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Synthesis of Highly Substituted Imidazole's *via* a Multi-Component Condensation Using Sulphamic Acid Promoted by Microwave

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ABSTRACT
A significant, synthesized by the eco-friendly benign, one-pot synthetic method has been established for 1,2,4,5-tetrasubstituted imidazoles. Under the mild conditions, the synthetic series provides excellent isolated yields via a multi-component condensation in presence of sulphamic acid under the microwave-assisted. Make this protocol green and fascinating. The utility of this methodology is demonstrated by its short synthesis, smooth reaction profile, extremely cheap reagents, mild reaction condition, simple workup, and high yield.
Keywords: *tetra substituted imidazole, sulphamic acid, multi-component reaction, microwave synthesis.*

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INTRODUCTION

Microwave heating is totally different from conventional techniques, the heat gradient in conventional heating is from the heating device to the medium, heat is disintegrated inside the irradiation moderately mass heating, and heat is transferred from the media from the outside. Afterward, in the case of conventional techniques heat transfer is depending on thermal resistance, temperature variation throughout the material, thermal conduction, and thus temperature rise is frequently slow. Due to the mass warming effect, microwave irradiation can produce mash faster temperature increase, depending on microwave power and loss factor of the material getting irradiating [1]. The rapid heating systemformed in microwave ovens is frequently used by a noteworthy proportion of eco-friendly. The methodology has also been used in a variety of degradation processes, along with the hydrolysis of peptides and proteins. In organic synthesis, that can advantage enormously from these methods, but still, a lot to improve. Microwave-assisted organic synthesis is a rapidly evolving field of organic synthesis. Recently, numerous synthetically important reactions such as oxidation, condensation, halogenation, and alkylation have been reported previously [2, 3].

Because of their broad range of uses in pharmacological libraries for drug development, multi-component, and one-pot synthesis are extremely significant [4-8]. MCRs are highly divergent, resulting in a remarkable increase in molecular diversity in a single step. As a synthesized helpful approach for the synthesis of a high range of heterocyclic compounds, microwave irradiation-induced organic condensation has sparked a lot of attention [9-12].

The imidazole's moiety is found in a large spectrum of certainly generating molecules as a key constituent of five-membered heterocyclic compounds [13, 14]. Imidazole moiety has a wide range of medicinal and pharmaceutical activity[15, 16]. The imidazole's compounds pharmacological importance has given it a prominent structure in many synthetic drugs, such as fungicides [17], herbicides [18], plant growth regulators [19] and therapeutic agents [20] and etc. The synthesis of tetra-substituted imidazoles, numerous methods have been planned, including4-component condensation of 1,2-diketone, aromatic aldehydes, primary amines along with ammonium acetate in presence of under among many catalyst, Hy-zeolites [21], carbon-based solid acid [22], molecular iodine [23], $InCl_3.3H_2O$ [24], 3-methyl-1-(4-sulfonic acid)-butylimiduzoliuhydrogen sulfate [25], $HClO_4$-SiO_2 [26], BF3. SiO_2[27], bronsted acidic liquid [28],ZnO[29], and heterocope rearrangement [30] under the microwave-assisted condensation of 1,2-diketone with aryl nitril along with primary amine [31].

Unfortunately, numerous procedures for making tera-substituted imidazoles derivatives have limitations such as long reaction durations, poor yields, and in some cases, violent reaction conditions [32]. To

overcome these drawbacks, researchers are still looking for mild, significant catalysts with high catalytic activity, and a faster reaction time that can be used in solvent free conditions and microwave irradiation techniques to synthesis tetra-substituted imidazoles derivatives.

Sulphamic acid might have been identified as useful as an attractive solid acid catalyst for reactions such as polymeric ether synthesis, isoarmyl acetate synthesis, and protection of functional groups. In addition, some significant organic modifications, such as the Biginelli condensations and Beckmann rearrangement, were found [33], Have been effectively carrying in the presence of sulphamic acid. For the synthesizing of substituted imidazoles, we developed a significant and environmentally favorable approach. The synthesis of highly substituted imidazoles is described here using a one-pot synthesis of benzyl with substituted benzaldehyde, aniline, ammonium acetate in the presence of an affordable catalyst and non-toxic, sulphamic acids.

MATERIAL AND METHODS

Analytical grade chemicals were utilized in the experiment. On pre-coated silica gel 60 F254 plates, analytical TLC was performed, and the spots detected were used under UV light or an iodine chamber. The melting point given by the open capillary tube is uncorrected. IR-spectra were acquired using a Perkin-Elmer FTIR spectrophotometer for all products. 1H-NMR spectra were collected using an NMR spectrometer in chloroform. On a GCMS-QR 2010 mass spectrometers are used to the mass spectra. The reaction occurred in microwave open (CE2977 Samsung).

