Solid acid catalyst TS-1 zeolite-assisted solvent-free one-pot synthesis of poly-substituted 2,4,6-triaryl-pyridines

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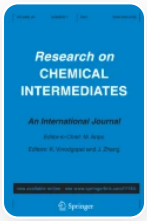

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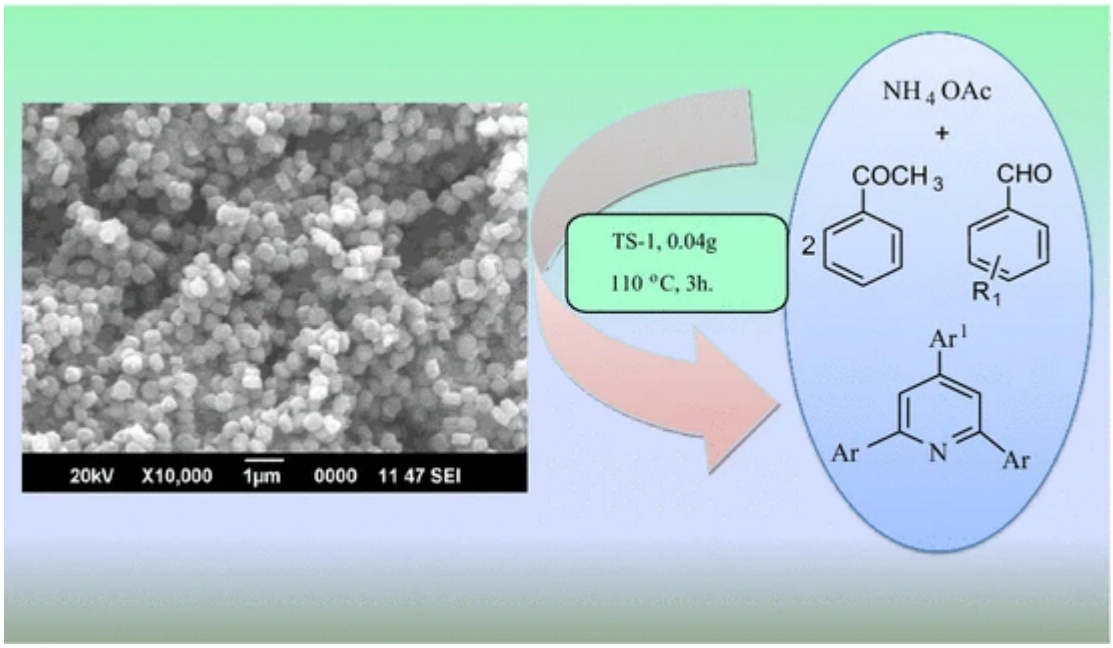
Abstract

A new method is described for one-pot solvent-free synthesis of 2,4,6-triaryl pyridines in the presence of a solid acid catalyst, titanium silicate (TS-1) via cyclocondensation of acetophenone, aryl aldehyde and ammonium acetate. The present method illustrates several advantages, such as eco-friendly reaction conditions, simplicity, short reaction time (3 h), easy separation of catalyst and high yields of the products (85–93%). Furthermore, the TS-1 catalyst was reused for four catalytic cycles with consistent catalytic activity.

Graphical Abstract



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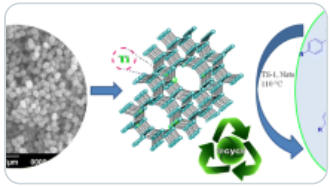

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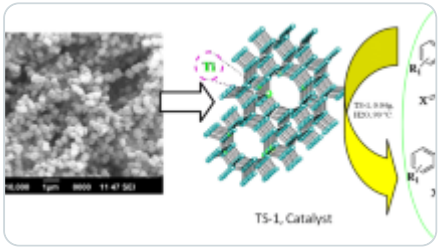

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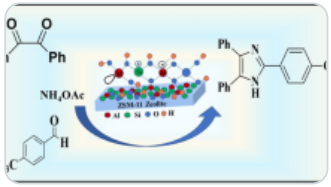

