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[Bmim]HSO_4-Catalyzed Synthesis of Tetrasubstituted Imidazoles as Potential Mutant Isocitrate Dehydrogenase 1 Inhibitors

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Abstract—An ecofriendly, simple, and efficient one-pot protocol has been developed for the synthesis of 1,2,4,5-tetrasubstituted imidazoles by the reaction of benzil, aryl aldehydes, aliphatic or aromatic amines, and ammonium acetate, catalyzed by the acidic halogen-free ionic liquid [bmim]HSO_4 under solvent-free conditions. The advantages of the proposed protocol include high yields, short reaction time, operational simplicity, and recyclability of the catalyst. Eight previously unknown tetrasubstituted imidazoles were synthesized, among which those bearing a cyclopropyl substituent on N^1 can be considered as potential mutant isocitrate dehydrogenase 1 (IDH1) inhibitors.

Keywords: 1,2,4,5-tetrasubstituted imidazoles, [bmim]HSO_4, ionic liquid, synthesis

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INTRODUCTION

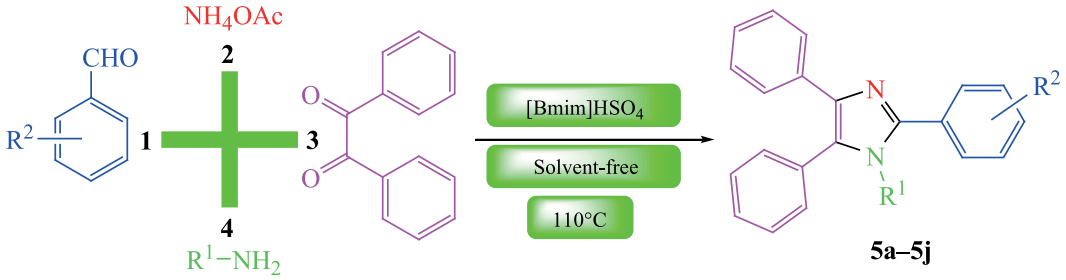
Imidazoles is an important class of heterocycles that exhibit diverse biological activities, including herbicidal [1], antiallergic [2], analgesic [3], antidepressant [4], antitubercular [5], anticancer [6], anti-inflammatory [7], antifungal [8], and antiviral [9]. Imidazoles are present in histidine [10] and histamine [11], and some of imidazole structures are used in electronic and optoelectronic devices [12].

Tetrasubstituted imidazoles have been synthesized by the multi-component cyclocondensation of 1,2-diketone, aldehydes, a primary amine, and ammonium acetate, using various Lewis or protic acidic reagents, including BF_3–SiO_2 [13], silica gel/$NaHSO_4$ [14], HPA–EtOH [15], L-proline [16], K_5CoW_{12}–$O_{40}\cdot 3H_2O$ [17], heteropolyacids [18], zeolite-HY–$Cu(NO_3)_2$ [19], under microwave irradiation [20], and the reaction of 1,3-oxazolium-5-olates with *N*-(arylmethylene)benzenesulfonamides [21]. Some imidazole cyclopropylamine analogs were found to exhibit improved activity as isocitrate dehydrogenase 1 (IDH1) inhibitors [22].

On the other hand, ionic liquids (ILs) have been widely employed as an environmentally friendly alternative to organic solvents in various fields, such as separation of azeotropic or close-boiling mixtures [23], separation/purification of bioactive compounds [24–26], desulfurization [27], Diels-Alder reaction [28] and biodiesel production [29]. "Brønsted acidic ILs combining the advantages of extractant and acid catalyst [30, 31] were designed to replace traditional mineral liquid acids in current esterification protocols" [32–34]. For example, [bmim][HSO_4] was used as a dual catalyst–solvent for the esterification of hexanoic acid with *n*-butanol [35]. Also, [bmim][HSO_4] was utilized for the synthesis of heterocyclic compounds, such 3,4,5-substituted furan-2(5*H*)-ones [36] and 1,8-dioxo-octahydroxanthenes [37].

