

# A Facile and Green Synthesis of 2,4,6-Triarylpyridine Derivatives Using the Modified Mesoporous Organic Polymer Based on Calix [4]Resorcinarene: As an Efficient and Reusable Heterogeneous Acidic Catalyst

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**Abstract**—The work describes an efficient one-pot synthesis of 2,4,6-trisubstituted pyridine derivatives through a three-component catalytic reaction. The procedure involves mesoporous organic polymer based on calix[4]resorcinarene in and functionalized by N-propyl sulfamic acids a solid acid catalyst. Excellent yields, short reaction times, catalyst reusability, and easy reaction handling under solvent-free and mild acidic conditions are the most important advantages of the present protocol. The acidic catalyst was characterized by a number of spectroscopic methods such as Fourier-transform infrared spectroscopy, field emission scanning electron microscopy, transmission electron microscopy and elemental CHNS analysis.

**Keywords:** sulfamic acid functionalized mesoporous organic polymer, calix[4]resorcinarene, multi-component reactions, 2,4,6-trisubstituted pyridines, solid acid catalysts

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## INTRODUCTION

Multi-component reactions emerge as an attractive strategy for organic synthesis. It is an advantage to conduct several reactions in a single reactor without the need to use isolation and purification steps. Operation simplicity that does not involve any separation process and detection of intermediates reduces time, labour and cost [1]. Accordingly, these reactions are widely used for the synthesis of a wide variety of heterocyclic compounds, among which pyridine derivatives [2–8] are of significant interest owing to their key role in pharmaceuticals and agrochemicals production [9–11].

Over the recent years, synthetic porous organic frameworks (POFs) with micro (<2 nm) and meso (2–50 nm) pore size range have received an increasing attention in a wide range of applications such as heterogeneous catalysis, organic synthesis, chemical sensing, light harvesting, gas storage and separation [12–17]. In this area, several different classes of porous materials such as hyper-cross-linked polymers, poly-

mers of intrinsic microporosity, covalent organic frameworks, and porous organic polymers with low or high crystallinity were used [18–23]. Among different types of mesoporous materials, considerable attention has been given to design and synthesis of mesoporous organic polymers that are composed of light and non-metallic elements like C, H, O, and N. These materials are of intense interest as catalysts or catalyst supports because they combine textural properties of mesoporous materials such as large surface areas, low densities and tuneable structures with the advantages of organic polymers that contain aromatic fragments and show high thermal and chemical stability and high degree of hydrophobicity. The porous materials can be pre-synthetically or post-synthetically functionalized with a wide range of functionalities to make them promising candidates for heterogeneous catalysts in various types of chemical reactions [24–26].

Solid acids are recommended as suitable catalytic systems in organic synthesis because they have a number of advantages over their homogeneous counterparts. Non-toxicity, long catalytic life, environmental

compatibility, low cost, ease of isolation and reusability are the most important properties of these heterogeneous acidic systems [27–29]. In this way, polymeric supports can be equipped with organic spacers and the addition of the acidic agent to these active sites will result in the solid acid catalyst formation [30, 31]. Accordingly, the addition of acidic functional groups like sulfamic acid moieties into solid supports is one of the ways to produce efficient heterogeneous catalysts [32–35].

Polycalix [4]resorcinarene, which is composed of monomeric units of cupped hydrophobic cavities of calixarene molecules surrounded by hydrophilic groups, is among the novel mesoporous organic polymers amenable to post-synthetic functionalization for catalytic purposes [36].

This work is a continuation of our studies aimed at developing safer methods in organic synthesis [37–42]. It was of interest to explore the possibility of anchoring  $\text{SO}_3\text{H}$ -entities into our novel  $\text{NH}_2$ -functionalized mesoporous polymer based on calix [4]resorcinarene to produce a promising solid acid catalyst. We hoped that this catalyst would be suitable for the preparation of 2,4,6-trisubstituted pyridine derivatives through a one-pot reaction between acetophenone, aryl aldehydes and  $\text{NH}_4\text{OAc}$  under mild and solvent-free conditions.