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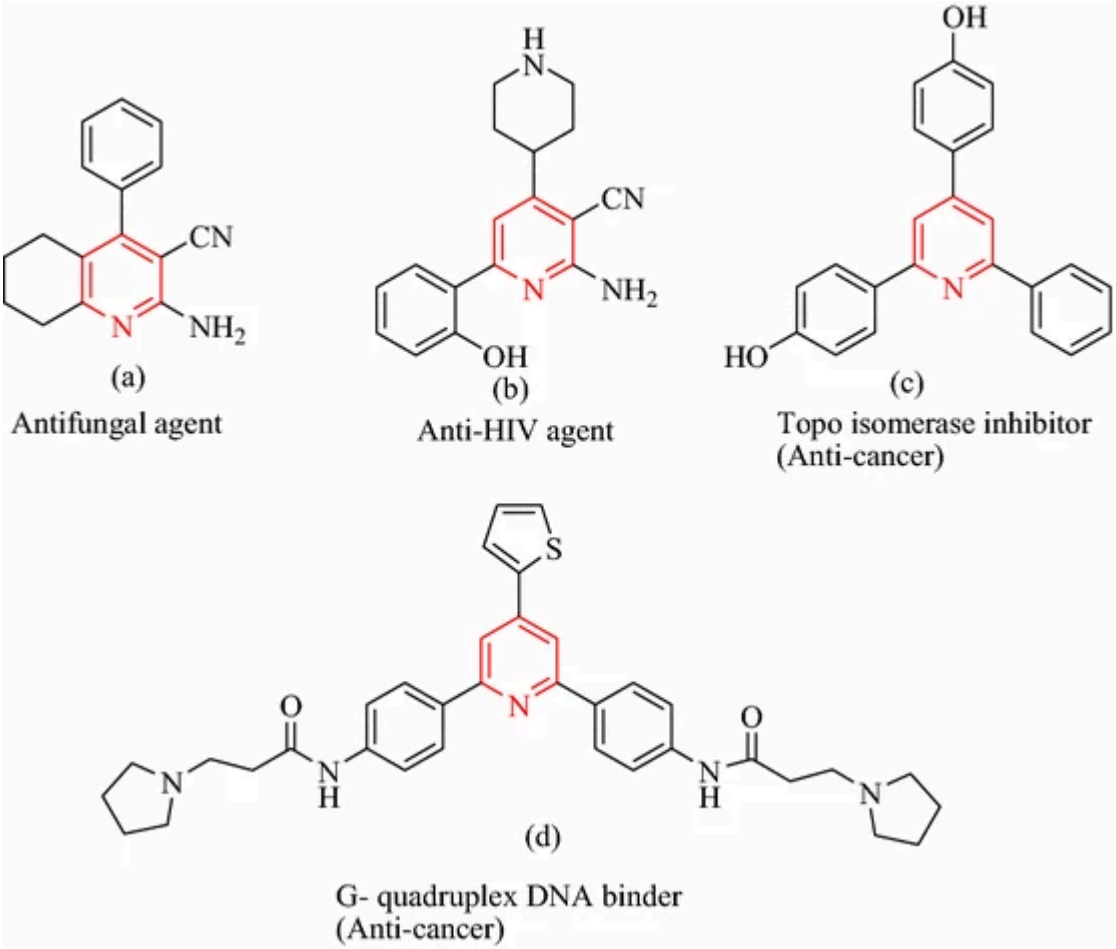
Introduction

The one-pot synthesis method has played a significant role in organic synthesis because of its several advantages such as atom economy, high selectivity, minimum reaction time and least by-products as compared to classical synthetic methods during last two decades, [1]. Nitrogen-containing heterocyclic compounds such as 2,4,6-triaryl pyridine have proved to be important building blocks of alkaloids and natural products [2]. They have also been found to possess biological and pharmaceutical activity such as anti-oxidant, anticancer, antibacterial, antiparasitic and antitubercular [3,4,5]. Some substituted pyridine derivatives show antifungal (a), anti-HIV (b), anticancer (c) and DNA binding activities (d), while dihydroxylated 2,6-diaryl-4-chlorophenyl pyridines are topoisomerase inhibitors and show anitcancer activity [6,7,8].



