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One-pot Synthesis of New Category of 2-aryl-quinazolinones Using of DSDABCO as an Efficient Heterocyclic Medium

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ABSTRACT

A series of 2-aryl-quinazolin-4(1H)-ones have been synthesized in short reaction times and excellent yields through one-pot reaction between isatoic anhydride, glycine and various aldehydes in the presence of DSDABCO (1,4-disulfo-1,4-diazoniabicyclo[2.2.2] octane chloride) as an efficient heterocyclic ionic liquid media at room temperature. This method offers many advantages such as green environment, mild conditions, excellent efficiency, simple method and reduction of environmental consequences. Using recyclable ionic liquid and no need to another solvent or catalyst is an important step in green chemistry. On the other hand, this method does not require heating and is done at room temperature. The ionic liquid was recovered and reused. To the best of our knowledge, this is the first report for the synthesis of a new library of quinazolin-4(1H)-ones derived from glycine as a natural substrate based on green chemistry conditions. The structures of 2-aryl-quinazolin-4(1H)-ones were confirmed by ^{1}H, ^{13}CNMR, HRMS and FTIR spectral data and elemental analyses. Prog. Color Colorants Coat. 14 (2021), 233-240© Institute for Color Science and Technology.

1. Introduction

Dihydroquinazolinone derivatives are an important family of fused heterocyclic shows a wide range of biological, medicinal and pharmacological properties such as anti-tumor, anti-biotic, diuretic, analgesic, anti-hypertonic, anti-pyretic, anti-depressant, anti-histamine and vasodilation activities [1].

Many scientific papers have reported different methods for the synthesis of quinazolinones such as cyclization of acylaminobenzamides [2], amidation-oxidative ring closure of isatoic anhydride [3], preparation of 2-arylaminoquinazolinones in solid state condition [4], reduction of the azide functional group [5], preparation from imines and isatoic anhydrides [6] and heterocyclization of nitoarenes using of Pd-catalyst [7, 8]. In 2005, an atom-economy procedure for the one-pot synthesis of 2,3-dihydroquinazolin-4(*1H*)-ones from isatoic anhydride, aldehydes and amines were reported [9, 10]. So far only a few acid catalysts, e.g.; *p*-toluenesulfonic acid [8], silica sulfuric acid [11], Zinc(II) perfluorooctanoate [12], Gallium(III) triflate [13], ionic liquid [14, 15], $Al(H_2PO_4)_3$ [16], I_2 [17], montmorillonite K-10 [18], amberlyst-15 [19], Al/Al_2O_3 and Fe_3O_4 nanoparticles [20, 21], have been reported to accomplish this three component reaction.

Some of these methods have drawbacks such as toxic solvents and catalysts, long reaction time, the use of expensive catalysts and adverse yields [9-21]. According to the above points, it is essential to provide a simple, efficient, environmentally friendly method to synthesize

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quinazolinones without those defects.

We report herein synthesis of novel 2-aryl-quinazolin-4(*1H*)-ones with novel substrates (isatoic anhydride and glycine) and various aldehydes in ionic liquid 1,4-disulfo-1,4-diazoniabicyclo [2.2.2] octane chloride (DSDABCO) at room temperature.

2. Experimental

Chemicals were purchased from Merck and Fluka and used as purchased. Melting points were measured on an Electro-thermal 9100 apparatus and are uncorrected. FT-IR spectra were recorded on a Shimadzu FT-IR-8400S spectrometer. For the ultrasound reactions, ultrasound apparatus astra 3D (9.5 dm^3, 45 kHz frequency, input power with heating, 305W, number of transducers, 2) from TECNO-GAZ was used. ^{1}HNMR and ^{13}CNMR spectra were obtained on a Bruker DRX 500A vance spectrometer in DMSO-d_6 as solvent and with TMS as internal standard. Elemental analyses were recorded on a Carlo-Erba EA1110CNNO-S analyzer.

