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(Carboxy-3-oxopropylamino)-3-propylsilylcellulose as a novel organocatalyst for the synthesis of substituted imidazoles under solvent-free conditions

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(Carboxy-3-oxopropylamino)-3-propylsilylcellulose (COPAPSC), a novel organocatalyst, has been prepared by a synthesis grafting of –COOH functionalized organosilanes on a cellulose using surface hydroxyl groups as anchor points. The $-CO_2H$ group-functionalized cellulose was synthesized *via* the consecutive surface functionalization with 3-aminopropyltriethoxysilane (3-APTES) followed by the condensation of the surface $-NH_2$ groups with succinic anhydride. COPAPSC is used as a catalyst for the synthesis of tri- and tetra-substituted imidazoles from the reaction of benzil, aromatic aldehydes, ammonium acetate, and amines under solvent-free conditions. The key advantages of this process are high yields, easy work-up, purification of products by non-chromatographic methods and the reusability of the catalyst.

Introduction

Nowadays, owing to the increasing concern about environmental consciousness, more attention has been focused on the development of new processes that minimize pollution and maximize sustainable development in chemical synthesis.[1]

In this respect, the immobilization of catalysts on solid supports has several important potential advantages such as removal, recovery, and reutilization of catalysts; reduced environmental contamination; good thermal and chemical stabilities; and good dispersion of the active catalytic sites.[2] For this purpose, exploring heterogeneous catalysis is obviously on the rise, especially in industries.[3] Many solid materials as supports, such as silica,[4,42] zeolites,[5] metal oxides,[6] graphene,[7] magnetic-materials,[8] and polymers,[9] have broadly been investigated for catalytic applications. A considerable stimulation of scientific and technological research has been triggered since the past 10 years in response to the growing global importance of renewable resources and environmentally compatible materials.[10] In the field of natural biopolymers, cellulose could especially be utilized as a support for catalytic applications.

Cellulose is the most widely spread organic polymer that is found in nature because it constitutes the main component of the plants' cell wall. It is well-known that cellulose is a fascinating, inexhaustible biopolymer and a renewable raw material. It was widely applied as an efficient support and/or template for the synthesis of inorganic and organic materials. For example, sulfuric acid has been supported on cellulose and the acid modified cellulose was used as an activated catalyst for the synthesis of α-amino amide derivatives through Ugi reactions, synthesis of 1,4-dihydropyridines through Hantzsch reactions, and synthesis of 3,4-dihydropyrimidin-2(1*H*)-ones/-thiones through Biginelli reactions.[11–13] In addition, carboxylate-functionalized cellulose has been prepared by the reaction of succinic anhydride and cellulose, and used as an effective biosorbent for heavy metals remediation.[14] Recently, cellulose has gained renewed interest as a raw material and still possesses a high potential for future applications. The imidazole nucleus is a fertile source of biologically important molecules[15] and is the core structural skeleton in many important biological molecules, such as histidine, histamine, and biotin, as well as several drug moieties[16,17] such as trifenagrel, eprosartan, and losartan. In addition, they are used in photography as photosensitive compounds. Some substituted triarylimidazoles are the selective antagonists of the glucagon receptors and inhibitors of IL-1 biosynthesis.[18]

The recent development of green chemistry and organometallic chemistry expands the utility of imidazoles as ionic liquids[19–21] and N-heterocyclic carbenes.[19,22,23] Radziszewski and Japp[24,25] proposed the first synthesis of the imidazole core in 1882, starting from 1,2-dicarbonyl compounds, aldehydes, and ammonia to obtain 2,4,5-triphenylimidazole.

From this discussion and our interest and experience in the area of heterogeneous catalysis in organic synthesis,[26–28] we report a new surface $-CO_2H$ group functionalized cellulose *via* the consecutive surface functionalization with 3-aminopropyltriethoxysilane (3-APTES) followed by the condensation of the

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surface $-NH_2$ groups with succinic anhydride (Scheme 1). Previously, $-CO_2H$ and $-SO_3H$ functionalized celluloses have been reported but the incorporation of the $-CO_2H$ group on a cellulose by condensation between an anhydride and a primary amine has not been studied so far. On the contrary, the increasing importance of metal-free catalysis motivated us to use this organically modified acid functionalized cellulose (COPAPSC) as a catalyst for the synthesis of imidazole from a mixture of an aromatic aldehyde, an amine and a α-diketone. To the best of our knowledge, organocatalysis promoted by a $-CO_2H$ functionalized cellulose for the synthesis of imidazole has not been explored so far.