They also show fluorescence properties and are used in thin film vortex fluidic device development [9]. Additionally, 2,4,6-triaryl pyridine derivatives have a unique position in medicinal chemistry, being building block synthons of supra-molecular chemistry, because of their π staking and H-bonding ability [10,11,12]. Considering these wide and

versatile applications, several workers have developed catalytic methods for the synthesis of 2,4,6-triaryl pyridine derivatives, such as heteropolyacid $H_{14}[NaP_5W_{30}O_{110}]$, MIL 101-$SO_3H$, ionic liquid, $BiCl_3$, bismuthtriflate, ZnO, $AlPO_4$ and $MgAl_2O_3$ with acetic acid [13,14,15,16,17,18,19,20], titanium-supported sulfated silica with toluene, and montmorilonite K10 clay [21,22,23]. Although the reported methods are quite efficient, some of them suffer from one or more limitations, such as longer reaction times, harsh reaction conditions, carcinogenic or volatile organic solvents, low yields and the use of expensive catalysts. It has also been found that some of the reported catalysts are not reusable, while a few methods require specific reaction conditions, such as microwave and sonication in the homogenous phase, like KOH in DMSO [24,25,26]. Some chemical transformations have required a higher temperature to synthesize the desired product from acetophenone with epoxy styrene [27]. Hence, to overcome these limitations, it is imperative on the part of researchers to find a sustainable, efficient, eco-friendly and cost-effective protocol for the synthesis of 2,4,6-triaryl pyridine derivatives.

Titanium silicate (TS-1) zeolite has been found to be an alternative efficient heterogeneous solid acid catalyst with unique properties, such as high surface area, thermal stability, low toxicity, reusability, ion exchange ability and ease of handling, and have hence been used in various industrial processes such as oxidation, epoxidation, alkylation, acylation and cyclic condensation reactions [28,29,30,31,32,33]. In view of the ever-increasing importance of green/sustainable synthetic protocols, it is planned to develop eco-friendly, efficient catalytic methods for organic synthesis [34, 35].

Silicate-1 zeolite is crystalline with a micro-porous MFI framework, and weak Lewis acidity. The acidity of silicate-1 can be enhanced by doping with transition metal cations, such as Ti (IV), which generates strong Lewis acidic sites in the framework, including Bronsted acidic sites. It has been reported that the acidity of TS-1 increases with increasing titanium content in a zeolite framework [36,37,38,39].

Considering this view and in continuation of our research work on the development of heterogeneous solid acid catalysts, we report here the synthesis of a TS-1 zeolite catalyst by incorporating Ti (IV) cations in the framework of silicate-1 [40, 41].

Experimental

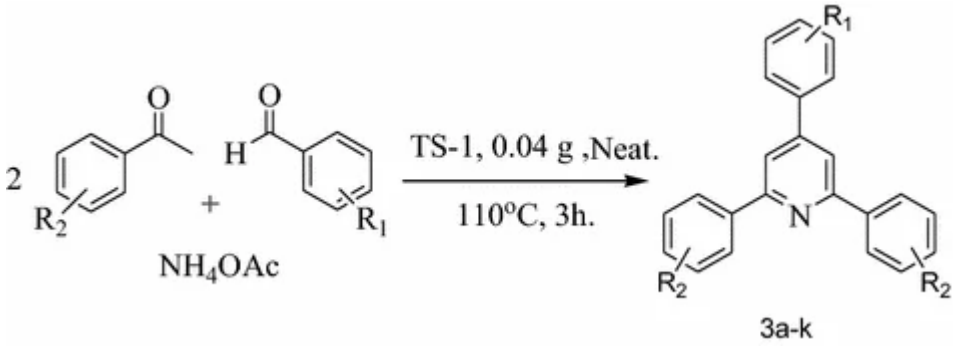
General Procedure for the Synthesis of the Catalyst