2.1. Procedure for the preparation of 1,4-disulfo-1,4-diazoniabicyclo[2.2.2]octane chloride (DSDABCO)

The method of preparing the DSDABCO ionic liquid was performed according to the method reported in our previous research work (Figure 1) [30]. The structures of 2-aryl-quinazolin-4(1*H*)-one compounds were characterized by comparison of their spectroscopic data (^{1}H, ^{13}CNMR and FTIR), elemental analyses.

2.2. General procedure for preparation of 2-aryl-quinazolin-4(1H)-ones using ionic liquid DSDABCO

A mixture of benzaldehydes (1 mmol), isatoic anhydride (1 mmol), Glycine (1 mmol) and DSDABCO (2 mL) were stirred at room temperature for the required reaction times (1-2 h). The progress of the reaction was monitored by TLC (EtOAc: petroleum ether 1:2). After completion of reaction, as indicated by TLC, the ionic liquid was separated by extraction with 2×15 mL of water. The solid residue was separated by recrystallization from EtOH. The pure products were collected in 86-97% yields.

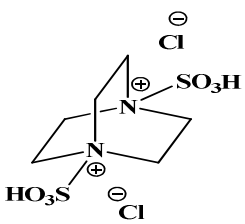


Figure 1: Structure of DSDABCO.

2-(1,2-dihydro-2-(4-nitrophenyl)-4-oxoquinazolin -3(4*H*)-yl)acetic acid (4a) 60 min, 96%, R_f 0.74, Yellow solid, mp 244-246 °C, FT-IR 3301 and 3197 (N-H), 3067 (aromatic C-H), 2912, 1651 (C=O), 1605, 1504 (aromatic C=C), 1531 (asymmetric NO_2), 1331 (symmetric NO_2). ^{1}HNMR δ: 3.59 (s, 2H), 5.75 (s, 1H), 6.68 (t, *J* = 7.2 Hz, 1H), 6.73 (d, *J* = 7.6 Hz, 1H), 7.14 (s, 1H), 7.24 (t, *J* = 6.8 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 2H), 7.58 (m, 2H), 8.34 (s, 1H, NH); ^{13}CNMR δ: 50.2, 66.2, 114.9, 115.3, 117.7, 122.0, 129.5, 131.6, 133.8, 141.2, 141.5, 148.0, 163.9 (C=O). Anal Calc. for $C_{16}H_{13}N_3O_5$: C, 58.72; H, 4.00; N, 12.84. Found: C, 58.70; H, 4.01; N, 12.87.

2-(1,2-dihydro-2-(3-nitrophenyl)-4-oxoquinazolin-3(4*H*)-yl)acetic acid (4b) 60 min, 94%, R_f 0.75, Yellow solid, mp 241-243 °C, FT-IR 3454 (N-H), 2921 (aromatic C-H), 2925 (aliphatic C-H), 1685 (C=O stretch), 1652 and 1620 (aromatic C-C), 1529 (NO_2, asymmetric stretch), 1346 (NO_2, symmetric stretch). ^{1}HNMR δ: 4.39 (s, 2H), 5.61 (s, 1H), 5.80 (s, 1H), 6.65-6.7 (m, 2H), 7.27-7.31 (td, *J* = 1.6, 7.6 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.91 (dd, *J* = 1.6, 8.0 Hz, 1H), 8.21 (dd, *J*= 1.2, 6.0 Hz, 1H), 8.39 (s, 1H). HRMS (m/z 327.09). Anal Calc. for $C_{16}H_{13}N_3O_5$: C, 58.72; H, 4.00; N, 12.84. Found: C, 58.75; H, 3.98; N, 12.82.