Experimental

General

Chemicals were purchased from Merck and Fluka chemical companies. IR spectra were obtained in KBr pellets in the reflection mode on an Avata Therma Nicolet FTIR instrument. ^1H NMR spectra were obtained on a Bruker Avance DPX-250 MHz spectrometer using TMS as an internal standard and $CDCl_3$ or DMSO as the solvent. ^{13}C NMR spectra were obtained on a Bruker Avance DPX-62.5 MHz spectrometer ($CDCl_3$ or DMSO solution). Mass spectra were obtained on a GC 17A, MS QP 5050 Shimadzu instrument. Elemental analysis for C, H, and N were obtained using an Elementar, Vario EL III. All the reactions were monitored by thin layer chromatography (TLC) on pre-coated sheets of silica gel G/UV-254 using UV light for visualization. Melting points were determined with an Electrothermal 9100 melting point apparatus.

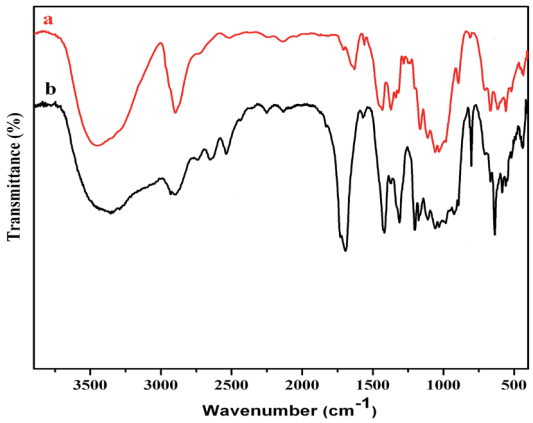
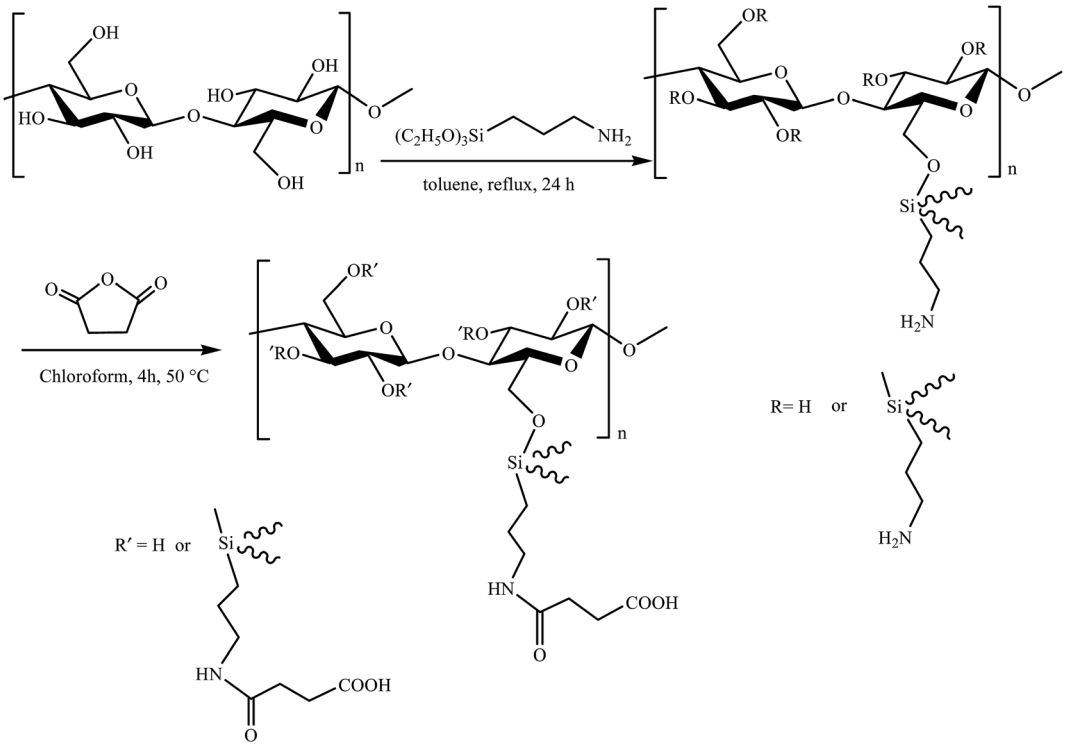