Amounts of 24.37 mL of tetraethyl orthosilicate and 25 mL, 20% tetrapropyl ammonium hydroxide were added to a flask with vigorous stirring. The resulting reaction mixture was stirred for 10 min at room temperature to form a silica sol. Next, 1.16 mL of Titanium tetrabutoxide was mixed in 30 mL of dry isopropyl alcohol, stirred vigorously for 10 min, and the resulting mixture was added dropwise into the silica sol with constant stirring at 70–75 °C with the pH maintained above 12. The resulting transparent viscous gel was transferred to a Teflon-lined stainless steel autoclave, and treated hydrothermally under static condition and autogenous pressure at 175 °C for 24 h. The solid product was filtered, dried in an oven at 100 °C for 1 h, and finally calcined at 500 °C for 4 h in a muffle furnace, under air atmosphere. The resulting material was cooled naturally, characterized and used as the catalyst.

General reaction procedure synthesis of modal reaction 3a

A mixture of acetophenone (240.30 mg, 2 mmol), 4-chloro-benzaldehyde (140.56 mg, 1 mmol), NH_4OAc (100.20 mg, 1.3 mmol), and 40.00 mg of the TS-1 catalyst was added and refluxed for 3–4 h, as shown in Scheme 1. The progress of the reaction was monitored by thin-layer chromatography using petroleum ether:ethyl acetate (8:2) as a solvent system. After completion of the reaction, the catalyst and product were separated. Then, the catalyst was activated and used for the next batch of reactions. The filtered organic layer containing the product was dried by using sodium sulfate, and then the filtrate was concentrated to obtain the crude product which was recrystallized from the ethanol solvent to afford the pure products (**3a–l**).

Scheme 1



Synthesis of 2,4,6-Triaryl-pyridine derivatives model reaction, **3a**

Spectral data

2,4,6-Triarylpyridine (3a)

Yiled = 93%, M. P. 132–134 °C, ^{1}H NMR ($CDCl_3$): δ 7.25–8.2 (m, 17H, Ar–H); **^{13}C NMR ($CDCl_3$)**: δ 157.37, 150.07, 139.45, 138.93, 128.99, 128.92, 128.85, 128.58, 128.33, 127.06, 127.00, and 117.01; **FT- IR (KBr, cm^{-1}):** 3058, 1576, 1549, 1449, 1399, 1074, 870, 735, and 692; **LC–MS:** M^{+1}, 308.14 found 308.13.

4-(4-Methylphenyl)-2,6-(biphenyl)pyridine (3b)

Yiled = 89%, M. P. 124–125 °C, **^{1}H NMR ($CDCl_3$):** δ 8.20 (dd, 4H, Ar–H), 7.22–7.65 (m, 10H, Ar–H), 7.86 (s, 2H, Pyridyl-H), and 2.42 (s, 3H, CH_3); **^{13}C NMR ($CDCl_3$)**: δ 157.49, 150.07, 139.71, 139.12, 136.11, 129.87, 129.03, 128.73, 127.17, 127.03, 116.94, and 21.31; **FT- IR (KBr):** 3031, 2919, 1595, 1545, 1449, 1418, 1391, 1252, 1209, 1178, 1032, 874, 769, and 619; **LC–MS:** M^{+1}, 322.15 found 322.13.

4-(2,6-Diphenylpyridin-4-yl)phenol (3c)

Yiled = 90%; M. P. 197–199 °C; **^{1}H NMR ($CDCl_3$):** δ 8.21 (dd, 4H, Ar–H), 7.75 (s, 2H, Pyridyl-H), 7.69 (dd, 2H, Ar H), 7.4–7.5(m, 6H, Ar H), 6.99 (d, 2H, Ar H), and 5.14 (s, 1H, OH); **^{13}C NMR ($CDCl_3$):** δ 157.58, 156.61, 149.67, 139.75, 131.61, 129.08, 128.78,128.66, 127.22, 116.73, and 116.10; **FT-IR (KBr, cm^{-1}):** 3035, 2954,2850, 1603, 1545, 1518, 1399, 1348, 1206, 733, and 688; **LC–MS:** M^{+1}, 324.13 found 324.12.