## EXPERIMENTAL

### *Materials and Methods*

Commercially available starting materials were purchased from Merck, Fluka and Aldrich chemical companies and used without further purification. TLC accomplished monitoring of the reaction progress. IR spectra were recorded using a BOMEM MB-102 FT-IR spectrophotometer (Hartmann & Braun, Canada, KBr).  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectra were obtained on a Bruker NMR spectrometer (Bruker, Germany) at 400 and 100 MHz, respectively, in  $\text{CDCl}_3$  with tetramethylsilane as an internal standard. Mass spectra were acquired on an Agilent 5975 mass spectrophotometer (Agilent, USA). Melting points were checked with a Thermo Scientific 9200 melting point apparatus (Thermo Scientific, USA), whereas pH measurements were recorded on a Metrohm 827 pH lab pH-meter apparatus (Metrohm AG, Zwisserland). Polymeric matrix morphology was examined by atomic force microscopy (AFM) with Nano Wizard II AFM microscope (JPK instruments, Germany). Field emission scanning electron microscopy (FE-SEM) images were collected on a MIRA 3 TESCAN apparatus (TESCAN, Czechia). The particle morphology and porosity were also evaluated using transmission electron microscopy (TEM) technique on a Zeiss-EM 10C (Germany) in 80 kV. In order to evaluate the thermal stability of the functionalized polymer, its TGA curves were recorded on a NETZSCH STA 409

PC/PG (NETZSCH, Germany) under a nitrogen atmosphere (rate of  $\text{N}_2 \sim 1 \text{ L/h}$ ). Elemental analysis was recorded on a Flash EA 1112 CHNS analyzer (Thermo Finnigan, Italy).

### *Synthesis of Calix [4]Resorcinarene 1*

Calix[4]resorcinarene **1** was prepared according to the previously reported procedure [43].

### *Synthesis of the 3D-Network Polymer Based on Calix [4]Resorcinarene 2*

The desired polymer was synthesized by adding 42 mmol of formaldehyde to 14 mmol of the prepared calix [4]resorcinarene **1** dissolved in 40  $\text{cm}^3$  aqueous NaOH solution (10%) at room temperature. The resultant mixture was heated to  $90^\circ\text{C}$  and was maintained at this temperature for 20 h. Next, the excess alkali was washed out of the gel formed with cold water. The gel was allowed to stand at  $100^\circ\text{C}$  for 1 h. Then, the gel was transformed into the acidic form by being treated with the 0.1 M HCl solution. The resulted solid was dried at  $100^\circ\text{C}$  for 10 h [48].

### *Synthesis of the $\text{NH}_2$ -Functionalized Polymer Based on Calix [4]Resorcinarene 3*

The synthesized polymer **2** obtained from the above procedure (1.00 g) and 3-(triethoxysilyl)propylamine (4.00 g, 18 mmol) were introduced into a 50  $\text{cm}^3$  round-bottomed flask containing 10  $\text{cm}^3$   $\text{H}_2\text{O}/\text{EtOn}$  at room temperature. The reaction mixture was heated to  $90^\circ\text{C}$  and stirred at this temperature for 20 h. The desired amine-functionalized polymer **3** was collected by filtration and washed several times with hot toluene before being dried at  $100^\circ\text{C}$  [45].

### *Determination of the Amount of Amine Functionalities Grafted onto the Polymer 2*

The amount of amine functionalities grafted onto the polymer **2** was determined by elemental analysis to be 3.5 mmol per gram of dry polymer [45].

### *Preparation of Polymer-Bonded N-Propyl Sulfamic Acid Based on Calix [4]Resorcinarene 4*

To a magnetically stirred mixture of the  $\text{NH}_2$ -functionalized polymer described in the previous section (5 g) in  $\text{CHCl}_3$  (20  $\text{cm}^3$ ) at  $0^\circ\text{C}$ , chlorosulfonic acid (5  $\text{cm}^3$ , 75.2 mmol) was added dropwise over 2 h. After the addition was completed, the mixture was stirred for 3 h until all HCl was removed from the reaction medium. The mixture was filtered, washed with ethanol (30  $\text{cm}^3$ ) and finally dried at room temperature.

### Determination of the Capacity of Sulfamic Acid Functionalities Grafted onto the Catalyst **4**

The capacity of sulfamic acid functionalities in the polymer-supported catalyst was determined by elemental analysis. The capacity was generally found to be 1.55 mmol H<sup>+</sup> per gram of the dry polymer-supported catalyst **4**.

In addition, the capacity of the acidic functionalities was determined through pH analysis. To this end, to an aqueous solution of NaCl (1 M, 25 cm<sup>3</sup>) with a primary pH equal to 6.01, catalyst **4** (50 mg) was added and the resulting mixture was stirred for 2 h after which the pH of the solution decreased to 2.49. This is equal to a loading of 1.62 mmol SO<sub>3</sub>H per gram of dry polymer [46].

### General Procedure for the Synthesis of 2,4,6-Trisubstituted Pyridine Derivatives **8a–g**

A mixture of aromatic aldehyde (1 mmol), acetophenone (2 mmol), NH<sub>4</sub>OAc (1.3 mmol, 0.103 g) and 0.05 mmol catalyst (0.025 g) was stirred in a sealed tube at 60°C. Upon the reaction completion monitored by TLC, hot ethanol was added to the mixture and the insoluble catalyst was filtered off. Pure products were obtained by recrystallization from hot ethanol.

