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Improving methodology for the preparation of highly substituted imidazoles using nano-MgAl$_2$O$_4$ as catalyst under microwave irradiation

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ABSTRACT

An efficient synthesis of 2,4,5-trisubstituted and 1,2,4,5-tetrasubstituted imidazoles by one-step condensation of an aldehyde, benzil, ammonium acetate and primary amine in the presence of nanocrystalline magnesium aluminate under microwave irradiation is described. The advantages of this catalyst are including simple work-up, low cost and reusability. Compared with conventional methods, the main advantages of the present procedure are its being a green method, its milder conditions, necessary shorter reaction time, and its higher yields and its selectivity. The structures of products were characterized by ^{1}H NMR, ^{13}C NMR, IR, elemental analyses, MS and UV spectral data. Their melting points were compared with literature report.

Keywords: Nanocrystalline magnesium aluminate; Imidazoles; Solvent free; MW irradiation; Multi-component reaction.

1. Introduction

In the recent years, being focused on green chemistry using environmentally benign reagents is one of the most fascinating developments in synthesis of widely used organic compounds. Combination of the supported reagents and microwave irradiations were used under solvent less conditions [1]. The application of microwave energy as a fast, simple, uniform and energy efficient heating method to accelerate organic reactions is of increasing interest. Microwave assisted reactions are extremely attractive to synthetic organic chemists due to their ability to shorten reaction times, enhances product purity, and in some cases improve regio- and/or chemoselectivity [2]. Imidazole and their derivatives, play important role as versatile building blocks for the synthesis of natural products and as therapeutic agents [3,4]. Compounds with imidazole ring system, also display various pharmaceutical activities such as anti-inflammatory, [5] analgesic, [6] antitumor, [7] and anti-allergic [8]. Additionally, many of the substituted imidazoles are known as anion sensors, [9] ionic liquids, [10] catalyst, [11–17] electrical and optical materials, [18–20] biological imaging, [21] fluorescence labeling agents, [22–24] and chromophores for non-linear optic systems [25] Due to their wide range of biological, industrial
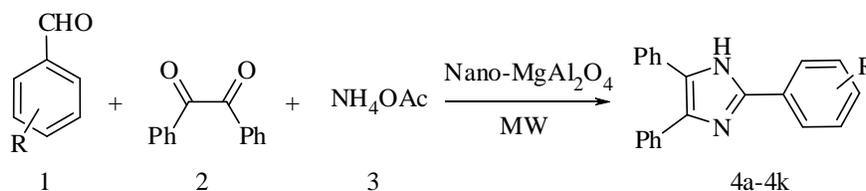
Scheme 1. Synthesis of 2,4,5-trisubstituted-1*H*-imidazole derivatives.

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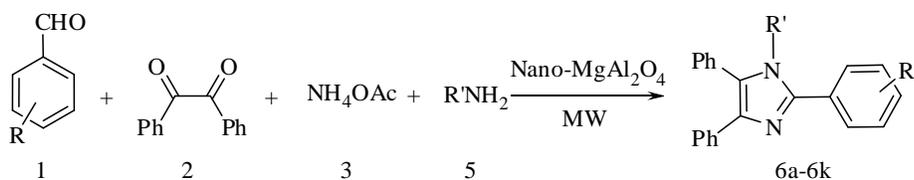
Scheme 2. Synthesis of 1,2,4,5- tetrasubstituted -1*H*-imidazole derivatives.