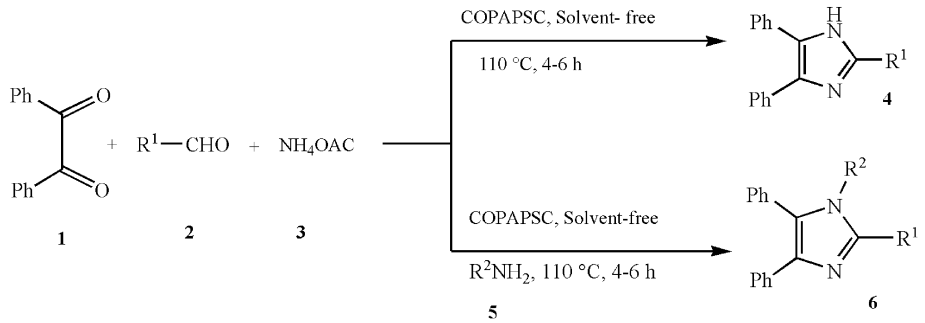
Fig. 1 FT-IR spectra of: (a) cellulose and (b) COPAPSC.

Preparation of COPAPSC

A magnetically stirred cellulose (5.0 g) was suspended in dry toluene (50 ml) and then an excess of 3-APTES (5.0 ml) was added. The suspension was mechanically stirred as it was heated under reflux for 24 h under an Ar atmosphere. The resulting white solid was filtered, washed repeatedly with toluene, ethanol–water, deionized water and methanol and finally dried under vacuum at 60 °C for 4 h to give 3-aminopropylsilylcellulose (3-APSC) (5.2 g). This 3-APSC functionalized cellulose was stirred with 0.15 g (15 mmol) of succinic anhydride dissolved in dry chloroform (20 ml) for 4 h at 50 °C, and the final product was collected by filtration, washed sequentially with chloroform, ethanol and diethyl ether. The product was



Scheme 1 Preparation of (carboxy-3-oxopropylamino)-3-propylsilylcellulose.



Scheme 2 COPAPSC catalyzed synthesis of trisubstituted and tetrasubstituted imidazoles.

dried in a vacuum desiccator to give COPAPSC as a white powder (5.39 g).

General procedure for the preparation of imidazole derivatives

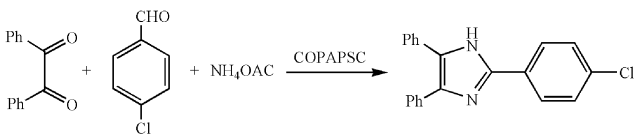
A mixture of 1,2-diketone (1 mmol), aromatic aldehyde (1 mmol), amine (1 mmol), ammonium acetate (1.5 mmol), and COPAPSC (0.2 g equal to 0.05 mmol of H^+) was stirred at 110 °C under solvent-free conditions for the given time (Tables 2 and 3). After completion of the reaction, appropriate amounts of hot EtOH (96%) was added and the mixture was stirred for 10 min and then the catalyst was separated by filtration. The catalyst was washed with warm ethanol (3 ml × 3) to clean effectively. The filtrate was concentrated in vacuum to remove ethanol. The residue was washed with cold water and crystallized from hot ethanol to afford the pure products. The recovered catalyst was dried and reused in the subsequent runs.