2,4,6-Triphenylpyridine (**8a**): white crystals, yield 90%, m.p. 134–136°C (from EtOH), MS *m/z*: 307

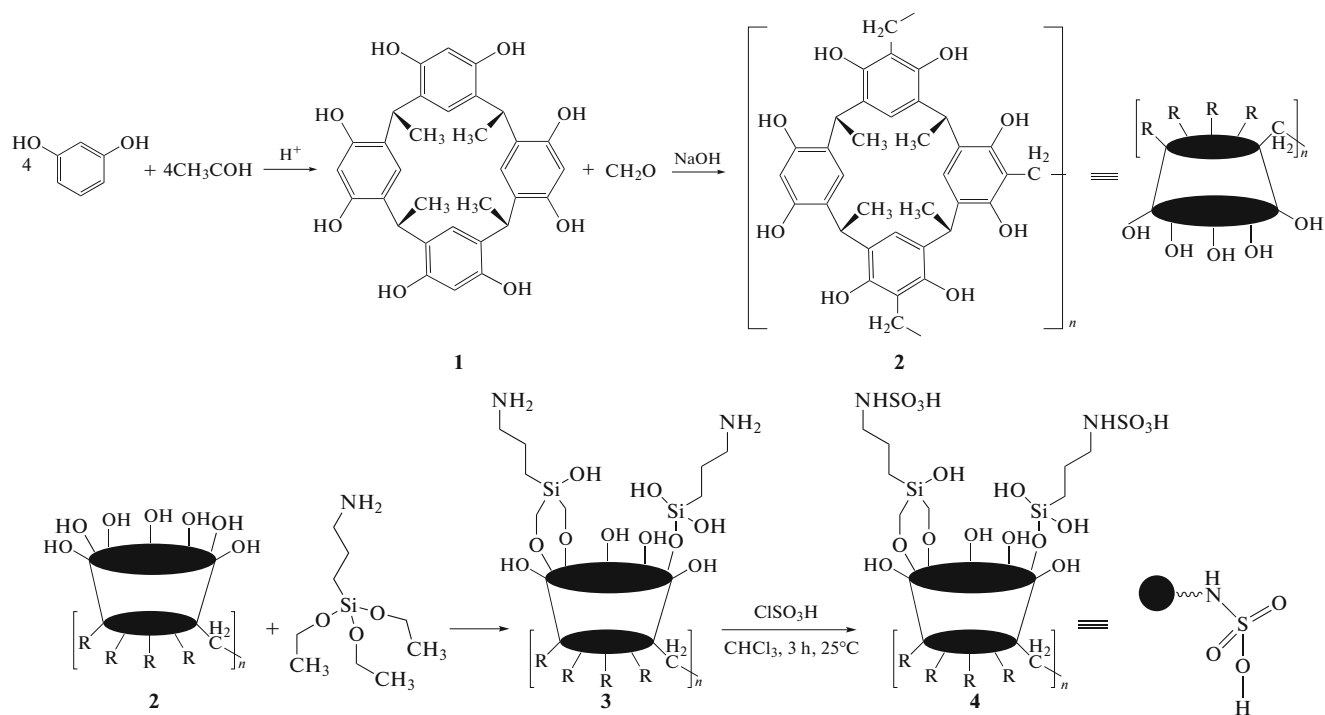
[M<sup>+</sup>]; <sup>1</sup>HNMR (400 MHz, CDCl<sub>3</sub>), δ, ppm: 7.47–7.77 (m, 11H, Ar–H), 8.22–8.24 (m, 4H, Ar–H), 7.92 (s, 2H, H<sup>1</sup>); <sup>13</sup>CNMR (100 MHz, CDCl<sub>3</sub>), δ, ppm: 117.2 (C3, C5), 127.2, 128.7, 129.0, 129.1 (ArC), 139.1, 139.5 (ArCq), 150.1 (C4), 157.4 (C2, C6).

