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N-(Phenylsulfonyl)benzenesulfonamide (NPBSA): A new organo-catalyst for synthesis of 2-aryl-4,5-diphenyl-1*H*-imidazoles.

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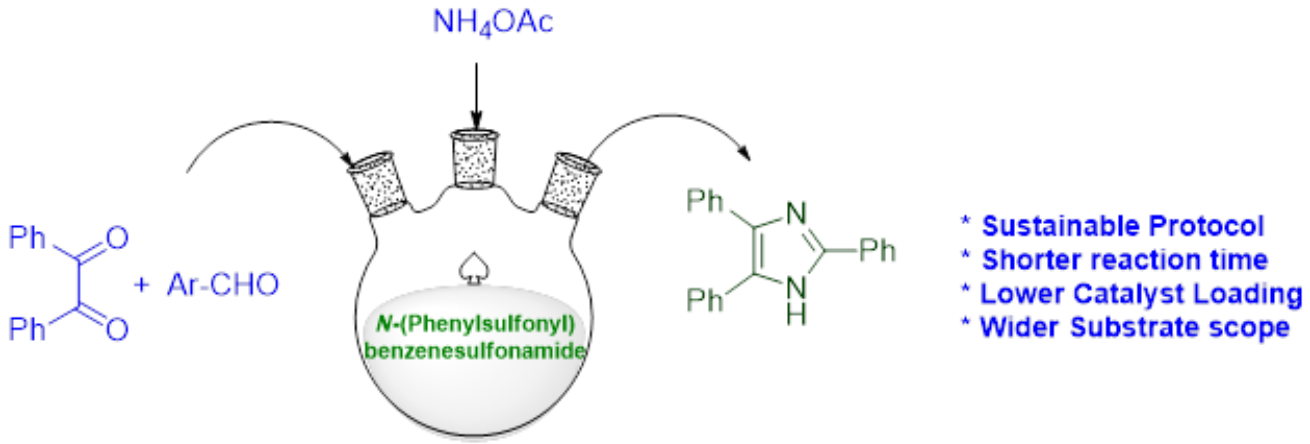
Abstract

A mild and efficient protocol to construct a series of 2-aryl-4,5-diphenyl-1*H*-imidazoles employing *N*-(phenylsulfonyl)benzenesulfonamide (NPBSA) as an effective organo-catalyst via solvent-free cyclo-condensation of structurally diverse organic aldehydes, benzil, and ammonium nitriles, has been developed and described. Prominent features of this environmentally friendly, high-yielding, cost-effective, and NPBSA-catalyzed protocol include a simple experimental procedure, a short reaction time, a simple work-up, the ability to tolerate a variety of functional groups, and excellent yields, making it a safer and more economical alternative to hazardous Lewis acid catalyzed methods.



Keywords: 2-Aryl-4,5-diphenyl-1*H*-imidazoles, *N*-(phenylsulfonyl)benzenesulfonamide

Introduction

Triaryl imidazole (Figure 1) derivatives have many pharmaceutical activities, such as anti-allergic,[1] anti-inflammatory,[2] anti-tumor,[3] anti-bacterial,[4] anti-viral,[5] and potent α-glucosidase inhibitor[6] properties. Triarylimidazoles are prepared from benzil, 2-hydoxy-benzaldehyde, and ammonium acetate, is one of the important routes[7] for the synthesis of the drug substance Trifenagrel. Due to the wide range of applications of substituted imidazoles, there are several methods reported in the literature for their synthesis. Refluxing aldehydes, benzil, and ammonium acetate in acetic acid for many hours is the classical route.[8]

