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Ultrasound assisted synthesis of 2, 4, 5-triarylimidazoles catalyzed by Baker's yeast

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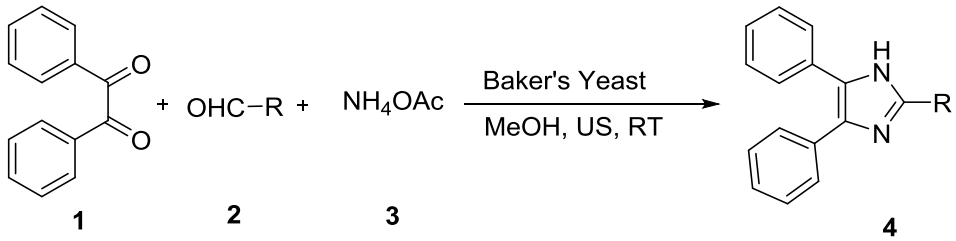
Abstract:
A very simple, efficient and sustainable biocatalytic route for obtaining 2,4,5-triarylimidazole derivatives is developed and presented. This involves one pot three component cyclocondensations of 1,2-diketone, ***(1)*** *aryl aldehydes* ***(2)*** *and ammonium acetate* ***(3)****, catalyzed by active dry baker's yeast as a whole cell biocatalyst at ambient temperature and pressure under ultrasonication as a non-conventional energy source.*



Key Words: *Triarylimidazoles, Biocatalysis, Baker's yeast, Ultrasonication, Green Chemistry.*

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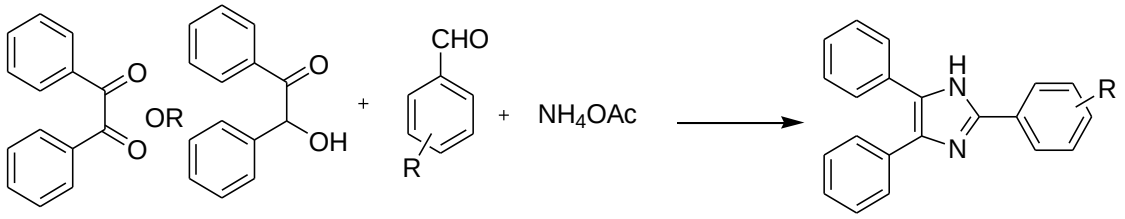
I. INTRODUCTION:

Highly substituted imidazole derivatives are the significant intermediates in the synthetic routes leading to molecules of therapeutical interest. Omeprazole [1], losarton, pimobendan [2], climetidine, lansoprazole [3], eprosartan and olmesartan are some of the leading drugs in the market containing imidazole nucleus as a part of their structures [4].

Numerous substituted imidazole derivatives are promising bioactive compounds and they exhibit herbicidal [5], fungicidal [6], analgesic [7], anti thrombotic [8], anticoagulant [9], anti-inflammatory [10], antiviral [11], anti tubercular [12], antibacterial [13], antitumor [14] and COX-2 inhibitory [15] activities. In addition triarylimidazole derivatives have also proved their importance in the field of photography as photosensitive materials [16].

Some substituted triarylimidazole derivatives are recognized as selective antagonists of the glucagon receptor [17], inhibitors of IL-1 biosynthesis [18], plant growth regulators [19] and pesticides [20]. They are also useful as potential corrosion inhibitors for transition metals such as iron, copper, zinc and their alloys [21].

Due to the wide applications and potential significances of triarylimidazoles in the field of synthetic as well as medicinal chemistry, number of synthetic methods have been developed and reported for their construction. In 1882, Radziszewski and Japp reported the first synthesis of a highly substituted imidazoles [22] from a 1,2-dicarbonyl compounds, aldehydes and ammonia. But the most popular method for synthesis of triarylimidazoles is one pot three-component cyclocondensation of 1,2-diketones/ α-hydroxyketones, aldehydes and ammonium acetate.