### Recyclability of Catalyst **4**

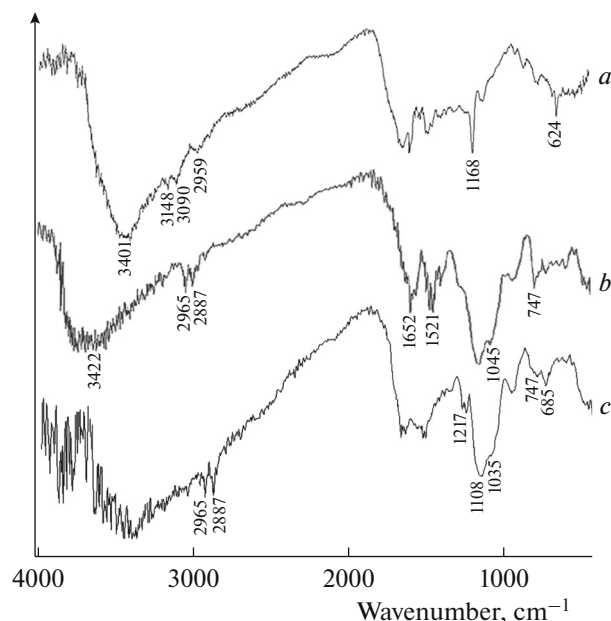
At the end of the reaction, the catalyst was filtered, the residue was consecutively washed with CH<sub>2</sub>Cl<sub>2</sub> (5 cm<sup>3</sup>), Et<sub>2</sub>O (5 cm<sup>3</sup>), EtOH (5 cm<sup>3</sup>), and *n*-hexane (5 cm<sup>3</sup>). After the catalyst had been dried in an oven at 60°C for 4 h, it could be reused directly without further purification.

## RESULTS AND DISCUSSION

Immobilization of SO<sub>3</sub>H—acidic group on highly efficient mesoporous materials provides the system that combines the advantages of homogeneous acidic catalysts with those of heterogeneous systems. In this project, attention was focused on the catalytic activity of the novel porous organic polymer based on calix [4]resorcinarene functionalized by sulfamic acid. The preparation of 2,4,6-trisubstituted pyridine derivatives served as a model reaction. The multi-step functionalization process involved in the catalyst preparation is designated in Scheme 1.



**Scheme 1.** Synthesis of calix [4]resorcinarene **1**, 3D-network polymer **2**, amine-functionalized polymer **3** and the N-propyl sulfamic acid functionalized polymeric catalyst **4**.



**Fig. 1.** The comparative FT-IR spectra for polymeric network **2** (a), amine functionalized polymer **3** (b), catalyst **4** (c).

The 3D-network polymer **2** was produced by the reaction between resorcinol and acetaldehyde, followed by the polycondensation of calix [4]resorcin-arene **1** with formaldehyde as designed in Scheme 1. The structure of compound **1** was determined unambiguously by the spectroscopic (IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR) data (see Electronic Supporting materials). The formation of polymer **2** was also confirmed by atomic force microscopy (AFM), X-ray diffraction (XRD) and scanning electron microscopy (SEM) (see Electronic Supporting materials). Subsequently, according to the schematic procedure describing the first step of post-functionalization, the prepared porous polymer **2** was functionalized through reacting with 3-(triethoxysilyl)propylamine as an organic spacer. In this way proper positions accessible to acidic agents can be created. The structure of the synthesized

$\text{NH}_2$ -functionalized polymer **3** was characterized through a series of analytical methods including IR spectroscopy, atomic force microscopy (AFM), field emission scanning electron microscopy (FE-SEM), thermal gravimetric analysis (TGA) and energy-dispersive X-ray spectroscopy (EDS) (see Electronic Supporting materials). In the following step,  $-\text{SO}_3\text{H}$  groups were anchored to the polymeric matrix **3** via the addition of chlorosulfonic acid to the reaction medium.

This new reported heterogeneous sulfamic acid functionalized catalyst **4** was prepared by the reaction of previously synthesized  $\text{NH}_2$ -functionalized polymer **3** and chlorosulfonic acid in chloroform. It can be postulated that a strong basicity of amine functional groups towards acidic reagents like chlorosulfonic acid is a driving force of intense acidic-basic interaction that plays a decisive role in the preparation of the catalyst **4**.

According to the IR spectral analysis of the acidic catalyst **4**, the N–S the band due to stretching vibrations was observed at  $685\text{ cm}^{-1}$ . The bands assignable to Si–O–Si symmetric and asymmetric stretching vibrations appeared at  $1035$  and  $747\text{ cm}^{-1}$ . The bands at  $1217$  and  $1124\text{ cm}^{-1}$  can be assigned to sulfonyl moieties in catalyst **4**. The presence of bending vibrations related to amine functionalities suggests the occurrence of amine groups, which avoided transformations into sulfamic acids (Fig. 1).

The sulphur content obtained from the elemental analysis showed a loading of  $1.55\text{ mmol H}^+/\text{g}$  on the polymeric matrix (Table 1).