4-chlorophenyl (3d): ^{1} HNMR ($CDCl_3$)

Yiled = 93%; M. P. 126–127 °C; **^{1}HNMR ($CDCl_3$):** δ 7.26–8.21 (m, 17 H, Ar–H); **FT-IR (KBr, cm^{-1}):** 3031, 1696, 1649, 1348, and 1233; LC–**MS**: M^{+1}, 342.10 found 342.09.

5-methoxy-2-(2,6-diphenylpyridin-4-yl)phenol (3k))

Yiled = 88%; M. P. 167–168 °C; **^{1}HNMR ($CDCl_3$):** δ 3.05–3.11 (dd, 1H, CH_2), 3.78 (dd, 1H CH_2), 3.81 (s, 3H OCH_3), 3.80–3.81 (s, 3H OCH_3), 5.15–5.20 (dd, 1H, CH), and 6.77–7.29 (m, 13 Ar–H); **^{13}C NMR ($CDCl_3$)**: δ 139.88, 129.45, 129.26, 129.12, 128.81, 128.62, 128.54, 127.29, 127.17, 127.02, 119.26, 116.07, 56.20, and 56.07; **FT-IR (KBr, cm^{-1}):** 3495, 2924, 1666, 1476, 1256, 1064, and 694; **LC–MS**: M^{+1}, 354.14 found 354.107.