Scheme 1

Literature survey reveals that number of attempts have been made to replace the traditional catalysts used in this route by Zn-Al_2O_3, [23] PCl_5 [24] in phosphoric acid [25] as well as in DMSO [26] and H_2SO_4 [27] to accelerate the cyclocondensation. There are reports on the use of silica-gel/HY [28], DMF [29], MW/Al_2O_3 [30] etc. Some researchers have also modified the route by employing cellulose supported sulfuric acid [31], potassium dihydrogen phosphate [32], ceric ammonium nitrate [33], ionic liquid [34], reflux acetic acid [35], L-proline [36], zirconium tetrachloride [37], silica-immobilized sulfuric acid [38], $InCl_3{\cdot}3H_2O$ [39], $NiCl_2{\cdot}6H_2O/Al_2O_3$ [40], iodine [41], sodium bisulfate [42], ammonium acetate [43], $BF_3{\cdot}SiO_2$ [44] silicagel/$NaHSO_4$ [45] or $HClO_4$-SiO_2 [46], molten TBAB [47], SBPPSA [48], ytterbium triflate [49] Caro's acid-silicagel [50], supported ionic liquid-like phase (SILLP) [51], silica chloride [52], I_2/HDMI [53], lactic acid [54] and Diethyl ammonium hydrogen phosphate [55] etc. Alternative energy resources such as microwave irradiation [56] and ultrasonication [57] have also been utilized to accelerate the synthetic route leading to triarylimidazoles.

Despite of their potential utility, many of these reported methods have disadvantages such as prolonged reaction times, drastic reaction conditions, expensive catalysts and use of toxic organic solvents. Further some of the methods have tedious work-up, possibility of side reactions resulting in poor yields and generation of acidic/metallic wastes.

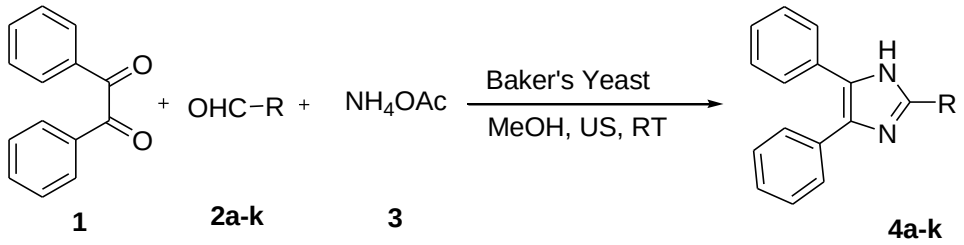
Recently the use of baker's yeast as catalyst (whole-cell biocatalyst) to perform cyclocondensations rapidly, leading to value added organics is valuable methodology in the organic syntheses. It has been revealed from the literature that baker's yeast has not yet been used in synthesis of 2,4,5-trisubstituted imidazoles.

Literature also reveals that the majority of baker's yeast mediated organic reactions have been carried in an aqueous medium so as to retain its catalytic activity. On the other hand an aqueous medium is not recommended for dehydration reactions and therefore the use of enzymes in non-aqueous media has been gaining much importance for condensation and cyclocondensation reactions. There are numerous, advantages of using biocatalysts in organic solvents such as (i) high solubility of most organic compounds in organic solvents, (ii) allowing reactions which are impossible to conduct in water, (iii) relative ease of product recovery from organic solvents and (iv) insolubility of enzymes in organic solvents which permits their easy recovery and reuse, eliminating the need for immobilization.

Considering all the above facts and in continuation with our journey towards synthesis of bioactive molecules accelerated by biocatalysts and biomimics [64-65] the key objective of the present work was to set and develop an efficient route for one pot three component cyclocondensation of 1,2-diketone, aryl aldehydes and ammonium acetate in non-aqueous media (organic solvents) under relatively mild reaction conditions using an easily available, cheaper whole cell biocatalyst, active dry baker's yeast instead of the catalysts reported in the literature [23-55]. The objective was also set in mind to use non-conventional energy source such as ultrasonication for acceleration of the cyclocondensation.

Present work

A very simple, efficient and sustainable biocatalytic route for obtaining 2,4,5-triarylimidazole derivatives is developed and presented. This involves one pot three component cyclocondensations of 1,2-diketone,**(1)** aryl aldehydes **(2a-k)** and ammonium acetate**(3)**, catalyzed by active dry baker's yeast as a whole cell biocatalyst at ambient temperature and pressure under ultrasonication as a non-conventional energy source (**Scheme 2)**.