2-(1,2-dihydro-2-(2-nitrophenyl)-4-oxoquinazolin-3(4*H*)-yl)acetic acid (4c) 60 min, 90%, R_f 0.76, Yellow solid, mp 192-194 °C, FT-IR 3762 (N-H), 3434 (O-H), 1637 (C=O), 1527 and 1608 (C=C), 1413 (C-N), 1301 (C-O), 1527 (NO_2, asymmetric stretch), 1363 (NO_2, symmetric stretch). ^{1}HNMR δ: 6.65-6.69 (m, 3H), 7.01 (s, 1H), 7.17 (td, *J* = 1.6, 8.8 Hz,1H), 7.42 (d, *J* = 8.0 Hz,1H), 7.55-7.59 (td, *J* = 1.2, 8.8Hz, 1H), 7.65-7.71 (m, 2H), 8.06 (d, *J* = 8.4 Hz, 1H); ^{13}CNMR δ: 47.7, 66.6, 114.8, 115.3, 117.7, 125.7, 127.8, 128.0, 130.2, 133.5, 134.4, 135.8, 145.8, 148.09, 162.4, 170.8 ppm. Anal Calc. for $C_{16}H_{13}N_3O_5$: C, 58.72; H, 4.00; N, 12.84. Found: C, 58.71; H, 3.97; N, 12.86.

2-(2-(4-chlorophenyl)-1,2-dihydro-4-oxoquinazolin -3(4*H*)-yl)acetic acid (4d) 60 min, 94%, R_f 0.72, Yellow solid, mp 245-247 °C, FT-IR 3744 (N-H), 3304 (N-H), 3063 (C-H), 2933 (C-H), 1655 (C=O), 1610 and 1508 (C=C), 1385. ^{1}HNMR δ: 6.35 (s, 1H), 3.82 (s, 2H), 6.64 (d, *J* = 8.0 Hz, 1H), 6.79 (t, *J* = 7.5 Hz, 1H), 7.23–7.27 (m, 2H), 7.36 (d, *J* = 8.2 Hz, 2H), 7.52 (d, *J* = 8.2 Hz, 2H), 8.48 (s, 1H, NH); ^{13}CNMR δ: 50.1, 66.1, 114.9, 115.3, 117.7, 127.8, 128.7, 129.1, 129.2, 131.5, 133.8, 148.1, 163.9 (C=O). Anal. Calcd. For $C_{16}H_{13}ClN_2O_3$: C, 60.67; H, 4.14; N, 8.84; Found: C, 60.69; H, 4.16; N, 8.86.

2-(2-(3-chlorophenyl)-1,2-dihydro-4-oxoquinazolin -3(4*H*)-yl)acetic acid (4e) 60 min, 91%, R_f 0.68, Yellow solid, mp 182-184 °C, FT-IR 3492 (N-H), 3386 (O-H), 1664 (C=O), 1502 and 1629 (C=C), 1394 (C-N), 1242 (C-O). ^{1}HNMR δ: 4.21-4.28 (m, 2H), 4.48 (d, *J*= 5.6 Hz, 1H), 7.24 (t, *J* = 7.2 Hz,1H), 7.29-7.30 (m, 2H), 7.34-7.36 (d, *J* = 6.8 Hz, 1H), 7.38 (s, 1H), 7.60-7.63 (m, 2H), 7.65 (s, 1H), 7.68-7.70 (d, *J* = 7.6 Hz, 2H), 9.99 (s, 1H). HRMS (m/z 316.06). Anal. Calcd. For $C_{16}H_{13}ClN_2O_3$: C, 60.67; H, 4.14; N, 8.84; Found: C, 60.69; H, 4.12; N, 8.85.