Later, a method from three components, i.e., condensation of benzil, benzaldehyde derivatives, and ammonium acetate was frequently employed. In this procedure, [HeMIM]BF_4,[9] $Eu(OTf)_3$,[10] Kagin type hetero-polyacid,[11] $HClO_4SiO_2$,[12] iodine,[13] zeolite HY and silica gel,[14] silica sulfuric acid,[15] $NiCl_2$ $6H_2O$,[16] $Yb(OTf)_3$,[17] $Y(TFA)_3$,[18] copper(II) trifluoroacetate,[19] boric acid,[20] SiO_2-Cl,[21] $ZrCl_2$,[22] sodium bisulfate,[23] $InCl_2.3H_2O$,[24] ionic liquids,[25-28] $(NH_4)_6Mo_7O_{24}·4H_2O$,[29] *N*-bromosaccharin (NBSa),[30] Fe_3O_4/SO_3H@zeolite-Y,[31] TMSOTf,[32] nanoparticaes,[33] Nanotubes,[34] Various sugars,[35] Fe_3O_4,[36] Fe_3O_4@HA,[37] Diethyl bromophosphate,[38] [Hmim]TFA,[39] microwave Irradiation[40] were used as catalysts to synthesize triaryl imidazoles. However, these methods have some disadvantages. Thus, the development of a simple and efficient protocol for the synthesis of 2-aryl-4,5-diphenyl-1*H*-imidazoles is an active area of research with scope for further improvements toward milder reaction conditions and higher product yields. To overcome the problems associated with literature methods, we employed the inexpensive, environment-friendly solid acid catalyst *N*-(phenylsulfonyl)benzene sulfonamide (Figure 1) for the synthesis of 2-aryl-4,5-diphenyl-1*H*-imidazoles.

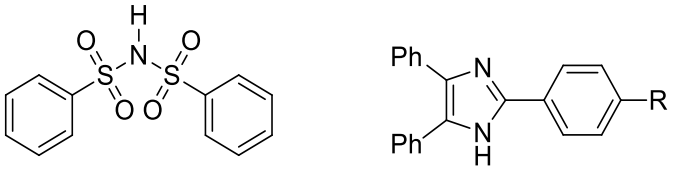
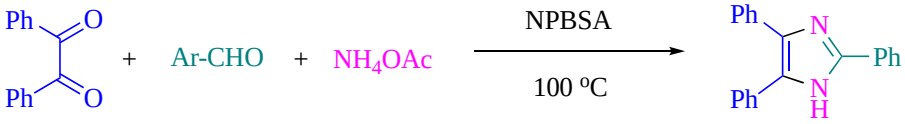


Figure 1. Structures of catalyst NPBSA and 2-aryl-4,5-diphenyl-1*H*-imidazoles.

The literature showed that *N*-(phenylsulfonyl)benzene sulfonamide, being a bi-sulfonamide, has a strong acidity with an pK_a of 1.45.[41] It is a commercially available organic compound (CAS No. 2618-96-4) used as a brightener in nickel baths[42] and pharmaceutical intermediates[43] our group has experience with similar research work.[44-46]

This research describes the first-time the first use of *N*-(phenylsulfonyl)benzene sulfonamide as an efficient and eco-friendly acidic organic catalyst for synthesizing 2-aryl-4,5-diphenyl-1*H*-imidazoles (Scheme 1) in solvent-free conditions with high yields.



Scheme 1. NPBSA catalyzed synthesis of 2-aryl-4,5-diphenyl-1*H*-imidazoles.

Results and Discussion

Initially, the reaction of benzaldehyde, benzil, and ammonium acetate in a mol ratio of 1:1:3 was chosen as a model reaction. To understand the importance of a catalyst for this reaction, control experiments were performed in the absence of the catalyst. From this study, it was observed that the reaction does not proceed in the absence of a catalyst at room temperature, and at increased temperature up to 80 ^{o}C, the reaction proceeds, but the yield of the product was only 25%. (Table 1, entry 2), this indicates that the catalyst is essential for this conversion.

The performance of *N*-(phenylsulfonyl) benzenesulfonamide as the catalyst was studied. The impact of catalyst loading on reaction time and yields was investigated. It was observed that an increase in the catalyst loading increased the product yield with a reduction in reaction time. The catalyst loading beyond 5 mol % was not advantageous (Table 1, entries 3-5).