Scheme 2

II. RESULTS AND DISCUSSION

The investigations were started with an optimization study of model reaction by allowing the cyclocondensation of benzil (5 mmol) 4-methyl benzaldehyde (5 mmol) and ammonium acetate (12 mmol) in the presence of baker's yeast (2 g). To see the effect of reaction medium on the rate and yield of the reaction, the model reaction was carried in various solvents like water, dichloromethane, ethanol and methanol under stirring at room temperature (RT).

Table 1: Effect of solvents on the synthesis of 2,4,5-triaryl imidazole[a] **(Scheme 2)**

Entry	Solvent	Condition	Time (h)	Yield (%)[b]
1	Water	RT	24	nd
2	DCM	RT	24	nd
3	Ethanol	RT	24	42
4	Methanol	RT	24	65
5	Methanol	RT	24	nd[c]

[a]Reaction conditions: Benzil (5 mmol), 4-methyl benzaldehyde (5 mmol), ammonium acetate (12 mmol), baker's yeast (2g) in methanol (30 ml) under ultrasonication.
[b]Isolated yield. [c]Reaction without baker's yeast, nd= Product not detected.

Initially when the reaction was run in water at room temperature, it was found that the cyclocondensation did not occurred even after prolonged stirring (24 h) **(Table 1, entry 1)**. The model reaction was then performed in organic solvent dichloromethane (DCM) and found that the desired product was not formed **(Table 1, entry 2)** even after prolonged stirring at RT (24 h). When the cyclocondensation was carried in ethanol, the noticeable yield of 4,5-diphenyl-2-p-tolyl-1*H*-imidazole (4g) was observed (**Table 1, entry 3**). Inspired by this, then the condensation was run under similar reaction conditions in methanol, the yield has been found to be increased to 65 % (**Table 1, entry 4**). In view of these observations methanol was selected as a reaction medium for the present cyclocondensation reaction.

After having these results the model reaction was carried in methanol under ultrasonication, with the hope to reduce reaction time and increase the yield of the product. It has been known from literature that ultrasonication is one of the most widely used laboratory methods for the disruption of the cells of baker's yeast [61] for the fast release of enzymes. Same reaction when carried in methanol under ultrasonication the reaction time has been decreased to 4.5 h and the yield was found to be increased to 92 % (**Table 2**). To investigate the role of baker's yeast in cyclocondensation, the model reaction was run in the absence of baker's yeast and noted that there was no formation of the desired product even after 24 h of stirring at room temperature in methanol **(Table 1, entry 5)**and even 5 h under ultrasonication (**Table 2,entry 4**).

Table 2: Effect of ultrasonication on reaction time and yields of imidazoles.