General procedure forsynthesis of 1,2,4,5-tetraaryl imidazole's

A mixture ofbenzil (10mmol), aniline(10mmol), ammonium acetate (20mmol), aromatic aldehyde (10mmol), and catalyst amount of sulphamic acid (2 mmol) were liquified in 15 ml ethanol in a 50 ml beaker, stirred for few seconds and placed in microwave oven for irradiation at 600 Watt for 135 to 168 sec. After completion of the reaction was checked by TLC. The reaction mixture was cooled to ambient temperature and poured in ice-cold H_2O to get the precipitate. It was isolated by filtration, wash with H_2O, and dried to give the solid product. The solid product was purified by crystallization by using ethanol as a solvent to obtain a solid product (1a to 1j).

Spectral data of synthesized compounds

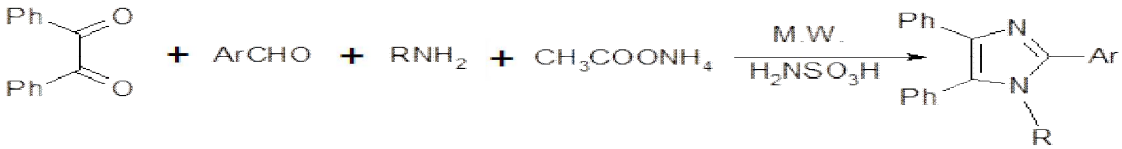
1b. 2-(4-Chloro-phenyl)-1,4,5 triphenyl imidazole: M.P. 152°C.FT-IR (KBr):1620, 1585, cm^{-1}, 1H NMR ($CDCl_3$, δ ppm): δ = 7.23-7.56 (m, 15H), 7.65-7.68(d, 2H), 7.89-7.95(d, 2H).

1j. 1-Benzyl-2,4,5 –triphenyl imidazole: M.P. 163°C.FT-IR (KBr): 2987,1621, 1585, 1482 cm^{-1}, 1H NMR ($CDCl_3$): δ 5.16 (s, 2H, CH_2), 7.10-7.83 (m, 20H, Ph).

RESULTS AND DISCUSSION

Microwave irradiation in organic synthesis for accelerating reaction rate is a new approach. Microwaves have grown popular among chemists as a strategy to increase both classical and novel chemical processes having shortening reaction time and increasing yield. The presence of sufficient commercial microwave equipment, household ovens, and mono mode reactors has also played a vital role in the development of this approach.

As a result, we decided to look into the efficacy of sulphamic acid as anacid catalyst in the synthesis of 1,2,4,5-tetrasubstituted imidazoles by using microwave techniques in this study.Under microwave irradiation, benzoin, aniline, and ammonium acetate with benzaldehyde react fast with sulphamic acid catalyst and alcohol give corresponding 1,2,4,5-tetrasubstituted imidazoles All the reaction products are depicted in **Table 1.**Fascinatingly, 4-Hydroxy, 4-Chloro, 2- Nitro, and 4-Dimethylamine, and aromatic aldehyde with electron-donating and electron-withdrawing substituent to obtain the desired1,2,4,5-tetrasubstituted imidazoles effectively and high yield.



Scheme 1: one-pot condensation of 1,2,4,5-tetraaryl imidazoles (1a-j)

CONCLUSION

Finally, using sulphamic acid as an environmentally benign, affordable, and efficient catalyst, we developed a straightforward technique for the synthesized 1,2,4,5-tetrasubstituted imidazoles. This approach has a number of advantages, including quick reaction times, high yield, ease of operation, and simple set-up.

Table – 1 - Physical data of the synthesis compounds (1a-j)

Entry	Ar	R	Watt W	Time Sec.	[a]Yield (%)	[c] M. P. (^{0}C) Found
1a	C_6H_5	C_6H_5	600	141	92	218[25]
1b	4-Cl- C_6H_5	C_6H_5	600	148	91	152[25]
1c	4-OH- C_6H_5	$C_6H_4CH_2$	600	168	94	134[25]
1d	4-N$(CH_3)_2$- C_6H_5	C_6H_5	600	135	90	245[26]
1e	2-NO_2- C_6H_5	$C_6H_4CH_2$	600	152	90	152[26]
1f	2-Cl- C_6H_5	$C_6H_4CH_2$	600	138	94	140[26]
1g	3-OH- C_6H_5	C_6H_5	600	147	91	198
1h	4-OCH_3- C_6H_5	$C_6H_4CH_2$	600	145	85	158[25]
1i	3,4-$(OCH_3)_2$- C_6H_5	C_6H_5	600	165	84	181[25]
1j	C_6H_5	$C_6H_4CH_2$	600	145	87	163[26]

[a]Isolated yield
[c]All product m.p. compare with literature

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