2-(2-(2-chlorophenyl)-1,2-dihydro-4-oxoquinazolin -3(4*H*)-yl)acetic acid (4f) 60 min, 87%, R_f 0.72, Yellow solid, mp 185-187 °C, FT-IR 3483 (N-H), 3396 (O-H), 1689 and 1724 (C=O), 1550 and 1610 (C=C), 1382 (C-N), 1242 (C-O). ^{1}HNMR δ: 4.26 (dd, *J*= 6.8,14.0 Hz, 1H), 6.51 (t, *J* = 7.6 Hz, 1H), 6.62 (d, *J* = 8.0 Hz, 1H), 6.69-6.71 (m, 2H), 6.75 (d, *J* = 8.4 Hz, 1H), 7.25 (t, *J* = 6.8 Hz, 1H), 7.36-7.38 (m, 1H), 7.49-7.53 (m,1H), 7.62-7.67 (m,1H), 7.69 (d, *J* = 8 Hz, 1H), 10.34 (s, 1H); ^{13}CNMR δ: 60.2, 68.9, 127.7, 128.1, 129.0, 130.1, 131.0, 132.2, 134.2, 135.4, 136.2, 137.1, 146.8, 151.7, 163.3, 167.8 ppm. Anal. Calcd. For $C_{16}H_{13}ClN_2O_3$: C, 60.67; H, 4.14; N, 8.84; Found: C, 60.65; H, 4.16; N, 8.83.

2-(1,2-dihydro-2-(4-methoxyphenyl)-4-oxoquina-zolin -3(4*H*)-yl)acetic acid (4g) 90 min, 88%, R_f 0.67, Yellow solid, mp 243-245 °C, FT-IR 3406 (N-H), 3191 (N-H), 3072 (aromatic C-H), 1645 (C=O), 1514, 1458 (aromatic C=C), 746. ^{1}HNMR δ: 3.50 (s, 2H), 3.70 (s, 3H, OCH_3), 6.01 (s, 1H), 6.68 (d, *J* = 7.2 Hz, 1H), 6.79–6.83 (m, 2H), 6.86 (d, *J* = 8.0 Hz, 1H), 7.14 (t, *J* = 7.2 Hz, 1H), 7.23 (t, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 7.2 Hz, 1H), 7.62 (d, *J* = 7.2 Hz, 1H), 7.96 (s, 1H, NH); ^{13}CNMR δ: 50.1, 53.2 (OCH_3), 64.3, 115.6, 116.1, 118.6, 127.5, 127.9, 128.8, 130.4, 130.7, 138.5, 148.2, 166.0 (C=O). Anal. Calcd. For $C_{17}H_{16}N_2O_4$: C, 65.38; H, 5.16; N, 8.97; Found: C, 65.41; H, 5.18; N, 9.00.

2-(4-oxo-2-(*p*-tolyl)-1,2-dihydroquinazolin-3(*4H*)-yl) acetic acid (4h) 90 min, 89%, R_f 0.63, Yellow solid, mp 230-232 °C, FT-IR 3272 (N-H), 3236 (O-H), 1685 (C=O), 1550 and 1606 (C=C), 1458 (C-N), 1377 (C-O). ^{1}HNMR δ: 2.50 (s, 3H), 3.58 (s, 2H), 7.09-7.11 (d, *J* = 7.6 Hz, 2H), 7.20-7.23 (m, 3H), 7.48-7.52 (t, *J* = 7.2 Hz, 2H), 7.73-7.75 (d, *J* = 7.6 Hz, 2H), 8.54 (s, 1H), 10.36 (s, 1H). HRMS (m/z 296.12). Anal. Calcd. For $C_{17}H_{16}N_2O_3$: C, 68.91; H, 5.44; N, 9.45; Found: C, 68.88; H, 5.42; N, 9.47.

2-(2-(4-hydroxyphenyl)-4-oxo-1,2-dihydroquina-zolin -3(*4H*)-yl)acetic acid (4i) 120 min, 90%, R_f 0.71, Yellow solid, mp 212-214 °C, FT-IR FT-IR (KBr): (KBr): 3469 (N-H), 3369 (O-H), 1704 (C=O), 1560 and 1616 (C=C), 1386 (C-N), 1242 and 1163 (C-O). ^{1}HNMR δ: 4.22-4.27 (m, 1H), 6.08 (s, 1H), 7.23 (s, 1H), 7.32 (s, 1H), 7.40-7.42 (d, *J* = 7.2 Hz, 1H), 7.47-7.49 (d, *J* = 7.2 Hz, 2H), 7.68-7.70 (d, *J* = 8.0 Hz, 2H), 8.14 (s, 1H), 9.77 (s, 1H), 10.01 (s, 1H). HRMS (m/z 298.10). Anal. Calcd. For $C_{16}H_{14}N_2O_4$: C, 64.42; H, 4.73; N, 9.39; Found: C, 64.40; H, 4.72; N, 9.41.