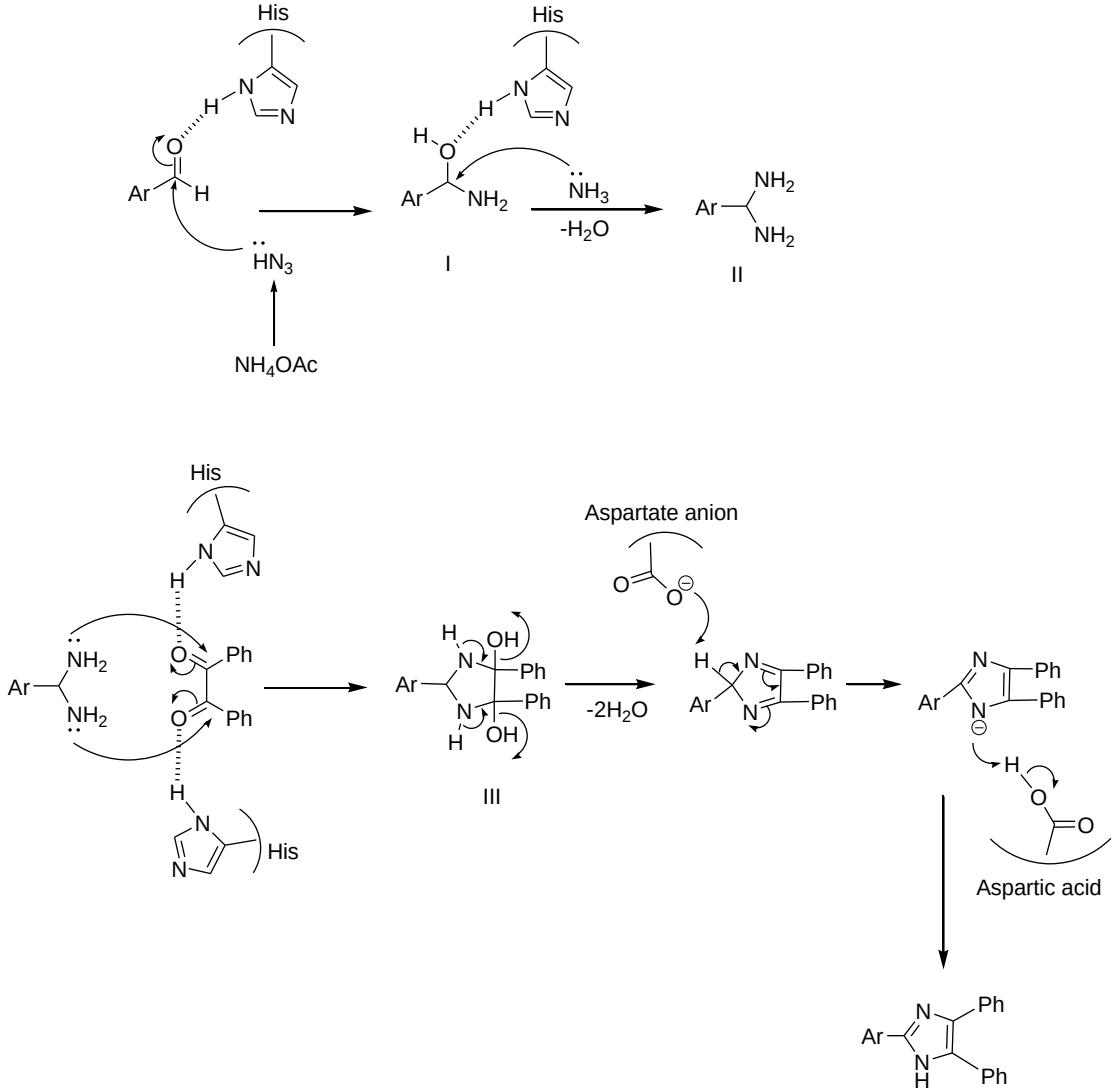
Entry	Solvent	Condition	Time (h)	Yield (%)[a]
1	Methanol	US	4	77
2	Methanol	US	4.5	92
3	Methanol	US	5	92
4	Methanol	US	5	nd[b]

[a]Isolated yield. [b] Reaction without baker's yeast, n.d.= Product not detected.

Subsequently the cyclocondensation of other aryl aldehydes, benzil and ammonium acetate has been carried under the optimized reaction conditions and respective triarylimidazoles have been synthesized. The results are recorded in **Table 3 (Scheme 2)**. All the reactions proceeded smoothly to afford the corresponding 2,4,5-triarylimidazoles.

The imidazoles synthesized by this modified/ developed protocol are already known and their melting points are in good agreement with those reported in the literature. [38,43,58] The purity of the compounds was confirmed by thin layer chromatography and supported by spectral analyses. Spectral data of one of the representative compounds along with spectra are given in supplementary information.