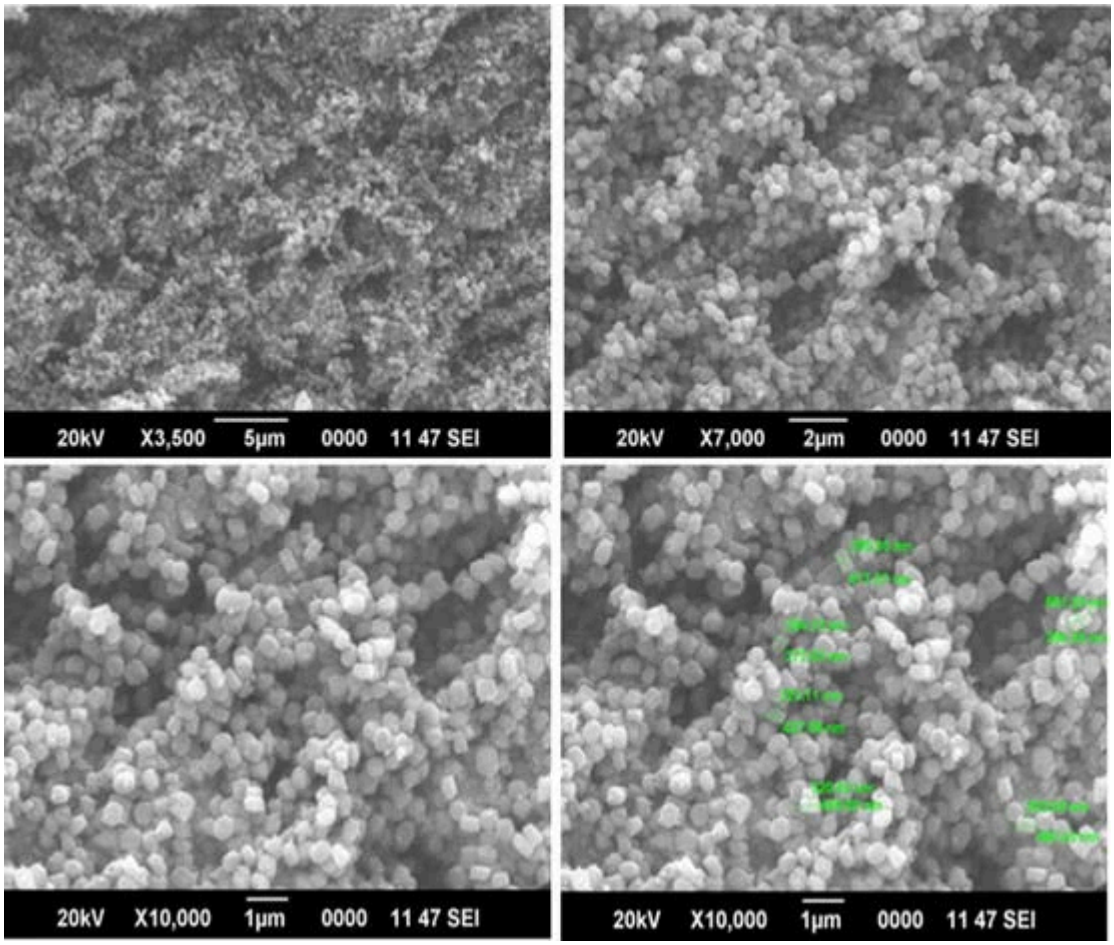
Results and discussion

The TS-1 catalyst was synthesized and characterized by X-ray diffraction, Fourrier-transform infrared spectroscopy, scanning electron microscopy and BET surface area analysis. The XRD of TS-1, depicted in Fig. S1 (Supplementary information), shows intense peaks at $2\theta°$ = 14.63, 21.81, 22.76, 22.59, 24.04, and 26.62, with the corresponding planes (112), (023), (150), (511), (250), and (004) respectively; the planes (150) and (511) indicate the presence of an ordered orthorhombic TS-1 framework [42, 43].

The FT-IR spectrum of TS-1 is shown in Fig. S2 (Supplementary information). The absorption band at 530−551 cm^{-1} corresponds to the presence of S5R, which confirms the MFI topology of TS-1 [44, 45]. The band appearing between 550 and 623 cm^{-1} is due to Ti−O bond vibration [46]. The sharp absorption band appearing at 800 cm^{-1} is ascribed to Si−O−Si symmetric stretching vibration [47]. The absorption band observed in the range 952−967 cm^{-1} is due to Si−O−Ti bond vibration, which confirms the presence of Ti (IV) content in the TS-1 framework. The characteristic band observed in the range 1228−1265 cm^{-1} is for the MFI topology of the TS-1 zeolite [43]. However, the broad band in the range 3404−3799 cm^{-1} is due to Si−OH−Si or Si−OH−Ti vibrations [48].

The surface morphology and textural properties of the TS-1 catalyst have been studied by scanning electron microscopy, which shows uniform sphere-shaped crystals, which is a characteristic morphology of TS-1-type zeolites. The SEM image suggests that the synthesized catalyst has a uniform particle size and ordered morphology, as shown in Fig. 1. The BET surface area, average pore diameter and pore volume of calcined TS-1 were found to be 408.3 m^2/g, 20.87 Å and 0.2130 cm^2/gm respectively calculated by the N_2 adsorption−desorption BJH isotherm.

Fig. 1



SEM images of TS-1 zeolite calcined at 500 °C

The synthesized catalyst was used for the one-pot solvent-free synthesis of poly-substituted 2,4,6-triaryl-pyridines via cyclocondensation of acetophenone, aryl aldehyde and NH_4OAc (Scheme 1). The structure of representative derivatives have been confirmed by ^1H and ^{13}C NMR, Mass, and FTIR techniques.

In order to explore the scope and importance of the investigated method, we have synthesized several pyridine derivatives using electronically diverse aldehydes (R_1) and acetophenones (R_2), and the results are summarized in Table 1. It was found that acetophenones (R_2) and aldehydes (R_1) with electron-donating groups like H, CH_3, 4-OH, 4-OCH_3 gave maximum yields as compared to electron-withdrawing groups like 4-Cl and 4-NO_2. However, 4-Cl gave more yield as compared to NO_2 because of a mesomeric effect.