and synthetic applications, these compounds have recently received an excessive attention. There are numerous methods in the literature for the synthesis of multisubstituted imidazoles such as *N*-alkylation of trisubstituted imidazoles, [26] cyclization of sulfonamides with mesoionic 1,3-oxazolium-5-olates, [27] conversion of *N*-(2-oxo)amides with ammonium trifluoroacetate under neutral conditions, [28] four-component condensation of 1,2-diones, aldehydes, primary amines and ammonium acetate in nano-crystalline sulfated zirconia (SZ), [29] and nano-TiCl$_4$.SiO$_2$, [30] and also condensation of β-carbonyl-*N*-acyl-*N*-alkylamines with ammonium acetate in refluxing acetic acid. [31] However, some of these synthetic protocols require hazardous and expensive reagents, harsh reaction conditions, prolonged time period, and sometimes tedious work-up. Therefore, the development of a novel catalytic system and an environmentally protocol to overcome these limitations for the synthesis of highly substituted imidazoles is an important task for organic chemists. Herein, we report a green and simple approach for the synthesis of 2,4,5-trisubstituted-1*H*-imidazole and 1,2,4,5-tetrasubstituted -1*H*-imidazole derivatives using nano-MgAl$_2$O$_4$ under microwave irradiation (Schemes 1 and 2). This catalyst is mild, recoverable, non-explosive, easy to handle, and reusable. We wish to explore the use of nano-MgAl$_2$O$_4$ under microwave irradiation in the synthesis of highly substituted imidazoles. Nanocrystalline MgAl$_2$O$_4$ is an efficient catalyst with high specific surface area, high activity and superior controlled selectivity. The crystallite sizes of the magnesium aluminate determined by TEM analysis was at 700 ºC were between 3–10 nm (Fig. 1). The pore volume and pore size were also calculated from the N$_2$ adsorption/desorption isotherm result, the approximately 1.10 cm^{3}g^{-1}. In addition the surface area is approximately 201 m^{2}g^{-1} [32].

2. Experimental

A domestic microwave oven (Bajaj, ET–B at 2450 MHz, 100% power, 1300 W) was used in all experiments. All melting points are uncorrected and were measured by open capillary method. IR spectra were run on Jasco–IR spectrometer. ^{1}H NMR and ^{13}C NMR spectra were recorded in DMSO-d_6 with TMS as internal reference on Brucker AC–300NMR spectrometer. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyzer carried out on Perkin–Elmer 240c analyzer.

2.1. General procedure for synthesis of nano crystalline MgAl$_2$O$_4$

This procedure was used according to the literature method [47]. In a typical preparation, magnesium nitrate Mg(NO$_3$)$_2$.6H$_2$O and aluminum nitrate Al(NO$_3$)$_3$.9H$_2$O were used as starting materials. The stoichiometric amounts of magnesium and aluminum nitrates and a desired amount of CTAB were added to deionized water. Then, ammonia solution was drop wised to the well stirring slurry (pH around 9). After precipitation, the slurry was stirred for another 30 min and then refluxed at 80 °C for 24 h. The final product was filtered and dried at 100 °C for 24 h.

2.2. General procedure for synthesis of 2, 4, 5-trisubstituted imidazoles

In a 50 mL round bottom flask mixture of benzil (1 mmol), benaldehyde (1 mmol), ammonium acetate (2 mmol), and and nano-MgAl$_2$O$_4$ (0.035 mol%) was stirred under microwave irradiation. The progress of the reaction was monitored by TLC (petroleum ether–ethyl acetate 9:1). After completion of the reaction, the reaction was allowed to cool and nano-MgAl$_2$O$_4$ filtered and dried at 80 ^{o}C and used for its reusability.

Fig. 1. TEM image of nanocrystalline MgAl$_2$O$_4$.

The organic layer was extracted with H_2O and was removed under reduced pressure. Finally, pure powder was recrystallized from ethanol, and dried to give products.

2.3. General procedure for synthesis of 1,2,4,5-tetrasubstituted imidazoles

A mixture of benzil (1 mmol), benzaldehyde (1 mmol), NH_4OAc (4 mmol), aromatic amine (4 mmol), and nano-$MgAl_2O_4$ (0.035 mol%) was added into 50 mL borosil beaker and was irradiated with microwaves. The progress of reaction was monitored by TLC using petroleum ether and ethyl acetate (9:1). After completion of the reaction, the reaction was allowed to cool and nano-$MgAl_2O_4$ filtered and dried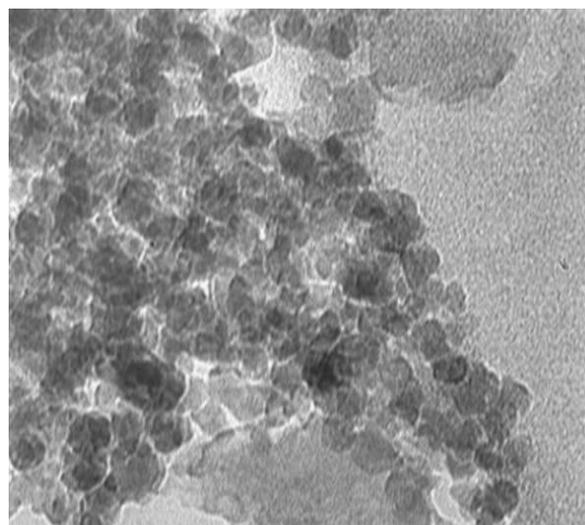 at 80 °C and used for its reusability. The organic layer was extracted with H_2O and was removed under reduced pressure. Finally, pure powder was recrystallized from ethanol, and dried to give products.

