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Saccharomyces cerevisiae catalyzed one pot synthesis of isoindolo[2,1-a]quinazoline performed under ultrasonication

Jemin R. Avalani, Devji S. Patel, Dipak K. Raval*

Department of Chemistry, Sardar Patel University, Vallabh Vidyanagar 388120, Gujarat, India

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ABSTRACT

An efficient and simple one pot method has been developed for the synthesis of isoindolo[2,1-a]quinazolines using *Saccharomyces cerevisiae* (baker's yeast) as a whole cell biocatalyst at room temperature. The synergetic effect of baker's yeast and ultrasound irradiation has been discussed.

1. Introduction

Biocatalyzed reactions in organic media have become a popular approach with several reports appearing in the literature [1–4]. The use of aqueous media for such transformations is not desirable as most of the organic substrates are insoluble in water [5]. Biocatalysis in organic solvents have several advantages; high solubility of organic substrates to perform the reaction which is complicated in aqueous media, easy isolation of products and insolubility of enzymes in organic solvents which permits their easy recovery and reuse [6]. Baker's yeast is known to play vital role in functional group conversion and also been reported to catalyze organic transformations effectively such as reduction [7–9], hydrolysis [10], oxidation [11] and condensation [12]. Baker's yeast has been extensively used to carry out various organic reactions and can be used in the synthesis of various heterocyclic compounds such as polyhydroquinolines [13], 4H-pyrans [5], isoxazolines [14] and 1,4-dihydropyridines [15]. Baker's yeast is also reported to show high activity and enantioselectivity in aqueous/organic biphasic systems [16–18]. Recently Silva et al. have reported asymmetric reduction of (4R)-(−)-carvone catalyzed by Baker's yeast in mono- and biphasic systems [19].

Isoindoles are ubiquitous structural motifs of the most common heterocyclic compounds found in both natural products and biologically active compounds. Due to the existence of an array of structurally diverse isoindoles, it is an important substructure of various biologically active natural products such as bhimamycin C, and bhimamycin D which display bioactivities against human ovarian cancer cell lines and are also EP4 receptor agonists in the treatment of pain [20]. It exhibits variety of biological properties such as anti-neoplastic, antiviral [21], antimalarial [22], antitumor [23], and antimicrobial activities [24]. The unique scaffold and pharmacological properties associated with quinazoline nucleus attracted the attention of chemists toward the syntheses of various types of useful compounds in medicinal chemistry. It possesses wide variety of activities like antibacterials [25], antivirals [26,27], antitumorals [28], and many other therapeutic activities [29]. Additionally fused indole derivatives such as indolocarbazoles [30,31], indoloisoquinolines [32–34], indoloquinolines [35–38], and indoloquinazolines [39–41] exhibit a number of remarkable pharmacological properties. Among them, isoindolo[2,1-a]quinazoline showed high cytotoxic activity against a wide range of human cancer cells and at the molecular level are inhibitors of tubulin polymerase and topoisomerase I. This confirms the prospects of using them as anticancer drugs [42,43] and TNF-α inhibition against various inflammatory disorders [44]. Recently the syntheses of these derivatives are reported using camphor sulphonic acid; *p*-toluenesulphonic acid and Montmorillonite K10 as the heterogeneous catalyst by conventional method with longer reaction time [44].

Ultrasound irradiation is a powerful technique, which is being used frequently to accelerate organic transformations [45–48]. It is one of the most widely used laboratory methods for the disruption of cells of baker's yeast for the fast release of enzymes [49].

* Corresponding author. Tel.: +91 2692226856x211.
E-mail addresses: dipanalka@yahoo.com, dk_raval@spuvvn.edu (D.K. Raval).

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The remarkable features of ultrasound irradiation are decrement in reaction time, high efficiency, formation of pure product and waste minimization compared to conventional methods [50].

As a part of our ongoing endeavor for the construction of various heterocyclic compounds [51–59], herein we document the role of baker's yeast under ultrasound at room temperature to afford isoindolo[2,1-a]quinazolines (Scheme 1).