Temperature played an important role in synthesizing 2-aryl-4,5-diphenyl-1*H*-imidazoles in the presence of the catalyst. The temperature effect was examined at ambient, 50 ^{o}C, and 100 ^{o}C with 5 mol % of NPBSA as a catalyst. The reaction proceeded well at 100 ^{o}C as compared to 50 ^{o}C. (Table 1, entries 6-7). Maximum yield was practically observed with 5 mol % catalyst at temp 100 ^{o}C; hence 5 mol % catalyst loading and temperature 100 °C were chosen for further study.

Table 1. Temperature and catalyst loading effect on the 2-aryl-4,5-diphenyl-1*H*-imidazoles

Entry	Catalyst Loading (mol %)	Temperature (^{o}C)	Time (min)	[b]Yield (%)
1	-	RT	300	5
2	-	80	300	25
3	2.5	r.t	100	50
4	5	r.t	100	60
5	10	r.t	100	60
6	5	50	60	75
7	5	100	60	91

[a]The condensation reactions of benzil (1 mmol), benzaldehyde (1 mmol) and ammonium acetate (3 mmol).
[b]Isolated yields.

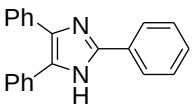
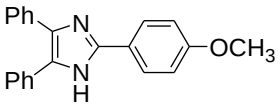
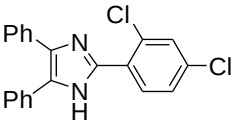
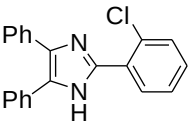
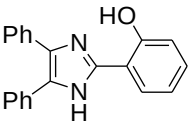
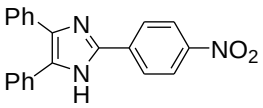
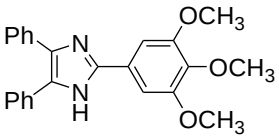
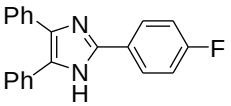
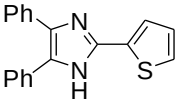
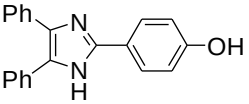
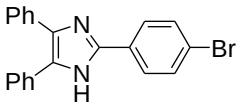
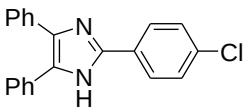
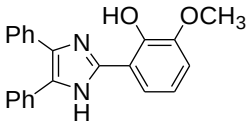
To investigate the substrate scope, optimized conditions were applied to substituted aromatic, and heterocyclic aldehydes and the results are shown in Table 2.

In the course of the study, we observed that this protocol showed good substrate compatibility for aromatic aldehydes to afford the corresponding 2-aryl-4,5-diphenyl-1*H*-imidazoles in high yields.

We found that for aldehydes bearing either electron-releasing (methoxy, hydroxy, dimethylamino, methylenedioxy) or electron-withdrawing substituents (chloro, nitro) in the *ortho* or *para* positions, the reaction proceeded very efficiently in all the cases. (Table 2).

Yields were in the range of 85 to 91%. It seems that the effect of substituted groups on the aromatic aldehydes is not very strong.

Table 2. Syntheses of 2, 4, 5-triarylimidazoles using NPBSA as a catalyst

				
2 a (45 min, 91 %)	**2 b (65 min, 88 %)**	**2 c (65 min, 88 %)**	**2 d (55 min, 85 %)**	**2 e (65 min, 86 %)**
				
2 f (65 min, 88 %)	**2 g (55 min, 86 %)**	**2 h (65 min, 88 %)**	**2 i (70 min, 86 %)**	**2 j (60 min, 91 %)**
				
2 k (min, 85 %)	**2 l (55 min, 90 %)**	**2 m (70 min, 85 %)**		

[a] The condensation reactions of benzil (1 mmol), benzaldehyde (1 mmol) and ammonium acetate (3 mmol).
[b] Isolated yields.