The selected spectral data

2-(2-Fluorophenyl)-1,4,5-triphenyl-1H-imidazole (6f).

White needles; Yeild: 93%; m.p. 165−168 °C; IR (KBr ν_{max}, cm^{-1}): 3050 (C-H aromatic), 1509 (C=C aromatic), 1095 (C=N), 1095 (C-F); ^{1}H NMR (400 MHz, DMSO-d_6, ppm;) δ: 7.11−7.30 (m, 15H, H-Ar), 7.41 (d, J = 8.0 Hz, 1H, H-Ar), 7.46 (d, J = 8.0 Hz, 2H, H-Ar), 7.53 (t, J = 8.0 Hz, 1H, H-Ar), ^{13}C NMR (100 MHz, DMSO- d_6, ppm): δ 129.82, 129.94, 130.51, 130.55, 130.64, 131.60, 132.75, 132.81, 133.65, 136.61, 136.82, 138.50, 140.50, 143.13, 144.90, 148.02; MS (70eV) m/z (%): 390 (M$^+$, 55), 371 (50), 295 (48); Anal. Calcd for $C_{27}H_{19}FN_2$: C, 83.06; H, 4.9; N, 7.17. Found: C, 83.5; H, 4.93; N, 7018%.

2,4,5-Triphenyl-1-propyl-1H-imidazole (6h).

Yellow needles; Yeild: 93%; m.p. 87−89 °C; IR (KBr ν_{max}, cm^{-1}): 3025 (C-H aromatic), 1597 (C=C aromatic), 1479 (C=N); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 0.51 (t, J = 6.8 Hz, 3H, CH$_3$), 1.32 (m, J = 6.8 Hz, 2H, CH$_2$), 3.81 (t, J = 7.2 Hz, 2H, CH$_2$), 7.10−7.55 (m, 13H, H-Ar), 7.7 (d, J = 6.8 Hz, 2H, H-Ar); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ: 11.07, 23.56, 46.32, 126.55, 128.50, 129.12, 129.17, 129.33, 129.62, 130.27, 131.30, 131.56, 131.78, 135.17, 137.0, 147.20; MS (70eV) m/z (%): 338 (M$^+$, 68), 323 (65), 309 (57), 295 (43), 15 (40); Anal. Calcd for $C_{24}H_{22}N_2$: C 85.17, H 6.55, N 8.28. Found: C 85.14, H 6.56, N 8.30%.

2-(4-Chlorophenyl)-4,5-diphenyl-1-propyl-1H-imidazole (6i).

Yellow crystals; Yeild: 96%; m.p. 85−87 °C; IR (KBr ν_{max}, cm^{-1}): 3025 (C-H aromatic), 1645 (C=C aromatic), 1489 (C=N), 1011 (C-Cl); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 0.51 (t, J = 6.8 Hz, 3H, CH$_3$), 1.34 (m, J = 6.8 Hz, 2H, CH$_2$), 3.81 (t, J = 7.2 Hz, 2H, CH$_2$), 7.15−7.30 (m, 12H, H-Ar), 7.34 d, J =7.2 Hz, 2H, H-Ar); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ: 11.08, 23.54, 46.39, 126.55, 126.66, 128.53, 129.24, 129.43, 129.65, 130.52, 130.65, 130.89, 131.29, 133.30, 134.96, 136.15, 146.98; MS (70eV) m/z (%): 435 (M$^+$, 55), 432 (53), 417 (50), 402 (44), 77 (32); Anal. Calcd for $C_{24}H_{22}ClN_2$: C 77.30, H 5.68, N 7.51. Found: C 77.32, H 5.69, N 7.48%.

2-(4-Methylphenyl)-4,5-diphenyl-1-propyl-1H-imidazole (6j).