Results and discussion

Catalyst characterization

Fig. 1 shows the FT-IR spectra of cellulose and COPAPSC. The absorbance at 3200–3500 cm^{-1} range can be attributed the O–H and N–H amid stretching vibrations of COPAPSC. The two bands at 2933 and 2898 cm^{-1} are attributed to the $-CH_2-$ asymmetric and symmetric stretching vibrations, respectively. The absorbance at 1728 and 1693 cm^{-1} are due to the carbonyl groups of acid and amide, respectively. Another band at 1419 cm^{-1} is attributed to the C–N stretching vibration, which overlapped with the –C–C–H vibration of cellulose. The absorbance at 1058 cm^{-1} is assigned to the –C–O– stretching

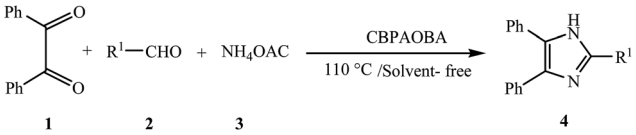
Table 1 Effect of solvent, temperature and amount of catalyst on the synthesis of 4d[a]



Entry	Catalyst (g)	Solvent	Temperature (°C)	Time (h)	Yield[b] (%)
1	Cellulose (0.2)	Solvent free	110	6	15
2	COPAPSC (0.08)	Solvent free	80	7	40
3	COPAPSC (0.10)	Solvent free	80	7	52
4	COPAPSC (0.20)	Solvent free	80	5	63
5	COPAPSC (0.08)	Solvent free	100	5	55
6	COPAPSC (0.10)	Solvent free	100	5	73
7	COPAPSC (0.20)	Solvent free	100	5	80
8	COPAPSC (0.08)	Solvent free	110	5	60
9	COPAPSC (0.10)	Solvent free	110	4	72
10	COPAPSC (0.2)	Solvent free	110	4	84
11	COPAPSC (0.3)	Solvent free	110	4	84
12	COPAPSC (0.20)	Water	Reflux	7	—
13	COPAPSC (0.20)	Methanol	Reflux	7	72
14	COPAPSC (0.20)	Ethanol	Reflux	5.5	70
15	COPAPSC (0.20)	Ethylene glycol	140	9	70
16	COPAPSC (0.20)	Glycerol	140	8	75

[a] Reaction conditions: 4-chlorobenzaldehyde (1 mmol), ammonium acetate (1.5 mmol), and benzil (1 mmol) in the presence of cellulose and COPAPSC. [b] Isolated yields.

Table 2 Synthesis of 2,4,5-trisubstituted imidazoles[a]



1 2 3 4

Entry	R^1	Product	Time (h)	MP °C (lit.) [ref.]	Yield[b] (%)
1	C_6H_5	4a	4	270–273 (274–276) 30	77
2	4-$OMeC_6H_4$	4b	4	226–228 (228–230) 30	85
3	4-MeC_6H_4	4c	4	227–229 (232–234) 30	82
4	4-ClC_6H_4	4d	4	260–261 (260–262) 30	84
5	4-$NO_2C_6H_4$	4e	4	235–238 (234–236) 30	82
6	3-$NO_2C_6H_4$	4f	4	263–265 (>300) 31	78
7	4-OHC_6H_4	4g	5	230–233 (234–236) 30	77
8	2-OHC_6H_4	4h	4	207–210 (202–205) 31	79
9	4-BrC_6H_4	4j	4	244–246 (254–256) 30	88
10	2-$NO_2C_6H_4$	4k	5.5	228–230 (230–231) 31	80
11	4-$N(Me)_2C_6H_4$	4l	6	256–259 (256–259) 31	75

[a] Reactions were performed with benzil (1 mmol), aromatic aldehyde (1 mmol), ammonium acetate (1.5 mmol) and COPAPSC (0.2 g equal to 0.05 mmol H^+) at 110 °C under solvent-free conditions. [b] Isolated yields of pure products.

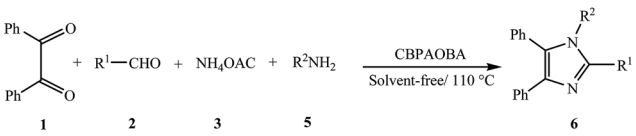
vibration.[29] The OH vibration of the carboxylic groups overlapped with the OH vibration of cellulose.