To prove the excellence of NPBSA in the one-pot synthesis of 2-aryl-4, 5-diphenyl-1*H*-imidazoles, the results obtained for the model reaction in the present work with NPBSA catalyst were compared with the previously reported catalysts.

Table 3. Comparaison of performance of Catalyst NPBSA with other catalysts

Entry	Catalyst	Solvent	Temp ^{o}C	Time	Yield	Reference
1	$InCl_3.3H_2O$	Methanol	RT	12 hr	73	24
2	Copper (II) trifluoroacetate	-	100	4 hr	85	41
3	SiO_2-Cl	-	100	30 min	78	21
4	lacticacid	-	160	180 min	90	43
5	$KMnO_4$	Ethanol	Reflux	69 min	89	42
6	NPBSA	-	100	60 min	91	This work

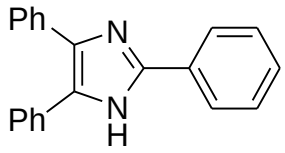
Conclusions

In conclusion, we have developed a simple method for synthesizing 2-aryl-4,5-diphenyl-1*H*-imidazoles using *N*-(phenylsulfonyl)benzenesulfonamide as an efficient acid catalyst. This method offers several advantages including high yields and a simple experimental work-up procedure, which is proved to be a useful method for the synthesis of 2-aryl-4,5-diphenyl-1*H*-imidazoles.

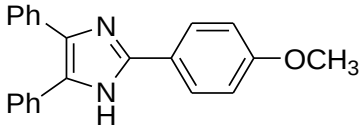
Experimental Section

General: All the chemicals and solvents used were purchased from commercial sources Aldrich, Sd-Fine, Spectrochem and Avra chemical companies. All those chemicals were used without further purification. The purity determination of the starting materials and reaction monitoring were accomplished by thin layer chromatography (TLC) on Merck silica gel G F 254 plates. Melting points of all the compounds were recorded on a Thermomik Campbell melting point apparatus, which has an oil bath system, and are uncorrected.
^{1}H-NMR spectra of products 2a to 2g were recorded on MR 400 Agilent Technologie NMR spectrometer using tetramethylsilane (TMS) as an internal standard and DMSO-d_6 as a solvent. ^{1}H-NMR spectra of products 2h to 2m were recorded on a Bruker instrument at 300 using tetramethylsilane (TMS) as an internal standard and DMSO-d_6 as a solvent.
Chemical shifts are reported in parts per million (ppm, δ), and coupling constants (*J*) are reported as Hertz (Hz). Splitting patterns are designated as singlet (s), doublet (d), triplet (t), broad (br), and quartet (q). Splitting patterns that could not be interpreted are designated multiple (m). All the products are known compounds and were identified by comparison of melting point, ^{1}H-NMR data with literature references.

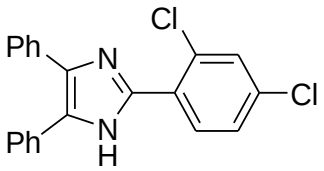
Procedure for preparation of 2-aryl-4,5-diphenyl-1*H*-imidazoles: In a 100 ml round bottom flask equipped with a stirrer and reflux condenser in an oil bath benzaldehyde 2.5 g (23.6 mmol), benzil 5 g (23.6 mmol), ammonium acetate 5.5 g (70.7 mmol), and NPBSA 0.35 g (1.17 mmol) were added. The reaction mixture was stirred at 100 ^{o}C. The progress of the reaction was monitored by thin layer chromatography (TLC) using mobile phase 10% EtOAc in petroleum ether. When TLC indicated the completion of the reaction, ice-cold water was added to the reaction mixture and obtained solid was filtered under suction, washed with ice-cold water, and dried to afford the pure product and re-crystallised in 10% acetone in methanol (if required). All products were characterized by comparing melting points and ^{1}H-NMR those reported in the literature.
Characterization of Synthesized Compounds (2a-m).