Milky needles; Yeild: 94%; m.p. 78−83 °C. IR (KBr ν_{max}, cm^{-1}): 3028 (C-H aromatic), 1620 (C=C aromatic), 1497 (C=N) ; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 0.52 (t, J = 6.8 Hz, 3H, CH$_3$), 1.30 (m, J = 6.8 Hz, 2H, CH$_2$), 2.50 (s, 3H, CH$_3$), 3.80 (t, J = 7.2 Hz, 2H, CH$_2$), 7.12−7.35 (m, 12H, H-Ar), 7.50 (d, J = 8 Hz, 2H, H-Ar); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ: 11.05, 21.32, 23.56, 46.30, 125.64, 126.49, 126.54, 128.47, 128.96, 129.05,12 129.28, 129.60, 129.68, 130.12, 131.65, 135.25, 136.88, 138.63, 147.26; MS (70eV) m/z (%): 352 (M$^+$, 70), 323 (52), 309 (59), 295 (46), 15 (37); Anal. Calcd for $C_{25}H_{24}N_2$: C 85.19, H 6.86, N 7.95. Found: C 85.17, H 9.21, N 7.55%.

2-(4-Methoxyphenyl)-4,5-diphenyl-1-propyl-1H-imidazole (6k).

White needles; Yeild: 92%; m.p. 76−80 °C, IR IR (KBr ν_{max}, cm^{-1}): 3016 (C-H aromatic), 1628 (C=C aromatic), 1510 (C=N), 1152 (C-O-Ar); ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 0.50 (t, J = 6.8 Hz, 3H, CH$_3$), 1.33 (m, J = 6.8 Hz, 2H, CH$_2$), 3.05 (s, 3H, CH$_3$), 3.81 (t, J = 7.2 Hz, 2H, CH$_2$), 7.10−7.30 (m, 12H, H-Ar), 7.46 (d, J = 8 Hz, 2H, H-Ar); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ: 10.69, 21.15, 45.38, 127.08, 129.64, 133.57, 138.01, 157.97, 174.82; MS (70eV) m/z (%): 368 (M$^+$, 59), 337 (52), 323 (59), 309 (48), 234 (46), 31 (30); Anal. Calcd for $C_{25}H_{24}N_2O$: C 81.49, H 6.56, N 7.60. Found: C 51.46, H 6.58, N 7.61%.

3. Results and discussion

Many recent papers are illustrating the use of nanosized catalysts in organic reactions. That is pointing out that they are a potential green catalyst due to low waste, ease of handling, high catalytic activity and recyclable in reaction conditions. In an initial study, we considered as a model reaction the condensation of benzaldehyde with aniline, benzil and ammonium acetate in ethanol under microwave irradiation as a model reaction.. Different catalysts such as $AlCl_3$, $SbCl_3$, Al_2O_3, BaO, Montmorilonit-K10, and nanocrystalline $MgAl_2O_4$ were tested under microwave irradiation, but the best results were found

Table 1. Effect of different catalysts on the reaction.

Entry	Catalyst	Time (min)	Yield (%)
1	$AlCl_3$	20	68
2	$SbCl_3$	16	72
3	Nanocrystalline $MgAl_2O_4$	11	93
4	Al_2O_3	35	55
5	BaO	30	70
6	Montmorilonit-K10	20	75

in case of nanocrystalline $MgAl_2O_4$. Therefore, nanocrystalline $MgAl_2O_4$ was the most effective catalyst in terms of reaction time, yields, and products purity while other catalysts formed the product with the lower yields in longer times (Table 1). To explore the advantages of nano $MgAl_2O_4$ as a catalyst for the synthesis of the model compound **4a**, we compared results reported in the literature for this reaction mediated by other catalysts (Table 2). It is clear from this table that nanocrystalline $MgAl_2O_4$ is an efficient and environmentally benign catalyst which could be useful in the synthesis of a series of highly substituted imidazoles. The most advantage of present catalyst than previous catalysts is high specific surface area, easy separation, short times, and mild reaction conditions. This catalyst system showed higher activity than the other catalysts listed in Table 2.

To show the effect of microwave irradiation in these reactions, the synthesis of 2-(4-methyl phenyl)-1,4,5-triphenylimidazole investigated as an example in the presence of various amounts of nanocrystalline $MgAl_2O_4$ in present of microwave and thermal condition (Table 3). In all cases, the results showed that the reaction times are shorter and the yields of the products are higher under microwave irradiation. In the absence of catalyst, the yield of the product was

Table 3. Comparison of reaction in present of MW and thermal condition to the synthesis of **6b**.