Proceeding with our research on the preparation of heterocyclic compounds, especially imidazole derivatives [38–42], in the present work we developed a mild and an effective one-pot four-component synthesis of 1,2,4,5-tetrasubstituted imidazoles from benzil, aryl aldehydes, aliphatic or aromatic amines, and ammonium acetate, using the acidic halogen-free ionic liquid [bmim]HSO_4 as a highly active and recyclable catalyst, under solvent-free conditions (Scheme 1).

Scheme 1.



5, R^1 = cyclopropyl, R^2 = 4-OMe (**a**); R^1 = cyclopropyl, R^2 = 4-OH (**b**); R^1 = cyclopropyl, R^2 = H (**c**); R^1 = cyclopropyl, R^2 = 2,6-Cl_2 (**d**); R^1 = C_6H_5, R^2 = 2,6-Cl_2 (**e**); R^1 = cyclopropyl, R^2 = 2-OH, 5-Br (**f**); R^1 = cyclopropyl, R^2 = 3,4-OMe_2 (**g**); R^1 = cyclopropyl, R^2 = 4-Br (**h**); R^1 = 4-Br-C_6H_4, R^2 = 4-NO_2-C_6H_4 (**i**); R^1 = C_6H_5, R^2 = 4-OMe (**j**).

RESULTS AND DISCUSSION

To study generality of this method, a variety of aryl aldehydes as well as aromatic and aliphatic amines, benzil, and ammonium acetate were employed for preparing tetrasubstituted imidazoles, using [bmim]HSO_4 as an acidic and a halogen-free ionic liquid. The reactions were performed with 1 mmol of benzil, 1 mmol of aldehyde, 1 mmol of ammonium acetate, 1 mmol of amine (1 mmol), and 20 mol % of [bmim]HSO_4 (20 mol %) under solvent-free conditions at 110°C. As seen from Scheme 2, 1,2,4,5-trisubstituted imidazoles **5a–5j** were obtained in good-to-excellent isolated yields over short reaction times. To study the substituent effect, a wide variety of aromatic aldehydes containing electron-acceptor (Cl, Br, and NO_2) or electron-donor substituents [OMe, $(MeO)_2$, and OH] were reacted to obtain equally good results with both groups of aldehydes (Scheme 1). Noteworthy is the fact that, along with the high yields of the target products, no by-products, like 2,4,5-triarylated imidazoles, oxidized anilines, or aldehydes, which are normally formed under strongly acidic conditions, were detected. Other advantages of the new method include simple isolation and purification of the products and reusability of the catalyst. Using the developed protocol, we synthesized eight previously unknown tetrasubstituted imidazoles **5a–5h**, of which cyclopropyamine derivatives deserve special mentioning (Schemes 1 and 2).

The suggested mechanism of the formation of tetrasubstituted imidazoles from aldehydes, benzil, amines, and ammonium acetate is shown in Scheme 3. The first step involves the reaction between a [bmim]HSO_4-activated aryl aldehyde, ammonium acetate, and an amine to give a diamine intermediate **6**. The latter then reacts with a [bmim]HSO_4-activated diketone **7** to form intermediate **8**, which undergoes dehydration to produce imidazole **5**.

To compare the merits of the proposed catalytic protocol with previously reported ones, we compared our results for the synthesis of 2-(4-methoxyphenyl)-1,4,5-triphenyl-1*H*-imidazole (**5j**) with respective results obtained with other catalysts (Table 1). As seen from the table 1, the present method is not worse or compares in efficiency with the others.