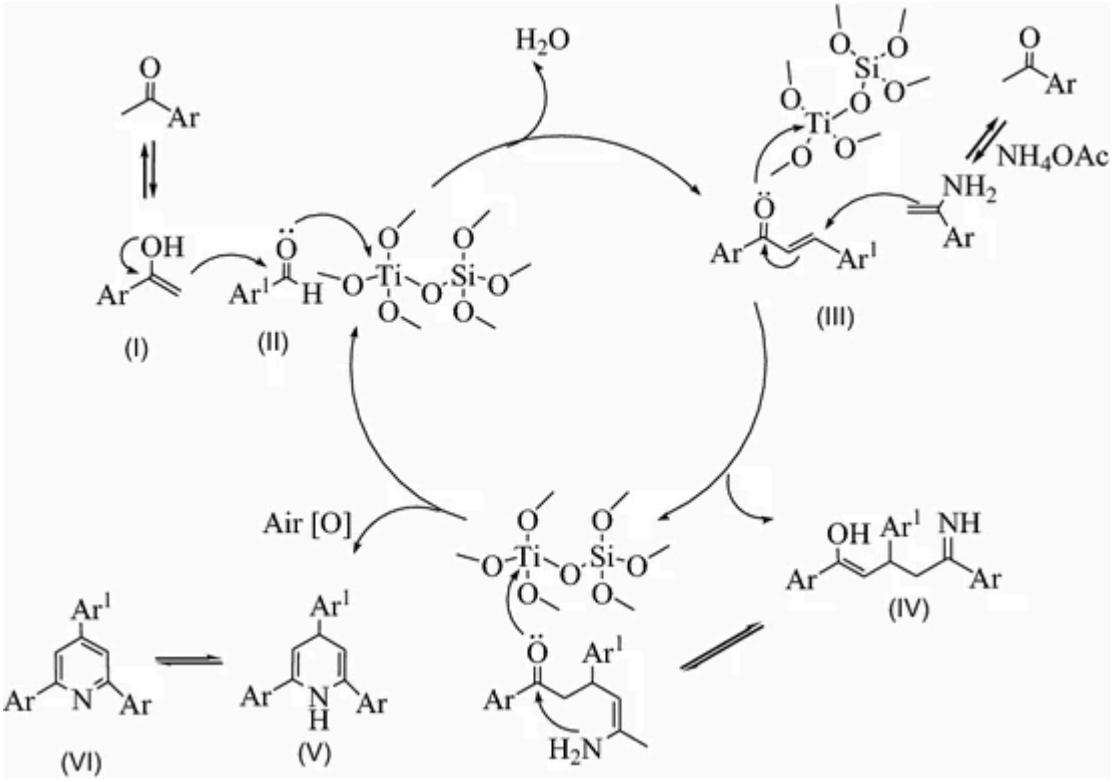
Table 1 TS-1 catalyzed synthesis of 2,4,6-triaryl-pyridine derivatives

Proposed mechanism for the formation pyridine derivatives

The aldehyde and acetophenone initially formed a chalcone intermediate in the presence of the TS-1 catalyst. It has been assumed that the reaction was initiated by the polarization of the > C=O group of the aldehyde, which perhaps simultaneously interacts with the > C=O group of the acetophenone to enhance the electrophilic interactions. In second step, a Michael addition reaction was facilitated by the two components, chalcone and enamine, which are in close proximity to each other. Subsequently, ring cyclization–dehydration–air oxidation–aromatization gave rise to the desired 2,4,6-triaryl-pyridine derivatives.

The posssible reaction mechanism for the formation of triaryl pyridines is depicted in Scheme 2. The enol form of acetophenone (I) gives a nucleophilic addition on the aldehyde (II) in the presence of TS-1 to afford the condensation product of chalcone intermediate (III). Then, the second molecule of acetophenone reacts with ammonia to form an amine, which undergoes a Michal addition reaction with (III) to form the (IV) intermediate. Simultaneously, cyclization and dehydration result in the formation of compound (V) in the presence of TS-1. Finally, the air oxidation process gave the target molecule, triaryl pyridines (**3a–l**).

Scheme 2



Proposed reaction mechanism for the synthesis of 2,4,6-triaryl-pyridine derivatives

To specify the advantages of the proposed method, the results of different reported methods are compared with our results and are summarized in Table 2, which confirms that the TS-1 catalyst promotes the reaction more efficiently than the other reported methods.