The yeast enzymes have various sites of activations. Among them aspartic acid and histidine are considered here to propose the mechanism of the cyclocondensation leading to substituted imidazoles. The histidine amino acid residue through it's NH group might be enhancing the electrophilic behavior of the carbonyl carbon of the aldehyde by non-covalent hydrogen bonding. Then subsequently nucleophilic ammonia (generated from ammonium acetate) attacks on the electrophilic carbonyl carbon of the aldehyde producing tetrahedral α-hydroxy amine intermediate (I). Dehydration and successive attack of another NH_3 leads to diamine intermediate (II). The condensation of diamine with benzil might be catalyzed by histidine residue as depicted in **Scheme 3** and yields cyclic diol (III). Finally the diol after dehydration and proton transfer gives desired product 2,4,5-triaryl imidazoles. The catalytic role displayed in these steps by aspartic acid residue has been depicted in the proposed mechanism **(Scheme3)**.



Scheme 3: Plausible mechanistic path for the cyclocondensation.

III. CONCLUSION:

In summary, the use of baker's yeast as whole cell biocatalyst to accelerate the cyclocondensation of substituted aryl aldehydes, benzil and ammonium acetate in methanol under ultrasonication, carried for obtaining 2,4,5-triaryl imidazoles have been demonstrated. The catalytic role of baker's yeast is demonstrated. The effect of ultrasonication in acceleration of the biocatalyzed cyclocondensation has also been mentioned. The newly developed protocol has following advantages.

1) Baker's yeast is inexpensive and readily available biocatalyst.
2) Reaction time has been notably reduced.
3) There is no any kind of toxic waste generated during the reaction.

Experimental Section

General procedure for the synthesis of 2,4,5-triaryl imidazoles (4a-k):

Active dry baker's yeast (2 g) was added to methanol (30 ml) and sonicated for 10 minutes. Then benzil (5 mmol), aryl aldehyde (5 mmol) and ammonium acetate (12 mmol) were added to the sonicated reaction mass. The reaction mass was further sonicated at 20 KHz for 4.5h at RT. The progress of the reaction was monitored by thin layer chromatography using ethyl acetate: pet ether (1:9) as solvent system. After 4.5 h the reaction mass was filtered through the bed of celite (2 g). From the filtrate, the solvent methanol was removed under reduced pressure and the crude products isolated were crystallized from ethanol to get corresponding 2,4,5-triarylimidazoles (4a-k). (**Table 3, 4a-k**).

Spectral data of representative compound of the series

Compound (4g): 4,5-diphenyl-2-p-tolyl-1*H*-imidazole

^{1}H-NMR (400 MHz, $CDCl_3$): δ ppm=2.37 (s, 3H, CH_3), 7.19-7.32(m, 8H, Ar-H) 7.50-7.51(m, 4H, Ph-H), 7.75(d, 2H, *J* =8 Hz, Ar-H) and 9.57 (bs,1H, NH, exchangeable with D_2O).

^{13}C-NMR (100 MHz, $CDCl_3$): δppm=21.5, 125.4, 127.3, 127.5, 128.0, 128.7, 129.7, 138.9 and 146.5.

HR-ESI-MS (*m/z*): Calculated for $C_{22}H_{18}N_2$ $[M+ H]^+$: 311.1543, found: 311.1528.

Table 3: Baker's yeast catalyzed synthesis of triarylimidazoles[a] **(Scheme 2)**

Entry	R	Product	Yield(%)[b]	M. P. (ºC)[a]
1	Ph	**4a**	92	274-275
2	4-Br C_6H_5	**4b**	90	253-255
3	4-OCH_3 C_6H_5	**4c**	93	229-230
4	2-Cl C_6H_5	**4d**	88	194-196
5	4-$N(CH_3)_2$ C_6H_5	**4e**	85	258-260
6	4-NO_2 C_6H_5	**4f**	90	240-241
7	4-CH_3 C_6H_5	**4g**	92	228-229
8	2-Furyl	**4h**	91	199-200
9	4-OH C_6H_5	**4i**	83	268-270
10	4-F C_6H_5	**4j**	87	190-191
11	4-Cl C_6H_5	**4k**	90	260-262

[a]Reaction conditions: Benzil (5 mmol), aldehyde (5 mmol), ammonium acetate (12 mmol), baker's yeast (2g) in methanol (30 ml) under ultrasonication.

[b]Isolated yields.

[c]The known 2,4,5-triaryl-1*H*-imidazoles, synthesized by this method are having their melting points in good agreement with those reported in the literature. [38,43,58]

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