In order to determine the percentage of amine groups converted to the corresponding sulfonic acid groups, pH analysis was carried out through a pH-meter apparatus and a loading equal to  $1.62\text{ mmol SO}_3\text{H}$  per  $1\text{ g}$  of the solid acid catalyst at  $25^\circ\text{C}$  was found. This value represents the catalyst proton exchange capacity, which is in good accordance with CHNS analysis results.

**Table 1.** Elemental analysis data for polymer **2**,  $\text{NH}_2$ -functionalized polymer **3** and the sulfamic acid functionalized catalyst **4**

Sample	C, wt %;	H, wt %	N, wt %	S, wt %
Polymer <b>2</b>	45.720	4.960	0.030	—
$\text{NH}_2$ -functionalized polymer <b>3</b>	45.500	5.020	4.950	—
Sulfamic acid functionalized catalyst <b>4</b>	44.429	6.071	5.001	4.782

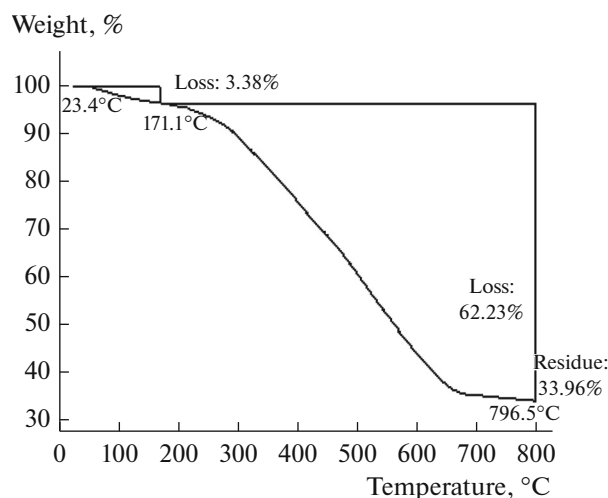


Fig. 2. TGA profile of the synthesized catalyst 4.

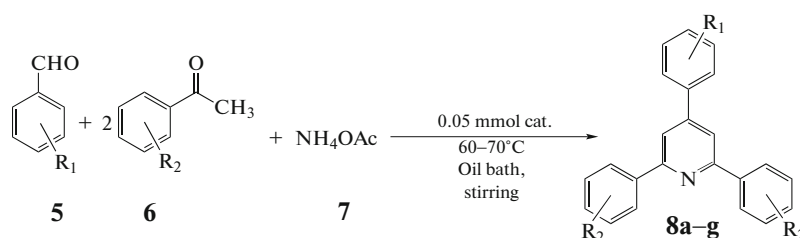
The thermal stability of the prepared catalyst was accessed by TGA measurements under  $N_2$  atmosphere. A mass loss of about 3% up to 172°C is due to the removal of water and residual amounts of solvents trapped inside the pores during the synthesis procedure. The second mass loss of about 62%, which occurs in the temperature range of nearly 172 to 796°C is mainly pertain to the cleavage of the grafted linkers and organic moieties. The retained mass of about 34%, which is attributed to the remained polymeric

architecture, is stable up to 800°C. These results achieved via TGA technique shows high stability of the synthesized structure (Fig. 2).

Field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM) techniques were employed to examine the morphology of this new developed polymeric system. As illustrated in Fig. 3, the obtained structure is consisted of meso-scale pores and spherical particles that are reminiscent of the morphology of the initial polymeric network (Figs. 3a and 3b).

As shown in Fig. 4, nitrogen adsorption–desorption isotherm of the new sulfamic acid functionalized catalyst is classified as nearly type IV isotherm with surface area, pore volume and mean pore diameter equal to 199  $m^2/g$ , 0.023  $cm^3/g$  and 4.67 nm, respectively.

In order to investigate the catalytic efficiency of novel catalyst, a three-component reaction between benzaldehydes, acetophenone and  $NH_4OAc$  was elected as the model reaction for the initial screening experiments (Scheme 2). To illustrate the usefulness of the catalyst for this reaction, firstly the reaction was carried out without the catalyst. In this case, as it was anticipated the reaction did not proceed properly (Table 2, entry 6). In the continuation of our research efforts, we tried to optimize the reaction conditions in the presence of the novel catalyst. A brief report of the results of the optimized parameters is depicted in Table 2.



Scheme 2. Synthesis of 2,4,6-trisubstituted pyridines

At the next step, different parameters of the reaction such as the catalyst amount, time and temperature were optimized. We were able to found that an increase in the catalyst quantity above 0.025 g did not appreciably affect the yield of products. Consequently, 0.025 g was selected as the optimum amount of catalyst (Table 2, entry 2). In the following, the optimum temperature was obtained in the range of 60–70°C.