Entry	$MgAl_2O_4$ (mol %)	Time (min) Yield (%) [a]	Time (min) Yield (%) [b]
1	0.000	11/5	80/8
2	0.007	11/20	80/25
3	0.014	11/37	80/39
4	0.020	11/56	80/59
5	0.028	11/78	80/81
6	0.035	11/93	80/86
7	0.042	17/92	120/86

[a]Microwave irradiation.

found to be very low. The best result was obtained using 0.035 mol% of the catalyst under both conditions.

The possibility of reusability of the catalyst was also investigated. For this purpose, the same model reaction was again studied under optimized conditions. After the completion of the reaction, catalysts were recovered and separated by centrifugation, and reused for a similar reaction. As indicated in Table 4, recycled catalyst showed no loss of efficiency with respect to reaction time and yield after five successive runs. Also, the FTIR spectrum of recovered catalyst was investigated, which it was identical with principal spectrum (Fig 2).

The generality of this process was demonstrated by a variety of aldehydes bearing either electron-donating or electron-withdrawing groups in the preparation of tri- and tetrasubstituted imidazole derivatives. As shown in Table 5, the condensation of benzil, aldehydes and ammonium acetate on exposure to microwave irradiation gave good to excellent yields of 2,4,5-trisubstituted-1*H*-imidazole derivatives **4a–4k** in presence of nano-$MgAl_2O_4$.

Table 2. Investigation of time and yield of synthesis of imidazoles **4a** using some other catalysts in literature.

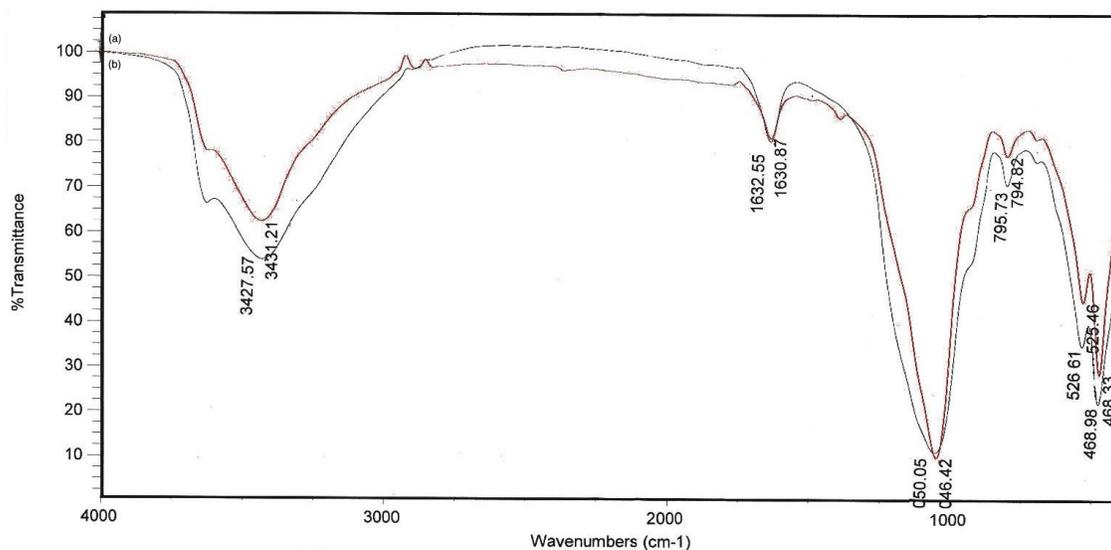| Entry	Catalyst	Time (min)	Yield (%)	Literature
1	$Zr(acac)_4$	150	90	[33]
2	PEG-400	90	88	[34]
3	$InCl_3.3H_2O$	498	82	[35]
4	Montmorilonite K10	90	70	[36]
5	Zeolite	60	80	[36]
6	Nano- crystalline SZ	45	87	[36]
7	*p*-TSA	60	88	[37]
8	SSA	240	73	[38]
9	CAN	360	75	[39]
10	[EMIM]OAc	45	87	[40]

Fig. 2. The FTIR spectra of (a) nano MgAl$_2$O$_4$, (b) Recovered nano MgAl$_2$O$_4$.