The nitrogen content in COPAPSC was determined to be 0.32% (0/23 mmol g^{-1}) by elemental analysis. The number of H^+ sites of COPAPSC was determined to be 0.25 mmol g^{-1} by an acid–base titration, which was very close to the nitrogen content. These results indicated that most of the nitrogen species on the sample were in the form of amide groups.

In continuation of our work on the development of useful synthetic methodologies toward the synthesis of heterocyclic compounds, the catalytic activity of the Bronsted acid COPAPSC was tested for the synthesis of 2,4,5-trisubstituted and 1,2,4,5-tetrasubstituted imidazoles under solvent-free conditions (Scheme 2).

We attempted to find technically simple, high yielding, and solvent-free conditions for the synthesis of these compounds. Therefore, we studied COPAPSC as a catalyst in the cyclocondensation of benzil, aldehyde, ammonium acetate, and amine. Firstly, the reaction of 4-chlorobenzaldehyde (1 mmol), ammonium acetate (1.5 mmol) and benzil (1 mmol) catalyzed

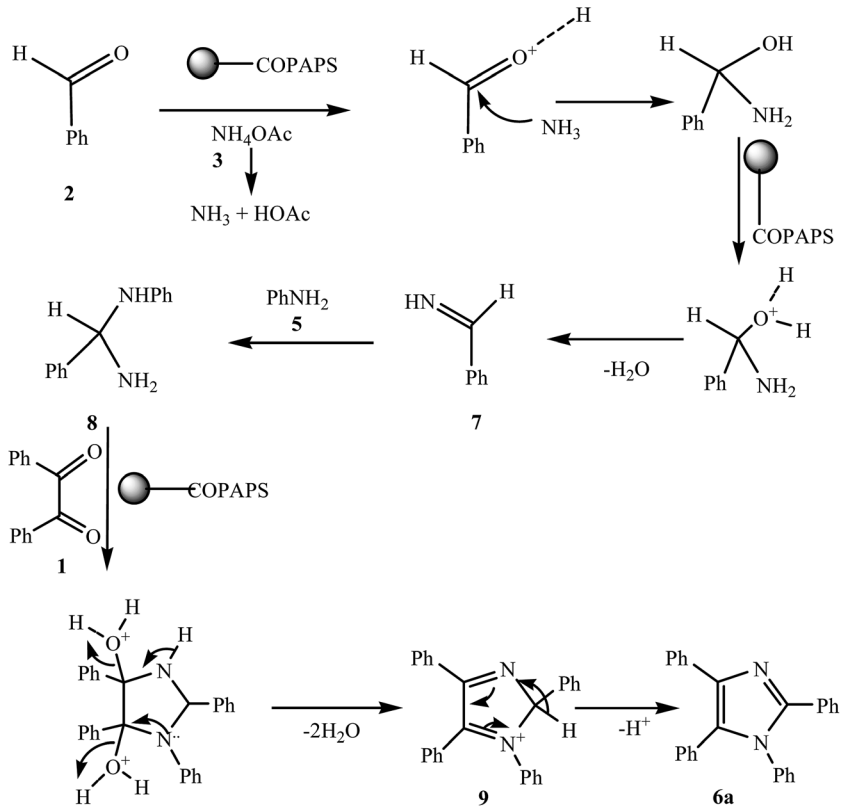
Table 3 Synthesis of 1,2,4,5-tetrasubstituted imidazoles[a]



1 2 3 5 6

Entry	R^1	R^2	Product	Time (h)	MP °C (lit.) [ref.]	Yield[b] (%)
1	C_6H_5	C_6H_5	6a	5.5	210–211 (214–216) 15	83
2	3-ClC_6H_4	Benzyl	6b	5	148–150 (144–146) 32	85
3	4-ClC_6H_4	Benzyl	6c	5	156–158 (160–162) 15	82
4	4-$OMeC_6H_4$	4-MeC_6H_4	6d	3.5	180–181 (176–178) 33	85
5	4-$OMeC_6H_4$	Benzyl	6e	4	160–163 (162–164) 15	87
6	2-ClC_6H_4	Benzyl	6f	5	137–139 (140–141) 34	75
7	4-OHC_6H_4	Benzyl	6g	4	137–138 (134–135) 34	84
8	4-BrC_6H_4	Benzyl	6h	5	175–178 (170–172) 34	75
9	4-$NO_2C_6H_4$	4-MeC_6H_4	6i	4	223–226 (219–220) 32	75
10	4-OHC_6H_4	Benzyl	6j	4.5	253–254 (257–259) 15	78
11	3-$NO_2C_6H_4$	4-MeC_6H_4	6k	4	150–152 (149–151) 32	87
12	4-MeC_6H_4	4-MeC_6H_4	6l	4	192–194 (188–191) 32	80