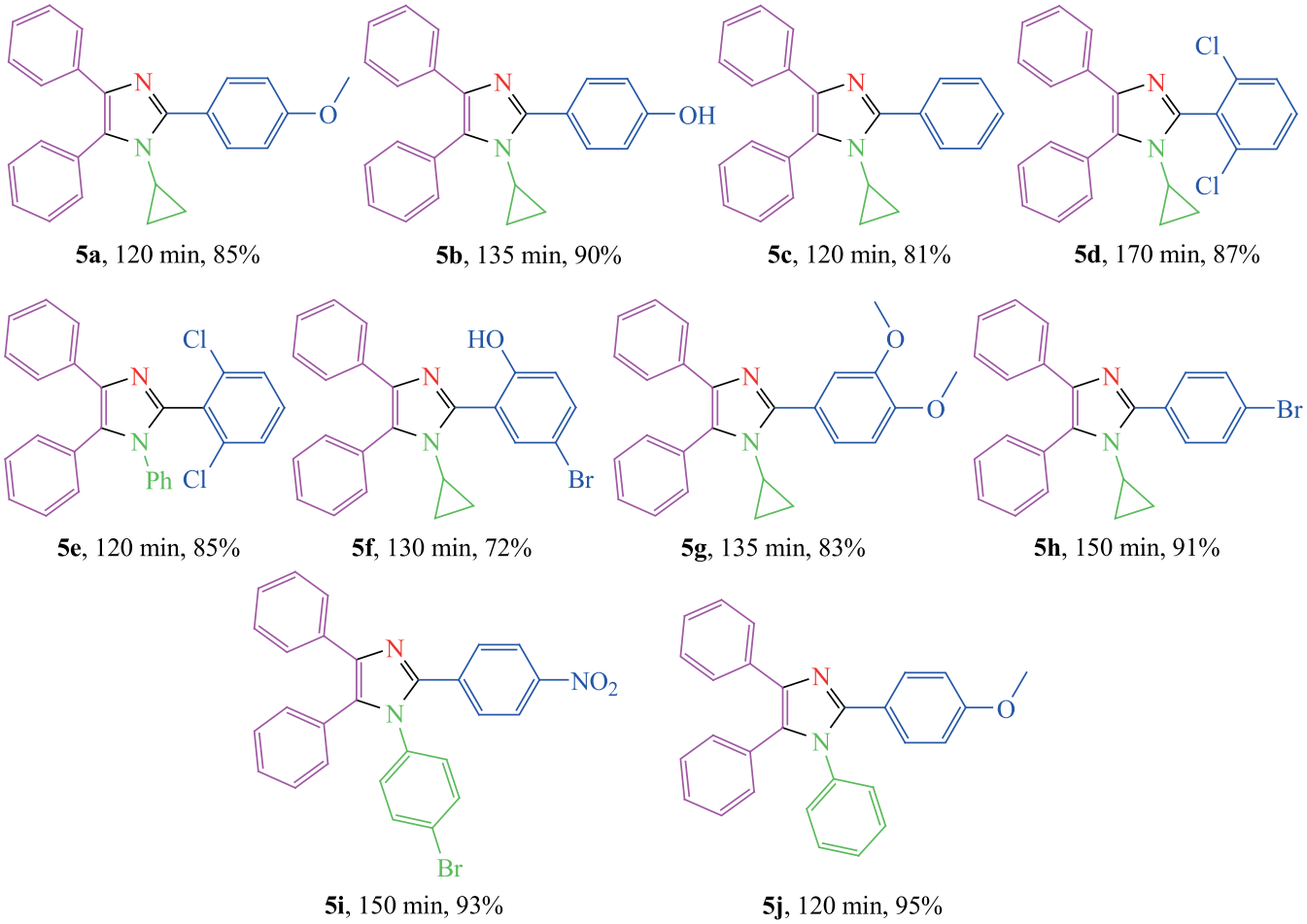
To our knowledge, [bmim]HSO_4 has never been used for the synthesis of tetrasubstituted imidazoles, especially novel cyclopropylamine imidazole derivatives which hold promise as potential IDH1 inhibitors. Therefore, the developed procedure can be considered a useful contribution to the methods of synthesis of tetrasubstituted imidazoles.

The synthesis of compounds **5j** was also used to assess the reusability of the [bmim]HSO_4 catalyst. After completion of the reaction, the catalyst was removed by filtration, dried under vacuum, and reused. In was found that the catalyst could be reused at least for times without appreciable loss of efficiency, yield of compound **5j** (run no.): 85 (1), 83 (2), 80 (3), 78 (4).

EXPERIMENTAL

The melting points were measured by on an Electrothermal IA9200 apparatus. The IR spectra were recorded on a Perkin Elmer FTIR spectrometer in the range 4000–400 cm^{-1} for KBr disks. The ^1H and ^{13}C NMR spectra were obtained on a Bruker DRX 300 MHz NMR instrument for $CDCl_3$ solutions. The mass spectra were taken on an Agilent 5973 Network Mass Selective Detector instrument.