Table 2 Catalytic performance comparison of TS-1 and other reported catalysts

Optimization of reaction conditions

In order to find the optimum loading of the catalyst, acetophenone (2 mmol), 4-chloro-benzaldehyde (1 mmol), and ammonium acetate (1.3 mmol) were refluxed with the TS-1 catalyst. Ammonium acetate was used as a source of ammonia which gave better results [13]. Initially, several protic and aprotic solvents with appropriate times and temperatures were screened and the obtained results for model reaction **3a**, are summarized in Table 3. Then, the effect of varying the amount of the catalyst was tested, as summarized in Table 4. It has been found that, in the absence of the catalyst, the reaction did not give satisfactory results of the desired product in the stipulated period; however, the reaction yield was found to be a maximum in the solvent-free condition, and therefore further optimization of the catalyst amount was performed in a solvent-free reaction condition. It was found that 0.04 g of the TS-1 catalyst is sufficient for the model reaction (Table 4). Finally, the effect of varying the temperature and time was optimized and the results are presented in Table 5.

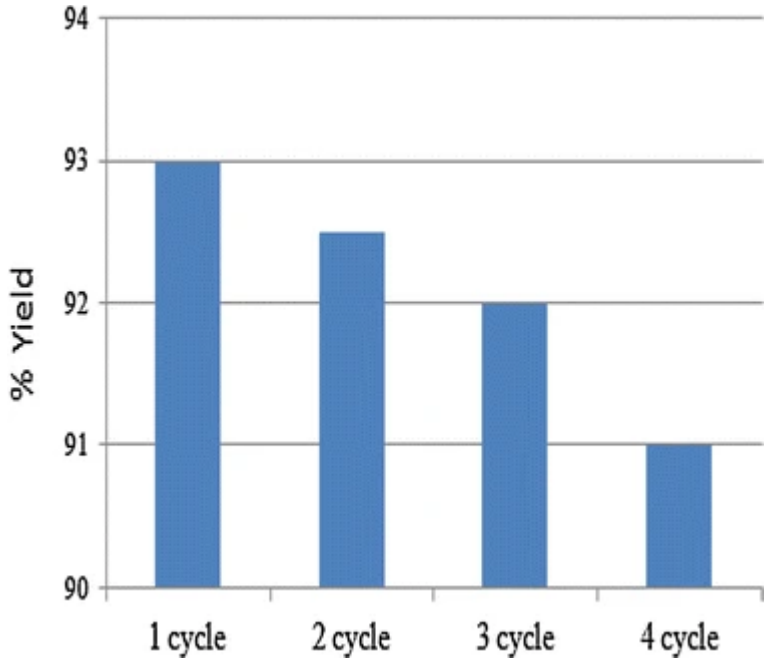
Table 3 Effect of various solvents on the model reaction 3a in the presence of 0.04 g of TS-1 catalyst

Table 4 Effect of the amount of TS-1 on the model reaction 3a

Table 5 Effect of temperature on the yield under solvent-free condition, 3a

After optimizing the reaction conditions, efforts were made towards the recovery and reusability of the catalyst. After completion of the reaction, the TS-1 catalyst was recovered from the reaction mixture by filtration and washed with acetone or another polar solvent and activated at 105 °C under vacuum for 10 min. The reusability of the catalyst was investigated for four runs on themodel reaction and it was found that the catalyst retained almost consistent catalytic activity (Fig. 2).

Fig. 2



Reusability test of TS-1 for the synthesis of **3a**

Conclusions

In summary, we have developed a conventional approach for the synthesis of 2,4,6-triaryl pyridines, assisted by the solid acid catalyst, TS-1 zeolite, from acetophenone, aryl aldehyde and ammonium acetate. It shows excellent catalytic potency for the synthesis of poly-substituted pyridines via a cyclic condensation reaction under solvent-free conditions. This method offers remarkable advantages over reported methods because of the non-toxic, non-corrosive and reusable nature of the TS-1 catalyst with excellent yields.

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Electronic supplementary material

Below is the link to the electronic supplementary material.

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Full experimental details and ^{1}H, ^{13}C NMR, HRMS spectra, XRD. BET, SEM FTIR, of other products in the "supplementary content" of this article web page are available. (DOCX 1110 kb)

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