2-(2-(2-hydroxyphenyl)-4-oxo-1,2-dihydroquina-zolin -3(*4H*)-yl)acetic acid (4j) 120 min, 89%, R_f 0.74, Yellow solid, mp 243-245 °C, FT-IR 3406 (N-H), 3191 (N-H), 3072 (aromatic C-H), 1645 (C=O), 1514, 1458 (aromatic C=C), 746. ^{1}HNMR δ: 3.50 (s, 2H), 3.70 (s, 3H, OCH_3), 6.01 (1H, s), 6.68 (1H, d, *J* = 7.2 Hz), 6.79–6.83 (2H, m), 6.86 (1H, d, *J* = 8.0 Hz), 7.14 (1H, t, *J*=7.2 Hz), 7.23 (1H, t, *J* = 7.2 Hz), 7.34 (1H, d, *J* = 7.2 Hz), 7.62 (1H, d, *J* = 7.2 Hz), 7.96 (1H, s, NH); ^{13}CNMR δ: 50.1, 53.2 (OCH_3), 64.3, 115.6, 116.1, 118.6, 127.5, 127.9, 128.8, 130.4, 130.7, 138.5, 148.2, 166.0 (C=O). HRMS (m/z 298.10%). Anal. Calcd. For $C_{17}H_{16}N_2O_4$: C, 65.38; H, 5.16; N, 8.97; Found: C, 65.40; H, 5.14; N, 8.95.

2-(1,2-dihydro-4-oxo-2-phenylquinazolin-3(4*H*)-yl) acetic acid (4k) 120 min, 92%, R_f 0.73, Yellow solid, mp 183-185 °C, FT-IR 3550 (N-H stretch), 3485 (O-H stretch), 1662 (C=O stretch), 1515 and 1606 (aromatic C=C stretch), 1402 (C-N stretch), 1348 (C-O stretch). ^{1}HNMR δ: 3.58-3.59 (d, *J* = 4.4 Hz, 2H), 6.46-6.50 (t,

J = 7.2 Hz, 1H), 6.67-6.72 (m, 1H), 7.10-7.26 (m, 1H), 7.38 (s, 1H), 7.48-7.50 (m, 3H), 7.54 (s, 1H), 7.56-7.59 (m, 1H), 7.63 (s, 1H), 7.67-7.69 (d, *J* = 7.6 Hz, 1H), 10.38 (s, 1H). HRMS (m/z 282.10). Anal. Calcd. For $C_{16}H_{14}N_2O_3$: C, 68.07; H, 5.00; N, 9.92; Found: C, 68.05; H, 4.98; N, 9.90.

3. Results and Discussion

Herein, in order to achieve a more efficient synthetic method, to deplete side effects, reduce reaction steps, increase efficiency and decrease reaction times, and also to continue our research on the synthesis of heterocyclic and pharmaceutical compounds [22-29], we wish to report the mild synthesis of some derivatives of 2-aryl-quinazolin-4(*1H*)-ones from isatoic anhydride, aldehydes and glycine using DSDABCO (Scheme 1).