Scheme 2.



5a, 120 min, 85% **5b**, 135 min, 90% **5c**, 120 min, 81% **5d**, 170 min, 87%

5e, 120 min, 85% **5f**, 130 min, 72% **5g**, 135 min, 83% **5h**, 150 min, 91%

5i, 150 min, 93% **5j**, 120 min, 95%

Tetrasubstituted imidazoles 5a–5j (*general procedure*). A mixture of benzil (1 mmol), aryl aldehyde (1 mmol), ammonium acetate (1 mmol), amine (1 mmol), and $[Bmim]HSO_4$ (20 mol %) was stirred at 110°C for 2–2.5 h. After completion of the reaction, the reaction mixture was cooled to room temperature and then diluted with water. The precipitate was filtered off and recrystallized from ethanol to obtain the desired product (Schemes 1 and 2).

1-Cyclopropyl-2-(4-methoxyphenyl)-4,5-diphenyl-1*H*-imidazole (5a). Cream solid, mp 141–143°C. IR spectrum (KBr), ν, cm^{-1}: 3065 (CH_{arom}), 1612 ($C{=}C_{arom}$), 1587 (C=C imidazole), 1446, 1309 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.82–0.90 m (2H, CH_2), 0.95–1.05 m (2H, CH_2), 3.29–3.37 m (1H, CH), 3.81 s (3H, OCH_3), 6.89 d (2H, *J* 8.8), 7.14–7.21 m ($2H_{arom}$), 7.32–7.55 m ($10H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 367 (19) $[M]^+$. Found, %: C 81.94; H 6.05; N 7.64. $C_{25}H_{22}N_2O$. Calculated, %: C 81.91; H 6.02; N 7.60.

4-(1-Cyclopropyl)-4,5-diphenyl-1*H*-imidazole-2-yl)phenol (5b). Yellow solid, mp 248–250°C. IR spectrum (KBr), ν, cm^{-1}: 3338 (OH), 3068 (CH_{arom}), 1600 ($C{=}C_{arom}$), 1584 (C=C imidazole), 1493, 1318 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.84–0.89 m (2H, CH_2), 1.01–1.06 m (2H, CH_2), 3.67–3.74 m (1H, CH), 4.78 s (1H, OH), 7.00 d.d (*J* 8.2, 2.5), 7.17–7.34 m ($10H_{arom}$), 7.48–7.53 m ($2H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 352 (15) $[M]^+$. Found, %: C 81.75; H 5.70; N 7.93. $C_{24}H_{20}N_2O$. Calculated, %: C 81.79; H 5.72; N 7.95.

1-Cyclopropyl-2,4,5-triphenyl-1*H*-imidazole (5c). Cream solid, mp 187–189°C. IR spectrum (KBr), ν, cm^{-1}: 3067 (CH_{arom}), 1621 ($C{=}C_{arom}$), 1584 (C=C imidazole), 1448, 1317 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.83–0.89 m (2H, CH_2), 1.01–1.04 m

Scheme 3.