It is found that the best yield was obtained using 5 mol % of catalyst 4 within 10 min in 60–70°C under solvent-free conditions (Table 2, entry 5).

Unexpectedly, the reaction was completed in a short period of time with high yields. This result can be attributed to the specific porous structure and high surface area of the catalyst which can facilitate the transportation of the reactants to the exposed active sites and consequently increase the reaction yields. In addition, the aromatic hydrophobic cavities of polycalixarene monomeric units provide proper pockets to accumulate the reactants and promote the reaction efficiency.

At the next step, as shown in Table 3, an attempt was made to evaluate the scope and limitations of the



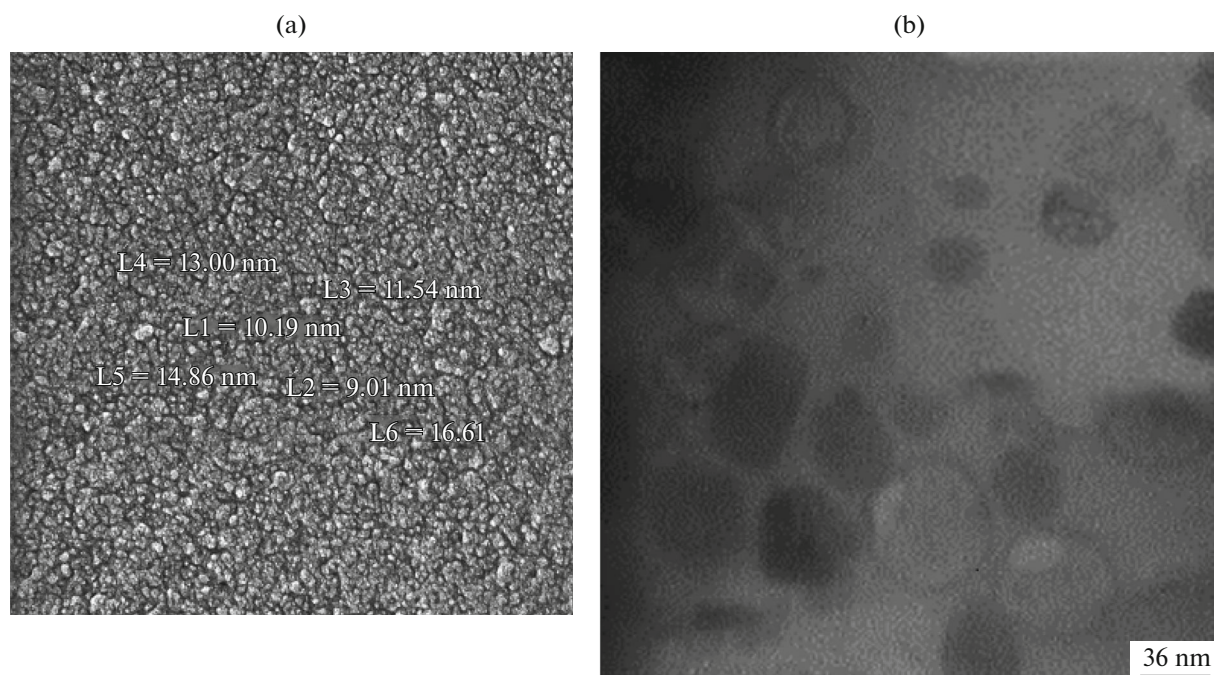


Fig. 3. FE-SEM (a) and TEM (b) images of the synthesized catalyst **4**.

methodology. The reaction was carried out under optimized conditions for a variety of substituted aromatic aldehydes and in each case, the corresponding 2,4,6-trisubstituted pyridines were obtained in good to excellent yields. The remarkable point is that aromatic aldehydes bearing electron-withdrawing groups (such

as  $\text{NO}_2$  and haloides, Table 3, entries 5, 6, and 7) at *para*-position of the aromatic ring afforded higher yields than those with electron-donating groups (such as  $\text{CH}_3$ ,  $\text{OCH}_3$ , and  $\text{N}(\text{CH}_3)_2$ ) (Table 3, entries 2, 3, and 4) in this position. At the same time, there is no adverse effect of substituent either electron-withdrawing or electron-donating groups on the product yield (Table 3). All the products are known compounds and they were characterized by comparing their IR and  $^1\text{H}$ -NMR spectral data as well as melting points with those reported in literature [2, 47].

It is worth noting that the final reaction products were easily purified through recrystallization without any need to use costly and perilous chromatographic methods.