2. Experimental

2.1. Materials and instruments

The following chemicals were used as received. Isatoic anhydride (96%), 2-carboxy benzaldehyde (97%) and amines (≥96%) were purchased from Sigma Aldrich. All organic solvents were obtained from commercial sources and were of analytical grade. Melting points were determined using μThermoCal$_{10}$ (Analab Scientific Pvt. Ltd.) melting point apparatus and are uncorrected. Progress of the reaction was monitored by thin layer chromatography on Merck's silica plates. ^{1}H and ^{13}C NMR spectra were recorded on Bruker Avance 400 MHz instruments using TMS as internal standard. Mass spectra were recorded on Shimadzu LCMS 2010 mass spectrometer. IR spectra were recorded on a FTIR Perkin Elmer Spectrum 100 spectrometer as KBr pellets with absorption in cm^{-1}. Elemental analysis was performed on a Perkin Elmer PE 2400 elemental analyzer. Baker's yeast was obtained from local market. Ultrasound irradiation was performed in a D-compact ultrasonic cleaner with a frequency of 30 kHz and an output power of 250 W. The reaction flask was kept at the maximum energy area in the cleaner, and the surface of the reactants was placed slightly lower than the level of water. The reaction temperature was controlled at 30 °C by addition or removal of water from ultrasonic bath.

2.2. Ultrasound-promoted general synthesis of isoindolo[2,1-a]quinazoline

To the solution of isatoic anhydride **1** (1.22 mmol), 2-carboxy benzaldehyde **2** (1.31 mmol) and amine **3** (1.40 mmol) in THF (5 mL), active dry baker's yeast (400 mg) was added. Then the reaction mixture was sonicated at 30 kHz for stipulated time mentioned in Table 2 at 30 °C. The progress of the reaction was monitored by thin layer chromatography by using ethyl acetate:hexane (5:5) as the eluent. After completion of the reaction the mass was filtered through the bed of celite. From the filtrate, the solvent was removed under reduced pressure and the crude products isolated were crystallized from hot ethanol.

2.2.1. Spectral data of 6-(2-hydroxyethyl)-6,6a-dihydroisoindolo[2,1-a]quinazoline-5,11-dione (*4c*)

^{1}H NMR ($CDCl_3$, 400 MHz): 7.28–8.13 (m, 8H), 6.43 (s, 1H), 4.04–4.20 (m, 2H), 3.75–3.95 (m, 2H), 2.06 (br s, 1H, OH). ^{13}C NMR ($CDCl_3$, 100 MHz): 164.99, 164.91, 138.21, 136.89, 133.70, 132.97, 132.59, 130.56, 128.97, 126.81, 125.23, 125.03, 120.31, 120.23, 71.76, 61.83, 45.40. IR (KBr): 3414 (—OH), 3095 (—CH), 1731 (—C=O), 1635 (—C=C), 1489 ($—CH_2$), 1063 (—C—O) cm^{-1}. ESI-MS: (*m*/*z*, %): 295.0 (100), 276.9 (41), 252.0 (5), 236.0 (11). Anal. Calcd. for $C_{17}H_{14}N_2O_3$ (294.23): C, 69.38; H, 4.79; N, 9.52. Found C, 69.22; H, 4.96; N, 9.70.

3. Result and discussion

A series of experiments were performed with the model reaction of isatoic anhydride **1**, 2-carboxy benzaldehyde **2**, and benzyl amine **3** in the presence of baker's yeast in organic medium under sonication at 30 °C.

3.1. Effects of solvents under ultrasound irradiation

To understand the effect of reaction medium on the rate and yield of the reaction, the model reaction was performed in various solvents like methanol, ethanol, tetrahydrofuran (THF), dichloromethane (DCM), acetonitrile, dimethyl formamide (DMF), Dimethyl acetamide, water and phosphate buffer (pH 6.0). The results are summarized in Table 1.