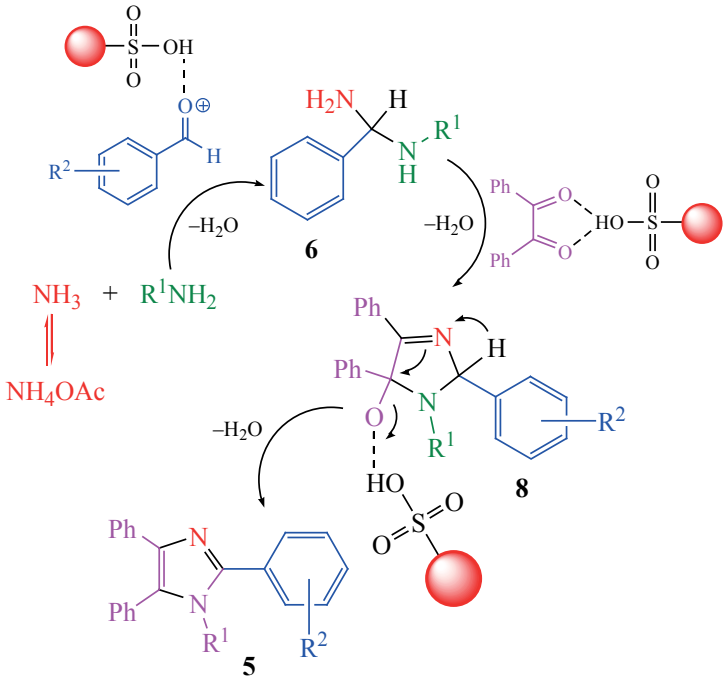


Table 1. Comparison of the efficacy of [bmim]HSO_4 and other catalysts in the synthesis of 2-(4-methoxyphenyl)-1,4,5-triphenyl-1*H*-imidazole (**5j**)

Entry	Catalyst	Time, h	*T*, °C	Solvent	Yield of **5j**, %	References
1	PEG-400 (5 mL)	1.5	reflux	PEG-400	96	[43]
2	Iodine (10 mol %)	0.2	75	EtOH–CH_2Cl_2	94	[44]
3	$Cu(NO_3)_2$–Zeolite (0.03 g)	0.5	80	None	95	[45]
4	MCS-GT@Co(II) (5 mg)	3	reflux	EtOH	99	[46]
5	MCM-41-SO_3H (0.04 g)	0.25	100	None	98	[47]
6	[DABCO-DOL][OAc] (5 mol %)	5	60	H_2O	96	[48]
7	CSC-Star-SO_3-$AlCl_2$ (9 mol %)	10	80	EtOH	94	[49]
8	*n*-CTW-SA (10 mol %)	1.35	120	None	93	[50]
9	nano-Fe_3O_4 (15 mol %)	60	140	None	92	[51]
10	Fe_3O_4–PEG–Cu (10 mol %)	55	110	None	92	[52]
11	[Bmim]HSO_4 (20 mol %)	2	110	None	95	This work

(2H, CH_2), 3.68–3.75 m (1H, CH), 7.19–7.50 m ($15H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 336 (20) $[M]^+$. Found, %: C 81.68; H 5.97; N 8.30. $C_{24}H_{20}N_2$. Calculated, %: C 85.68; H 5.99; N 8.33.

1-Cyclopropyl-2-(2,6-dichlorophenyl)-4,5-diphenyl-1*H*-imidazole (5d). Yellow solid, mp 182°C. IR spectrum (KBr), ν, cm^{-1}: 3065 (CH_{arom}), 1606 ($C{=}C_{arom}$), 1582 (C=C imidazole), 1445 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.95–0.99 m (2H, CH_2), 1.03–1.06 m (2H, CH_2), 3.62–3.72 m (1H, CH), 7.21–7.24 m ($2H_{arom}$), 7.38–7.52 m ($11H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 405 (22) $[M]^+$. Found, %: C 71.10; H 4.45; Cl 17.46; N 6.89. $C_{24}H_{18}Cl_2N_2$. Calculated, %: C 71.12; H 4.48; Cl 17.49; N 6.91.

2-(2,6-Dichlorophenyl)-1,4,5-triphenyl-1*H*-imidazole (5e). Brown solid, mp 205°C. IR spectrum (KBr), ν, cm^{-1}: 3067 (CH_{arom}), 1609 ($C{=}C_{arom}$), 1585 (C=C imidazole), 1447 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 7.23–7.67 m ($18H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 440 (19) $[M]^+$. Found, %: C 73.45; H 4.08; Cl 16.05; N 6.31. $C_{27}H_{18}Cl_2N_2$. Calculated, %: C 73.48; H 4.11; Cl 16.07; N 6.35.

4-Bromo-2-(1-cyclopropyl-4,5-diphenyl-1*H*-imidazole-2-yl)phenol (5f). White solid, mp 217°C. IR spectrum (KBr), ν, cm^{-1}: 3348 (OH), 3061 (CH_{arom}), 1600 ($C{=}C_{arom}$), 1532 (C=C imidazole), 1487, 1419, 1343 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 1.01–1.07 m (2H, CH_2), 1.13–1.18 m (2H, CH_2), 3.98–4.05 m (1H, CH), 5.82 s (1H, OH), 6.72 d (1H, *J* 8.6), 7.16 d.d (1H, *J* 8.6, 2.4), 7.39–7.49 m ($11H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 431 (19) $[M]^+$. Found, %: C 66.80; H 4.41; Br 18.50; N 6.47. $C_{24}H_{19}BrN_2O$. Calculated, %: C 66.83; H 4.44; Br 18.53; N 6.49.