Similar methodology was applied for the synthesis of 1,2,4,5-tetrasubstituted-1*H*-imidazole derivatives **6a–6g** by the condensation of benzil, aldehydes, aromatic and aliphatic amines and ammonium acetate in presence of nano-MgAl$_2$O$_4$ under solvent-free conditions (Table 6). It is clear that aldehydes bearing different types of substituents underwent the reaction in good to excellent yields with high purity. Therefore, all the reactions proceeded very efficiently and the results are summarized in Table 6. The nature of substituents on the aromatic ring did not show any remarkable difference in the yield of product and time of the reactions.

The synthesis of 2,4,5-trisubstituted and 1,2,4,5-tetrasubstituted-1*H*-imidazole derivatives using acetic acid for few hours is a well-established procedure [46].

Table 4. Recyclability study of catalyst for the model reaction.

Run	1	2	3	4	5
Yield (%)	93	93	92	91	91

However, this method suffering by several drawbacks such as drastic reaction conditions, low yields, tedious work-up, longer reaction time, difficult to handle. All such drawbacks were overcome in the present procedure as nano-MgAl$_2$O$_4$ is easy to handle including good yields, short reaction times, and simple work-up procedure. The neat reactions were also attempted under conventional heating, keeping similar reaction conditions. The direct heating of reactants without solvent took more time for completion of reactions and gave the products with low yields.

Table 5. Nanocrystalline MgAl$_2$O$_4$ promoted synthesis of triarylimidazoles under MW irradiation.

Entry	R	Product	Time (min)	Yield (%) [a,b]	m.p. (°C) Found	m.p. (°C) Reported
1	H	**4a**	14	93	273–275	274–276 [41]
2	4-Me	**4b**	11	96	230–232	232–234 [41]
3	4-OMe	**4c**	11	94	229–231	228–230 [41]
4	4-Cl	**4d**	13	92	261–262	260–262 [41]
5	3-Cl	**4e**	14	95	282–284	280–283 [42]
6	2-OMe	**4f**	10	98	207–209	208–210 [43]
7	3-NO$_2$	**4g**	13	91	>300	>300 [43]
8	2-OH	**4h**	12	94	207–209	209–210 [44]
9	4-OH	**4i**	12	92	269–271	268–270 [41]
10	4-Br	**4j**	11	95	251–253	254–256 [41]
11	4-N(Me)$_2$	**4k**	14	96	255–257	257–258 [45]

[a]Isolated products.
[b]All products are characterised by IR, ^{1}HNMR and compared with authentic samples.

Table 6. Nanocrystalline MgAl$_2$O$_4$ catalyzed synthesis of tetraarylimidazoles under MW irradiation.

Entry	R	R'	Time (min)	Product	M.p. (˚C)	Yield (%) [b, c]
1	H	C$_6$H$_5$	15	**6a**	217–218 [43]	91
2	4-Me	C$_6$H$_5$	17	**6b**	185–188 [43]	95
3	4-OMe	C$_6$H$_5$	18	**6c**	177–180 [44]	93
4	4-Cl	C$_6$H$_5$	12	**6d**	148–151 [43]	96
5	4-Br	C$_6$H$_5$	12	**6e**	151–153 [45]	94
6	2-F	C$_6$H$_5$	15	**6f**	165–168 [a]	93
7	2-OH	C$_6$H$_5$	18	**6g**	253–255 [44]	90
8	H	n-Pr	10	**6h**	87–89 [a]	93
9	4-Cl	n-Pr	8	**6i**	85–87 [a]	96
10	4-Me	n-Pr	12	**6j**	78–83 [a]	94
11	4-OMe	n-Pr	7	**6k**	76–80 [a]	92

[a]Spectroscopic data of new synthesized compounds.
[b]Isolated products.
[c]All products are characterized by IR, ^{1}H NMR and compared with authentic samples.

4. Conclusion

In conclusion, we have developed a general, facile and efficient one-pot multi-component synthesis of 2,4,5-trisubstituted-1*H*-imidazole and 1,2,4,5-tetrasubstituted-1*H*-imidazole derivatives using reusable catalysts nano-MgAl$_2$O$_4$ under microwave irradiation, which is an environmentally friendly method. It is expected that the combined use of microwave irradiation will make further development and utilization in organic synthesis and material chemistry. The merits of low pollution, ready operation, improved yields involved made it an attractive approach to such significant compounds.

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