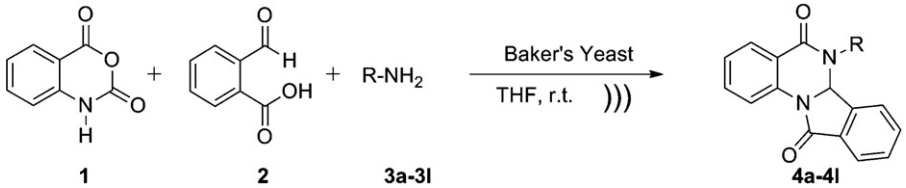
It is clear from Table 1 that the reaction performed without ultrasound afforded comparatively lower yields even after longer reaction time under ambient condition. When water and phosphate buffer were employed as the reaction medium, the starting materials were recovered (Table 1, entries 10 and 11). It might be due to insolubility of substrates in aqueous medium. In all cases, the reaction times are strikingly shorter and the yields of the products are higher under sonication. It was noted that the shortest reaction time and best yield were obtained in THF (84%, Table 1, entry 3) under sonic conditions. It is apparent that the ultrasound can accelerate the reaction significantly. Thus, THF was chosen as the solvent for this reaction under ultrasound irradiation.

3.2. Effect of different frequencies for ultrasound irradiation

In order to verify the effect of irradiation frequency, two experiments respectively at 30 and 40 kHz were performed. When the frequency was 30 kHz, the yield of **4a** was found to be 84% (Table 1, entry 3). The yield of **4a** (82%) (Table 1, entry 5) was found to be almost the same at 40 kHz. With increase of irradiation frequency from 30 to 40 kHz, the reaction yield was not affected to a considerable amount. Synthesis of **4a** at the frequency of 30 kHz at room temperature was found very effective and other amines were tried to synthesize a series of targeted compounds (**4b**–**4l**).

3.3. Synthesis of isoindolo[2,1-a]quinazoline derivatives 4 under ultrasound irradiation

After optimization of the conditions, to delineate this approach the methodology was evaluated by using different aromatic/aliphatic amines. A series of substituted isoindolo[2,1-a]quinazoline derivatives were synthesized using baker's yeast in THF at room temperature under sonic condition. Yeast was found to efficiently catalyze the reaction between isatoic anhydride, 2-carboxy benzaldehyde and a variety of amines affording moderate to good yields of the desired products within 2–5 h (Table 2).



Scheme 1. Synthetic pathway for the synthesis of isoindolo[2,1-a]quinazolines.

Table 1
Solvent selection for the synthesis of **4a** under ultrasound irradiation at room temperature.[a]

Entry	Solvent	Without ultrasound[b]		With ultrasound[c]	
		Time (h)	Yield (%)	Time (h)	Yield (%)
1	Methanol	24	33	4	40
2	Ethanol	24	33	4	35
3	**THF**	24	38	**2**	**84**
4[d]	THF[d]	40[d]	17[d]	2[d]	30[d]
5[e]	THF[e]	–	–	2[e]	82[e]
6	Acetonitrile	24	41	3.5	75
7	DCM	24	40	2	Trace
8	Dimethyl acetamide	24	36	2.5	50
9	DMF	24	31	2.5	30
10	Water	24	n.d.	4	n.d.
11	Phosphate buffer (pH 6.0)	24	n.d.	4	n.d.

n.d., not detected.
[a] Reaction conditions: isatoic anhydride **1** (1.22 mmol), 2-carboxy benzaldehyde **2** (1.31 mmol), benzyl amine **3a** (1.40 mmol) and solvent (5 mL), Baker's yeast (400 mg). Reaction temperature 30 °C.
[b] Reaction without ultrasound. Reaction temperature 30 °C.
[c] Reaction under ultrasonic irradiation at room temperature and the ultrasonic power 250 W and irradiation frequency 30 kHz.
[d] Without baker's yeast at 30 °C.
[e] Reaction under ultrasonic irradiation at room temperature and the ultrasonic power 250 W and irradiation frequency 40 kHz.

Both aliphatic and aromatic amines **3** underwent smooth reaction with isatoic anhydride **1** and 2-carboxy benzaldehyde **2** to give high yields of products. The varied nature of the substituents on the aromatic ring showed no obvious effect on this conversion. Both benzylamine and *p*-methoxy benzylamine were suitable substrates in this reaction, which resulted in the corresponding products 84% and 83% respectively. Cyclic amines (entries 4 and 12) also exhibited good yield. Aliphatic amines such as ethanolamine, methylamine, and ethylamine also afforded moderate yields of product (entries 3, 9 and 10). The structures of all the products **4** were established by IR, ^{1}H NMR, ^{13}C NMR, and MS spectroscopy. In ^{1}H NMR spectrum, singlet was observed around 6.3–7.05 δ and in ^{13}C NMR signal at 72.0–73.0 δ due to methine group adjacent to nitrogen atom. The appearance of two signals in ^{13}C NMR at 163–165 δ ppm clearly indicate the presence of two non equivalent C=O carbons. The IR spectrum showed strong band around 1650–1750 cm^{-1} due to C=O stretching.