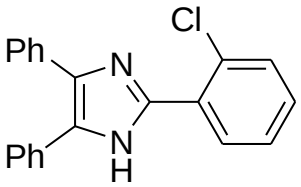
2, 4, 5-Triphenyl-1*H*-imidazole (2a). Colourless solid, Yield 91%. Observed Melting Point : 278 ^{o}C, Literature : 276 -277 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$, TMS): 7.91-7.92 (d, 2H), 7.26-7.66(m, 13 H), 9.41 (s, 1H, NH) ppm



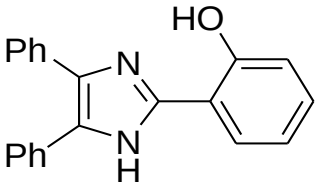
2-(4-Methoxyphenyl)-4,5-diphenyl-1*H*-imidazole (2b). Colourless solid, yield 88%. Melting Point Observed: 231^{o}C, Literature: 228- -231 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ (ppm): 3.86 (s, OCH_3),6.97 -6.98(d, J8.7 Hz, 2H), 7.26 -7.55 (m, 10H, Ph), 7.84-7.85 (d, J8.7Hz, 2H), 9.27 (s, 1H, NH) ppm.



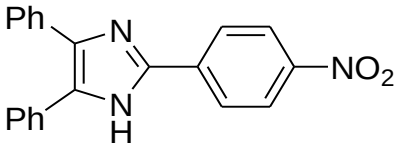
2-(2,4-Dichlorophenyl))-4,5-diphenyl-1*H*-imidazole (2c). Colourless solid. Yield 88%. Melting Point Observed: 177 °C, Literature: 176-178 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 12.66 (s,1H, N-H) 7.74-7.82 (d, 2H), 7.53-7.19 (m, 12H, Ph-H).



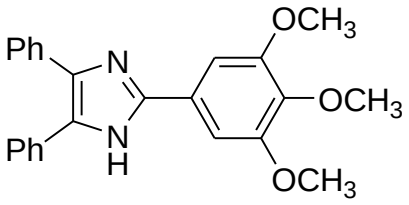
2-(2-Chlorophenyl)-4, 5-diphenyl-1*H*-imidazole (2d). Colourless solid. Yield 85%. Melting Point Observed: 196 °C, Literature: 195 -197 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 10.23 (s, 1H, NH), 8.45 -8.47 (d, 1H), 7.68–7.69 (d, 2H), 7.26–7.48 (m, 11H, Ph-H).



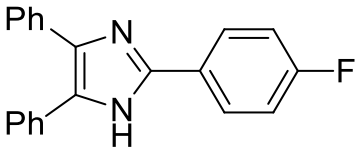
2-(2-Hydroxyphenyl)-4,5-diphenyl-1*H*-imidazole (2e). Colourless solid. Yield 86%. Melting Point Observed: 256 °C, Literature: 252 -257 °C. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): 9.47 (s, 1H, NH), 6.91 – 7.56 (m, 14 H, Ph-H), ppm



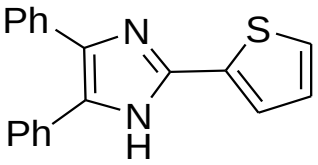
2-(4-Nitrophenyl)-4, 5-diphenyl-1*H*-imidazole (2f). Colourless solid. Yield 88%. Melting Point Observed: 231 °C, Literature: 227-231 °C. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): 7.50 – 7.22 (m, 10 H), 8.30 (m, 4 H), 13.09 (s, 1H, NH) ppm.