[a] Reactions were performed with benzil (1.0 mmol), aromatic aldehyde (1.0 mmol), ammonium acetate (1.0 mmol), amine (1.0 mmol), and COPAPSC (0.2 g equal to 0.05 mmol H^+) at 110 °C under solvent-free conditions. [b] Isolated yields of pure products.



Scheme 3 Probable mechanism for the formation of 6a using COPAPSC as catalyst.

by COPAPSC was investigated in different solvents as well as under solvent-free conditions, and the results are shown in Table 1.

Initial screening studies confirmed that the solvent-free technique is the optimal condition for this reaction. Another important point which could be elicited evidently from these results is that raising the reaction temperature from 80 °C to 110 °C and the amount of catalyst from 0.08 g to 0.2 g increased the yield and also improved the reaction rates (Table 1, entries 2–10). A further increase in the catalyst loading does not affect the yield (Table 1, entry 11). Moreover, it is worth mentioning that application of solvents, such as H_2O, EtOH, CH_3OH, ethylene glycol, and glycerol, did not lead to better results. Under these conditions, longer reaction times and lower yields can be observed clearly (Table 1, entries 12–16). On the contrary, due to the growing concern for the influence of the organic solvents on the environment as well as on the human body, organic reactions without the use of conventional organic solvents have attracted the attention of synthetic organic chemists. It is observed that the solvent-free conditions gave an excellent yield of product and shorter reaction times than that of in the presence of solvents. The development of solvent-free organic reactions is thus gaining prominence.

As a control experiment, the cyclocondensation of benzil, aldehyde, and ammonium acetate was also carried out at 110 °C in the presence cellulose for 6 hour under solvent-free conditions, which resulted in a yield of 15% (Table 1, entry 1).

After determining the optimum reaction conditions, we turned our attention to study the scope of this method. We applied this catalyst for the synthesis of trisubstituted and tetrasubstituted imidazoles using different aromatic aldehydes with a wide range of substitutions under solvent-free heating conditions to establish the catalytic importance of COPAPSC for this reaction. The corresponding results are given in Tables 2 and 3.

The proposed mechanism for the formation of the products can be explained by the pathway presented in Scheme 2. The reaction commenced through the acid catalyzed formation of imine (7), which underwent nucleophilic attack by aniline to

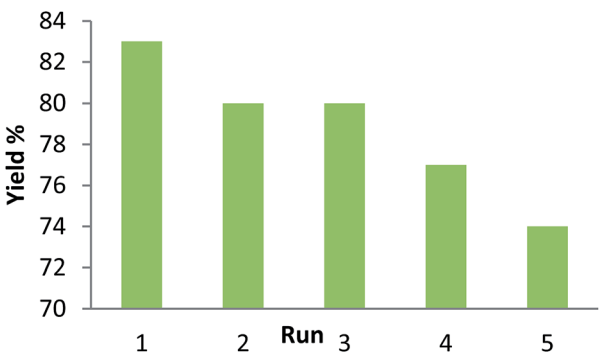


Fig. 2 Reusability of COPAPSC in condensation of benzaldehyde, ammonium acetate, aniline and benzyl at 110 °C under solvent-free conditions in 5 h.