3.4. Reaction mechanism

The cell of baker's yeast produces a variety of enzymes. It is known to provide specific enzymes to specific reactions [60]. Among these enzymes, lipase available in baker's yeast [61,62] might be thought responsible for accelerating the reaction. It is well known that lipase contains variety of different amino acid residues. The amino acid residues like aspartic acid, histidine and serine might be interacting with the substrate. —NH proton of histidine may be considered responsible [63] for enhancing electrophilic character of carbonyl carbon of isatoic anhydride and 2-carboxy benzaldehyde. Then decarboxylation occurs resulting in generation

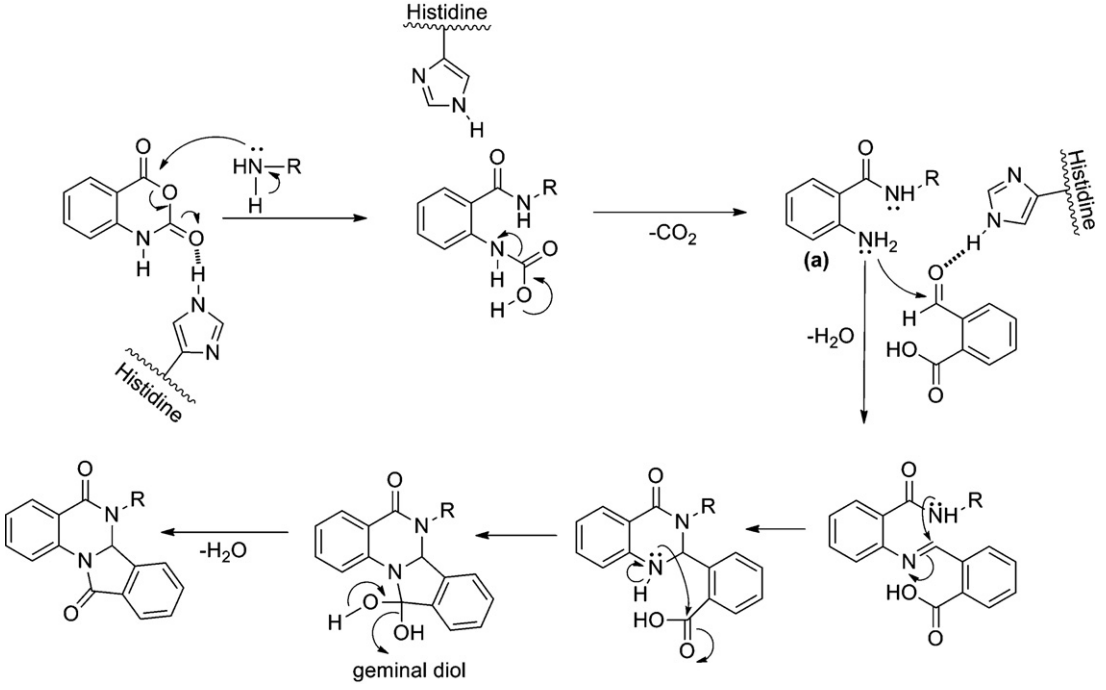


Fig. 1. Plausible mechanism for the formation of isoindolo[2,1-a]quinazoline.

Table 2
Synthesis of isoindolo[2,1-a]quinazolines using baker's yeast under sonic condition.[a]