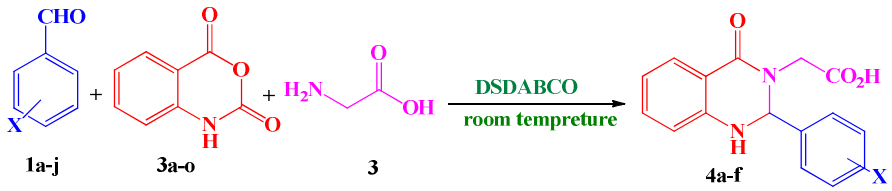
To try to discover and optimize different reaction conditions, we selected the one-pot three-component reaction of 4-nitrobenzaldehyde, isatoic anhydride and glycine as a model reaction. As a first step, we studied the model in the presence of various available catalysts such as $ZnCl_2$, silica gel and *L*-proline or some ionic liquids such as [BMIm][PF_6], [BMIm]Br, [BMIm]HSO_4, [BDBDMIm]Br, DSDABCO and some of previously reported catalyst [8, 16 ,18-20] for the synthesis of 2-(4-nitrophenyl)-2, 3-dihydroquinazolin-4-(*1H*)-one **4a**. As shown in Table 1, the study indicated that when the pattern reaction was performed using some of the existing catalysts, stirring or reflux, it lead to less efficiency and longer reaction time. Preliminary studies have shown that, ionic liquids have a good effect on efficiency and reaction time and are more suitable than other available catalysts, which mainly play the role of Lewis acid. On the other hand, in comparison with the studied ionic liquids, DSDABCO showed better results. While the same reaction took place in the DSDABCO media, to provide excellent performance of the product **4a** in short reaction times. It was observed that the reaction in the presence of 2 mL of DSDABCO gave the best result to synthesize quinazolinone **4a** with 96% isolated yield during 1 hour.

To present the generality and efficiency of the reaction, various benzaldehydes, isatoic anhydride and glycine were reacted in DSDABCO ionic liquid at room temperature (Scheme 1 and Table 2).

Table 1: Optimization of reaction condition for the synthesis of 2-aryl-quinazolin-4(1*H*)-one **4a.**

Entry	Catalyst [a] or Media [b]	Temperature	Time (h)	Yield (%)
1	silica gel	reflux	24	-
2	*p*-TSA[8]	reflux	8	72
3	$ZnCl_2$	reflux	24	59
4	$Al(H_2PO_4)_3$[16]	reflux	10	75
5	K10[18]	reflux	18	30
6	amberlyst-15[19]	reflux	8	73
7	nano-Fe_3O_4[20]	reflux	4	75
8	*L*-proline	reflux	12	52
9	[BMIm][PF_6]	60 °C	8	63
10	[BMIm]Br	60 °C	8	70
11	[BMIm]HSO_4	60 °C	10	70
12	[BDBDMIm]Br[26]	60 °C	5	71
13	[BDBDMIm]Br[26]	r.t.	5	75
14	DSDABCO	60 °C	1	84
15	DSDABCO	r.t.	1	96
16[c]	DSDABCO	r.t.	2	83
17[d]	DSDABCO	r.t.	1	96

[a] 15 mL of EtOH was used as a solvent; [b] 2 mL of ionic liquids were used; [c] 1 mL of ionic liquid was used; [d] 3 mL of ionic liquid was used.



4a: X = 4-NO_2 **4e:** X = 3-Cl **4i:** X = 4-OH
4b: X = 3-NO_2 **4f:** X = 2-Cl **4j:** X = 2-OH
4c: X = 2-NO_2 **4g:** X = 4-CH_3 **4k:** X = H
4d: X = 4-Cl **4h:** X = 4-OCH_3

Scheme 1: Multi-component synthesis of novel 2-aryl-quinazolin-4(1*H*)-ones.

Table 2: Synthesis of 2-aryl-quinazolin-4(1*H*)-ones using of ionic liquid DSDABCO.

Entry	product	X	Time (h)	Yield (%)[a]	Observed M.p (°C)
1	**4a**	4-NO_2	1	96	244-246
2	**4b**	3-NO_2	1	94	241-243
3	**4c**	2-NO_2	1	90	192-194
4	**4d**	4-Cl	1	94	245-247
5	**4e**	3-Cl	1	91	182-184
6	**4f**	2-Cl	1	87	185-187
7	**4g**	4-CH_3O	1.5	88	243-245
8	**4h**	4-CH_3	1.5	89	230-232
9	**4i**	4-OH	2	90	212-214
10	**4j**	2-OH	2	89	243-245
11	**4k**	H	2	92	183-185