1-Cyclopropyl-2-(3,4-dimethoxyphenyl)-4,5-diphenyl-1*H*-imidazole (5g). White solid, mp 205–207°C. IR spectrum (KBr), ν, cm^{-1}: 3065 (CH_{arom}), 1618 ($C{=}C_{arom}$), 1576 (C=C imidazole), 1448, 1320 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.87–0.95 m (2H, CH_2), 1.19–1.26 m (2H, CH_2), 3.66–3.73 m (1H, CH), 3.88 s (3H, OCH_3), 3.95 s (3H, OCH_3), 6.82 d (2H, *J* 8.3), 7.42–7.53 m ($11H_{arom}$). Mass spectrum, *m/z* (I_{rel}, %): 396 (28) $[M]^+$. Found, %: C 78.73; H 6.06; N 7.04. $C_{26}H_{24}N_2O_2$. Calculated, %: C 78.76; H 6.10; N 7.07.

2-(4-Bromophenyl)-1-cyclopropyl-4,5-diphenyl-1*H*-imidazole (5h). Cream solid, mp 174–176°C. IR spectrum (KBr), ν, cm^{-1}: 3066 (CH_{arom}), 1599 ($C{=}C_{arom}$), 1586 (C=C imidazole), 1486, 1449, 1322 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.86–0.96 m (2H, CH_2), 1.18–1.26 m (2H, CH_2), 3.64–3.71 m (1H, CH), 7.18–7.50 m ($11H_{arom}$) 7.94 d (3H, *J* 7.4). Mass spectrum, *m/z* (I_{rel}, %): 414 (21) $[M]^+$. Found, %: C 69.39; H 4.58; Br 19.22; N 6.71. $C_{24}H_{19}BrN_2$. Calculated, %: C 69.41; H 4.61; Br 19.24; N 6.74.

1-(4-Bromophenyl)-2-(4-nitrophenyl)-4,5-diphenyl-1*H*-imidazole (5i). Yellow solid, mp 257–259°C [53]. IR spectrum (KBr), ν, cm^{-1}: 3093 (CH_{arom}), 1595 ($C{=}C_{arom}$), 1578 (C=C imidazole), 1479, 1449, 1353 (C–N). ^1H NMR spectrum, δ, ppm (*J*, Hz): 7.14 d (2H, *J* 8.4), 7.49–7.56 m ($7H_{arom}$), 7.67 t (3H, *J* 8.0), 7.98 d (2H, *J* 7.5), 8.24 d (2H, *J* 7.6), 8.34 d (2H, *J* 7.6).

2-(4-Methoxyphenyl)-1,4,5-triphenyl-1*H*-imidazole (5j). Cream solid, mp 178–180°C [54]. IR spectrum (KBr), ν, cm^{-1}: 3063, 1593, 1578, 1492, 1417, 1324. ^1H NMR spectrum, δ, ppm (*J*, Hz): 3.75 s (3H, OCH_3), 6.96–7.04 m ($2H_{arom}$), 7.16–7.68 m ($11H_{arom}$), 7.77–7.99 m ($6H_{arom}$).

CONCLUSIONS

In summary, in the present work we developed a simple, an efficient, and an environmentally benign one-pot multicomponent method for the synthesis of 1,2,4,5-tetrasubstituted imidazoles in solvent-free conditions, catalyzed by the ionic liquid $[bmim]HSO_4$. This method was used to success to synthesize a variety of imidazole derivatives from aromatic aldehydes and aliphatic and aromatic amines in good-to-excellent yields. The synthesized cyclopropylamine imidazole derivatives hold promise as potential mutant IDH1 inhibitors. Further advantages of the developed protocol consist in easy work-up of the reaction mixture and lack of the need of column chromatography for purification of the products.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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