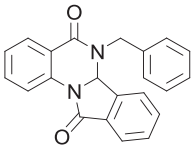
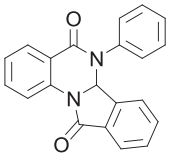
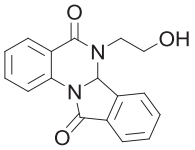
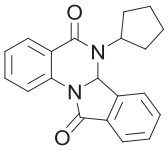
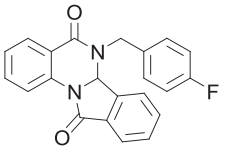
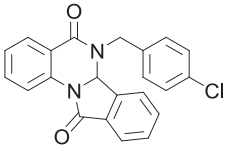
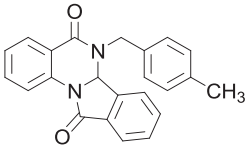
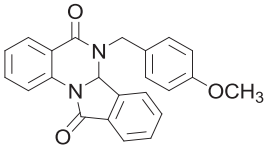
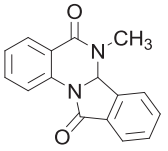
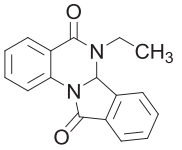
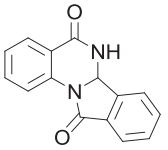
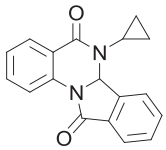
Entry	R	Product (4)		Time (HH:MM)	Yield (%)[b]
1	$C_6H_5CH_2$-		**4a**	02:00	84
2	C_6H_5-		**4b**	04:00	80
3	C_2H_5O-		**4c**	03:00	78
4	C_5H_9-		**4d**	03:45	81
5	4-F-$C_6H_4CH_2$-		**4e**	05:00	75
6	4-Cl-$C_6H_4CH_2$-		**4f**	05:00	78
7	4-CH_3-$C_6H_4CH_2$-		**4g**	04:30	65
8	4-OCH_3-$C_6H_4CH_2$-		**4h**	04:15	83
9	CH_3-		**4i**	02:45	76
10	CH_3CH_2-		**4j**	02:00	77
11	$(NH)_4CO_3$		**4k**	02:45	75

Table 2 (*Continued*)

Entry	R	Product (4)		Time (HH:MM)	Yield (%)[b]
12	C_3H_5-		**4l**	03:15	81

[a] Reaction conditions: isatoic anhydride **1** (1.22 mmol), 2-carboxy benzaldehyde **2** (1.31 mmol), amine **3** (1.40 mmol), THF (5 mL) and baker's yeast 400 mg.
[b] Isolated yield.

of 2-amino-*N*-substituted-benzamide (Fig. 1, **a**). The intermediate **(a)** reacts with 2-carboxy benzaldehyde generating imine intermediate, which is converted into the final product with loss of water molecule. The mechanism is schematically presented in Fig. 1.

To ascertain the path way, the model reaction was stopped after 1 h. The collected sample was purified by column chromatography. 2-Amino-N-benzylbenzamide **(a)** was successfully isolated which provided the support to the above proposed mechanism. This intermediate was characterized by ^{1}H NMR. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21–7.38 (m, 7H, Ar—H), 6.63–6.72 (m, 2H, Ar—H), 6.35 (s, 1H, —NH), 5.50 (s, 2H, —NH_2), 4.63 (d, J = 5.6 Hz, 2H, —CH_2).

3.5. Role of baker's yeast

To examine the catalytic role of baker's yeast, the control experiment was performed without yeast (Table 1, entry 4), using the same reaction protocol as described above. The reaction did not undergo completion due to partial conversion of intermediate to product **4a**. The absence of baker's yeast was found to strongly affect the reaction yield with and without US irradiation. Baker's yeast as well as ultrasound both are essential for shifting the reaction to the product side. In the presence of both, the reactions not only went to completion efficiently but also furnish the products in good to moderate yield.

3.6. Effect of ultrasound irradiation

To check ultrasound irradiation was effective or not, we performed all the experiments without ultrasound irradiation (Table 1) and we found that the reaction without ultrasound took very long time (24 h) and the yields were relatively low (30–40%). While the same transformations were successfully carried out under ultrasound irradiation in comparatively short duration (2–5 h) with moderate to good yield (65–84%). Therefore, in the present system, ultrasound was found to have beneficial effect on the synthesis of title compounds.

4. Conclusion

This is the first report to demonstrate the use of baker's yeast as the whole cell biocatalyst to accelerate the synthesis of isoindolo[2,1-a]quinazoline derivatives in organic medium. The role of ultrasonication in expedition of the biocatalyzed reaction is highlighted. This methodology may prove useful for other transformations also which has produced the targeted moiety in good to moderate yield at room temperature under ultrasonic irradiation.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molcatb.2013.01.024.

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