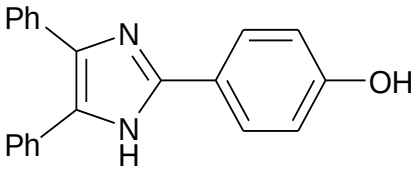
2-(3, 4, 5-Trimethoxylphenyl)-4,5-diphenyl-1*H*-imidazole (2g). Colourless solid, Yield 86%. Melting Point Observed: 164 °C, Literature: 163-165 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 12.56 (s, 1 H, NH), 7.53 – 7.18 (m, 12H, Ar H), 3.84 (6 H, 2 OCH_3), 3.68 (3H, OCH_3).



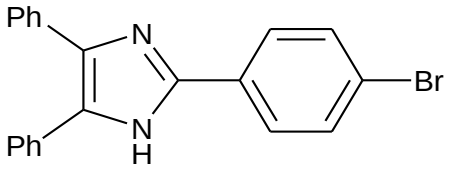
2-(4-Flurophenyl)-4, 5-diphenyl-1*H*-imidazole (2h). Solid, Yield 88%. Melting Point Observed: 239 °C, Literature: 239-241 °C. ^{1}H NMR (300 MHz, DMSO-d_6): 12.71 (s, 1H), 8.09 (d, 2H), 7.48 – 7.31 (m, 4H), 7.31 – 7.29 (m, 8H).



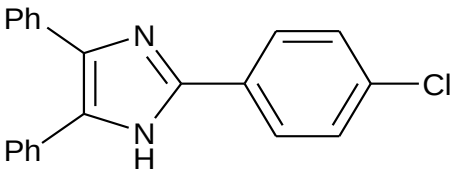
4, 5-Diphenyl-2-(thien-2-yl)-1*H*-imidazole (2i). Light Brown solid, Yield 86%. Melting Point Observed: 261-262 °C, Literature: 260 – 261 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 12.83 (s, 1H), 7.70 –7.67 (m, 1H), 7.54 – 7.45 (m, 5H), 7.40 – 7.23 (m, 6H), 7.12 (q, J 4.0 Hz, 1H).



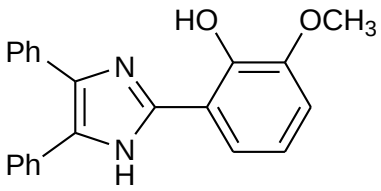
2-(4-Hydroxyphenyl)-4,5-diphenyl-1*H*-imidazole (2j). Colourless solid, Yield 91%. Melting Point Observed: 260-261 °C, Literature: 260-262 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 12.27 (s, 1H, N-H), 9.54 (s, 1H, OH), 7.89 (d, 2H, 8.5, Ph-H), 7.547.46 (m, 5H, Ph-H),7.37-7.30 (m, 5H,Ph-H), 6.89-6.87 (d, 2H, Ph-H).



2-(4-Bromophenyl)-4,5-diphenyl-1*H*-imidazole (2k). Colourless solid, yield 85%. Melting Point Observed: 222 -224 °C, Literature: 223-225 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 12.79 (s, 1H), 8.01 (d, J 7.7 Hz, 3H), 7.69 – 7.59 (m, 3H), 7.52 – 7.46 (m, 5H), 7.36 – 7.30 (m, 3H).



2-(4-chlorophenyl)-4,5-diphenyl-1*H*-imidazole (2l). Colourless solid, Yield 90%. Melting Point Observed: 263 °C, Literature: 263-264 °C. ^{1}H NMR (300 MHz, DMSO-d_6): δ 12.79 (s, 1H), 8.07 (t, J 6.4 Hz, 2H), 7.56 – 7.43 (m, 6H), 7.43 – 7.33 (m, 3H), 7.32 – 7.21 (m, 3H).



2-methoxy-6-(4,5-diphenyl-1*H*-imidazol-2-yl) phenol(2m). Colourless solid, Yield 85%. Melting Point Observed: 218-220 °C, Literature: 220-221 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 13.02 (s, 1H, N-H), 7.62 – 6.87 (m, 13 H, Ph-H)

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SupplementaryMaterial

Copies of ^{1}H-NMR Spectra of compounds **2a–m** are given in the Supplementary Material file associated with this paper.

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