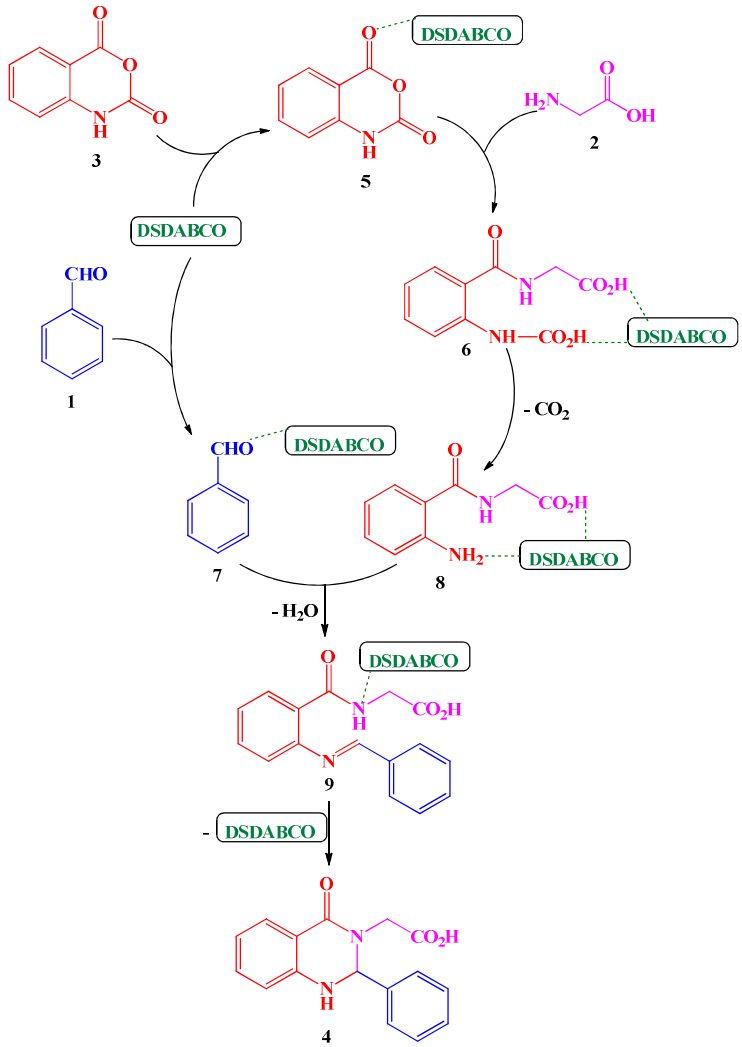
a. Isolated yield

Table 3: Recycling of ionic liquid DSDABCO.

Run of reusability	1	2	3	4	5	6
Yield (%)	96	95	96	95	95	84

In order to evaluate the reusability of DSDABCO, ionic liquid recycling was tested. The catalyst can be recycled up to six times without significant decrease in efficiency (Table 3).

In the proposed mechanism for this reaction, the carbonyl ester group is activated by DSDABCO as a protic acid or ionic liquid. Then, nuleophilic attack of amino group of glycine lead to by the intermediate **6**. The intermediate **8** resulting from the departure of carbon dioxide reacts with active aldehyde **7** to produce the intermediate **9**. Finally, with an intramolecular nuleophilic reaction, product **4** was observed (Scheme 2).



Scheme 2: Proposed mechanism for synthesis of 2-aryl-quinazolin-4(1*H*)-ones.

4. Conclusions

In conclusion, we have developed a simple, green and efficient protocol for the synthesis of 2-aryl-quinazolin-4(1*H*)-ones using DSDABCO as an efficient and new procedure. Simplicity, easy practice, integrated with inexpensive, environmentally friendly and reusable ionic liquid is notable attributes of this new method. To the best of our knowledge, this is the first report for the synthesis of a new library of quinazolin-4(1*H*)-ones derived from glycine as a natural substrate based on green chemistry conditions.

Acknowledgements

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