The plausible mechanism for the formation of 2,4,6-triarylpyridine derivatives is designated in Scheme 3. A series of processes including Aldol condensation, Michael addition, cyclization and oxidation eventually result in the desired product **8**. Initially, the activated aryl aldehyde **5**, in the presence of the catalyst **4** undergoes nucleophilic addition by the enol form of acetophenone **6** to give rise to the aldol condensation product **A**. Then the second mole of acetophenone, in the presence of ammonium acetate is converted to the enamine molecule **B**, followed by attacking **A** in a Michael addition affording intermediate **C**. In the next step, dihydropyridine **D** is formed by cyclization and finally air oxidation of **D** gives the final product **8** (Scheme 3).

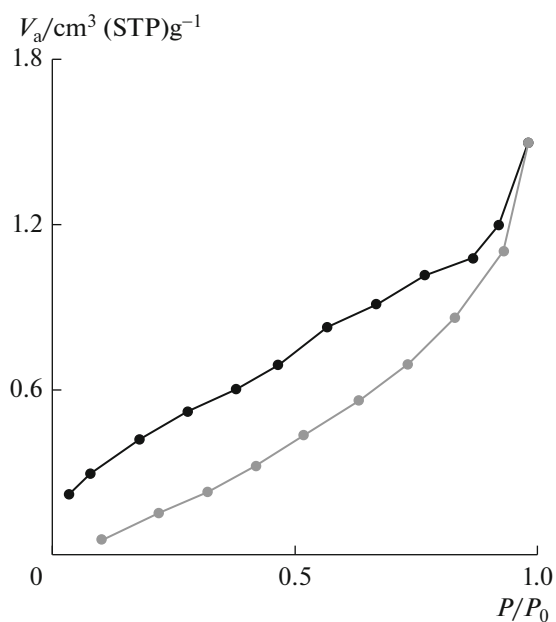


Fig. 4. Nitrogen adsorption-desorption isotherm of N-propyl sulfamic acid functionalized polymeric catalyst **4**.

**Table 2.** Optimization of the reaction conditions

Entry	Catalyst amount, mmol	Temp, °C	Time, min	Yield, %*
1	0.025	100	15	85
2	0.050	100	10	92
3	0.075	100	10	90
4	0.050	25	120	—
5	0.050	60–70	10	90
6	—	120	120	Negligible

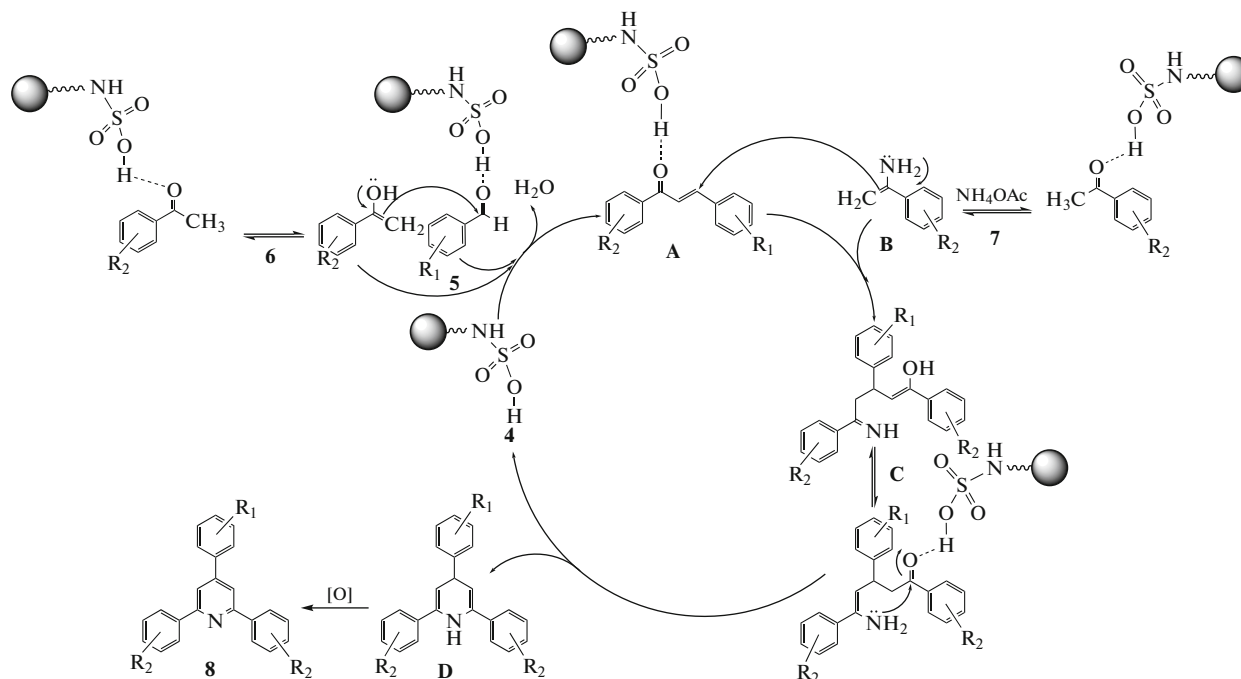
\* Reported yields.