Table 4 Comparison of results using COPAPSC with results obtained by other works for the synthesis of 4d

Entry	Catalyst	Condition	Yield (%)	Ref.
1	COPAPSC	Solvent free, 110 °C, 4 h	84	—
2	$Yb(OPf)_3$	HOAc, $C_{10}F_{18}$, 80 °C, 6 h	83	35
3	Acetic acid	Chloroform, 160, MWI, 15 min	90	36
4	$InCl_3 \cdot 3H_2O$	MeOH, r.t., 9.4 h	71	37
5	Diethyl bromophosphate	CH_3CN, ultrason., 30 min	95	38
6	DABCO	*t*-BuOH, 65 °C, 12 h	92	39
7	CSC-Star-SO_3AlCl_2	EtOH, 80 °C, 10 h	94	40
8	L-Proline	MeOH, 60 °C, 9 h	88	31
9	MCM-41	Reflux in AcOH, 32 min	82	41

give the *in situ* intermediate (**8**). An acid catalyzed condensation between this intermediate (**8**) and diketone (**1**) produced another *in situ* intermediate (**9**), which on subsequent aromatization produces the tetrasubstituted imidazole (**6a**) and releases the catalyst for the next catalytic cycle (Scheme 3).[15]

The recyclability of the catalyst was also examined. To this end, the catalyst that was recovered from the reaction between benzil, aniline, benzaldehyde, and ammonium acetate by filtration was washed three times with warm ethanol and dried at 80 °C for a period of 5 h in a vacuum oven. The recovered catalyst can be reused four times in subsequent reactions without any significant loss in its activity. The results with the recyclable COPAPSC are summarized in Fig. 2.

In Table 4, the efficiency of our method for the synthesis of imidazoles is compared with some other published works in literature. The reaction of 4-chloro benzaldehyde, benzil, and ammonium acetate was used as a model reaction. Each of these methods have their own advantages, but they often suffer from some troubles, including the use of organic solvent, (entries 2–5) and long reaction time (entries 6–9).

In summary, COPAPSC, an efficient, reusable, green and solidly supportive biodegradable acid catalyst, has been prepared and utilized for the synthesis of imidazole derivatives by a one-pot coupling reaction of benzil, benzaldehyde compounds, ammonium acetate, and primary amine. Moreover, the broad scope, operational simplicity, and practicability render it as an attractive approach for the generation of different compounds with potential properties for use in medicinal chemistry programs.

Selected experimental data

2-(4-Methoxyphenyl)-4,5-diphenyl-1*H*-imidazole (4b). White solid, (85%, 0.277 g) mp: 231–233 °C (Lit.[30] mp 228–230 °C). IR (KBr, *ν*: cm^{-1}): 3425, 3029, 2956, 1610, 1495, 1249; ^1H NMR (250 MHz, DMSO-d_6) (*δ*, ppm): 3.79 (s, 3H), 7.05 (d, $J = 8.4$ Hz, 2H), 7.50–7.28 (m, 10H), 8.03 (d, $J = 8.1$ Hz, 2H), 12.50 (s, 1H); ^{13}C NMR (62.9 MHz, DMSO-d_6) (*δ*, ppm): 55.2, 114.1, 123.5, 126.8, 127.7, 128.4, 129, 131.6, 135.7, 137.2, 146.1, 159.8; anal. calcd for $C_{22}H_{18}N_2O$: C, 80.96; H, 5.56; N, 8.58. Found: C, 80.90; H, 5.51; N, 8.63.

1-(4-Methylphenyl)-2-(3-nitrophenyl)-4,5-diphenyl-1*H*-imidazole (6k). Yellow solid, (87%, 0.375 g) mp: 150–152 °C (Lit.[33] 149–151 °C). IR (KBr, *ν*: cm^{-1}): 3052, 1596, 1525, 1350; ^1H NMR (250 MHz, DMSO-d_6) (*δ*, ppm): 2.34 (s, 3H), 7.45–6.94 (m, 14H), 7.50 (t, 1H), 8.11 (d, $J = 7.8$ Hz, 1H), 8.24 (d, $J = 7.5$ Hz, 1H), 8.52 (s, 1H); ^{13}C NMR (62.9 MHz, DMSO-d_6) (*δ*, ppm): 21.2, 122.7, 123.4, 126.9, 127.3, 127.9, 128.2, 128.8, 129.1, 131.0, 132.0, 132.2, 133.7, 134.3, 138.7, 139.1, 144.3, 148.0; anal. calcd for $C_{28}H_{21}N_3O_2$: C, 77.94; H, 4.91; N, 9.74. Found: C, 77.89; H, 4.97; N, 9.78.

Acknowledgements

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