Reflux	2-3 h	86-96	[39]
4	$Zn(OAc)_2$	H_2O	Reflux	2-3 h	84-94	[39]
5	*L*-Proline	H_2O	Reflux	2-3 h	82-97	[39]
6	[Bmim]Br	-	90°C	15-90 min	85-97	[40]
7	Nano TiO_2	EtOH	Reflux	60-105 min	82-91	[41]
8	SBSSA[a]	EtOH	Reflux	1-4.5 h	84-96	[17]
9	SBNPSA[b]	EtOH	Reflux	2-5 h	86-93	[42]
10	SiO_2-Pr-SO_3H	-	120°C	2 h	82-95	[32]
11	LUS-Pr-SO_3H	-	120°C	0.5-3 min	88-97	This work

[a]Silica-Bonded S-Sulfonic Acid.
[b]Silica-bonded N-Propyl Sulfamic Acid.

3.3. Synthesis and functionalization of LUS-1

The mesoporous compound LUS-1 was prepared and functionalized according to a previously reported method [43]. The catalyst was analyzed by different methods such as TGA, XRD, BET and SEM methods, which have confirmed that propyl sulfonic acid groups were immobilized into the pores [43].

Fig. 2 shows the thermal gravimetric analysis (TGA) of LUS-Pr-SO_3H which has two major decomposition states, the first state is below 100°C (8% weight loss), which corresponded to loss of surface water and other mass loss between 200 °C and 600°C (15% weight loss), assigned to the loss of organic groups grafted onto LUS-1. From the weight loss in TGA diagram the concentration of immobilized acid can be estimated as 1.3 mmol g^{-1}.

The volumetric and porosity characteristics of the LUS-1 and LUS-Pr-SO_3H were measured by volumetric analyses. The "Type IV" N_2 adsorption-desorption isotherms with "H1-type" hysteresis for condensation and evaporation steps in both materials (LUS-1 and LUS-Pr-SO_3H) are characteristic of periodic mesoporous materials (Fig. 3).

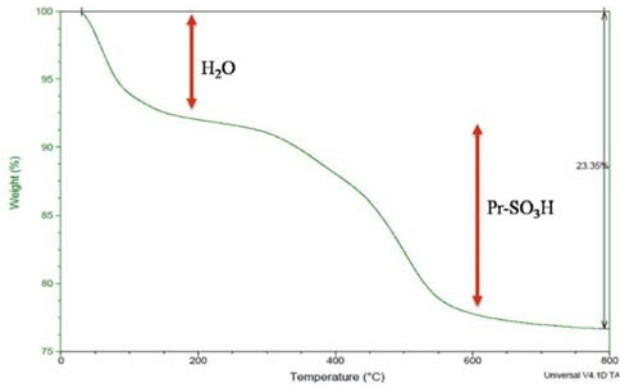


Fig. 2. TGA diagram of LUS-Pr-SO_3H.

Table 4 shows the texture properties of LUS-Pr-SO_3H calculated by the BET method such as, surface area (534 m^2g^{-1}), pore volume (0.545 cm^3g^{-1}) and average pore diameter (2.4 nm) which are smaller than those of LUS-1 and indicates that organic groups were grafted into the pore walls.

Fig. 4 illustrates low-angle XRD pattern (Left) and the SEM images (Right) of LUS and LUS-Pr-SO_3H. XRD pattern showed the same ordered mesoscopic structured silica with (100), (110) and (200) reflections for both materials, which exhibit a two-dimensional hexagonal symmetrical array of nano-channels. It means the structural nature of LUS-1 did not change during the surface modifications. On the other hand, the SEM image of both materials shows the same morphology. It means the grafting of organic group did not affect the morphology of solid.

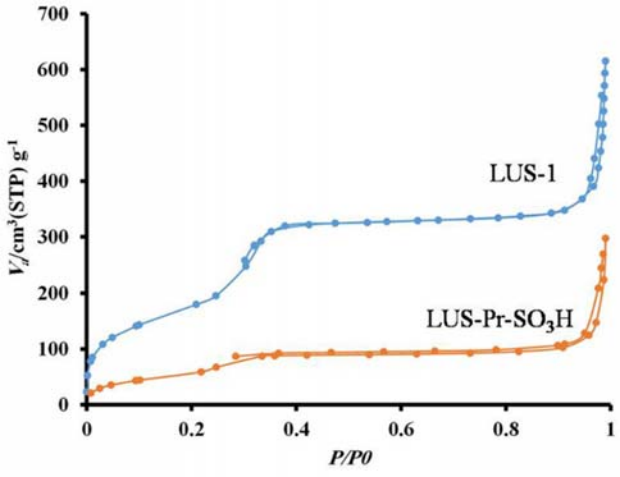


Fig. 3. Nitrogen adsorption-desorption isotherms of LUS-1 and LUS-Pr-SO_3H.

Table 4. Porosimetery values for LUS-1 and LUS-Pr-SO_3H.

	Surface area (m^2g^{-1})	Pore volume (cm^3g^{-1})	Pore diameter (nm)
LUS-1	870	0.841	2.9
LUS-Pr-SO_3H	534	0.545	2.4

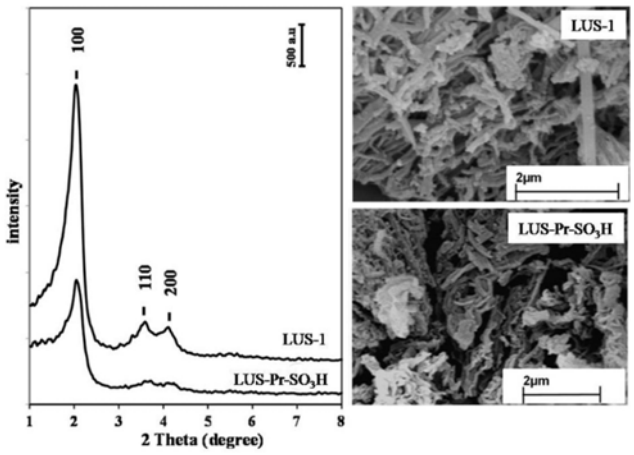


Fig. 4. X-ray diffraction pattern (Left) and SEM images (Right) of LUS-1 and LUS-Pr-SO_3H.

4. Conclusions

In conclusion, the efficient acidic functionalization of mesoporous silica LUS-1 and its application as a recyclable and environmentally benign catalyst for one-pot synthesis of 1,8-dioxo-decahydroacridine derivatives has been reported. The reactions were carried out easily to produce acridine-1,8-diones in very short times and excellent yields. The catalyst could be recovered and reused for several times without considerable loss of reactivity. Antibacterial and antifungal activities of synthesized compounds were measured against *Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, *Pseudomonas aeruginosa* and *Candida albicans.* From these compounds only compound **5e** displays activity against *Bacillus subtilis*.

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