**Table 3.** One-pot synthesis of 2,4,6-trisubstituted pyridines\*

Entry	R <sup>1</sup>	R <sup>2</sup>	Product	Yield, %**	Time, min	T <sub>m</sub> , °C (T <sub>m</sub> , °C [Ref])
1	H	H	<b>8a</b>	90	10	134–136 (134–135 [47])
2	4-Methyl	H	<b>8b</b>	85	15	122–124 (123–124 [47])
3	4-Methoxy	H	<b>8c</b>	82	20	99–101 (100–103 [47])
4	4-N,N-dimethyl amine	H	<b>8d</b>	88	5	141–143 (138–140 [47])
5	4-Chloro	H	<b>8e</b>	98	5	126–128 (125–127 [47])
6	4-Bromo	H	<b>8f</b>	96	10	166–168 (164–166 [2])
7	4-Nitro	H	<b>8g</b>	95	5	198–200 (195–197 [47])

\* Reaction conditions: aromatic aldehyde (1 mmol), acetophenone (2 mmol), NH<sub>4</sub>OAc (1.3 mmol), catalyst (0.05 mmol), 60–70°C, solvent-free.

\*\* Isolated yields.

**Scheme 3.** Plausible mechanism for the synthesis of 2,4,6-trisubstituted pyridines catalyzed by catalyst **4**.

As seen in Table 4, catalyst **4** emerges as a more efficient catalyst in comparison with the previously

known catalytic systems in terms of the product yields and also the reaction time and conditions.

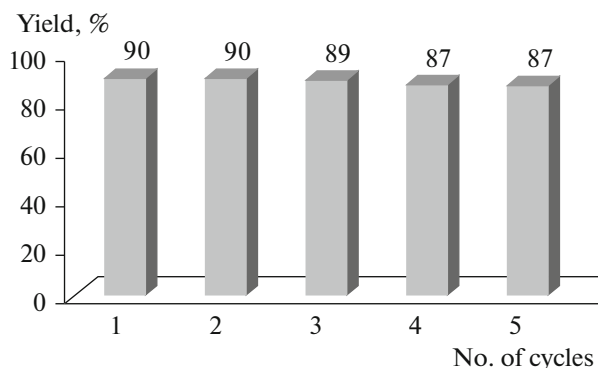
**Table 4.** The comparison of this work catalyst with reported catalysts in the synthesis of triarylpyridines

Catalyst	Time, h	Temperature, °C	Yield, %*	Ref.
Montmorillonite K10 clay catalyst	4	120	97	[48]
Mesoporous nanocrystalline $\text{MgAl}_2\text{O}_4$	3	120	85	[1]
AcOH	4	100	97	[47]
$\text{H}_2\text{SO}_4\text{--SiO}_2$	3	120	87	[49]
$\text{AlPO}_4$	4	120	87	[50]
Catalyst <b>4</b>	0.17	60	90	This work

\* 2,4,6-Triphenylpyridine as the base product in all reported yields.

Finally, the recycling potential of the catalyst was examined. For this purpose, upon the completion of the reaction, the catalyst was recovered by simple filtration through a sintered glass-bed, washed with dichloromethane, diethyl ether, ethanol and *n*-hexane and after being dried it was reused in 5 consecutive runs without considerable decrease in catalytic activity (Fig. 5).

A noteworthy synthetic advantage of this facile cost-effective one-pot reaction conducted under solvent-free conditions is that it can be accomplished with readily available starting materials and with a simplified work up and purification. The synthesis affords excellent product yields and does not involve any need to separate and employ hazardous materials or column chromatography. These are the main advantages of this ecofriendly multi-component protocol in the synthesis of triarylpyridine derivatives. In addition, the catalyst can be recycled for at least five consecutive runs without appreciable loss in the product yields.

**Fig. 5.** Recyclability of the catalyst **4**.

## CONCLUSIONS

In summary, applying this new porous organic polymer as a heterogeneous catalytic system in the three-component synthesis of multi-substituted pyridines resulted in the reaction that can be accomplished in a short span of time through solvent-free conditions with excellent yields of products. Additionally, simple work-up in products isolation, mild reaction conditions, a green methodology and also the catalyst recyclability are the most noteworthy features of this novel procedure.

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