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Glycerol based ionic liquid with a boron core: A new highly efficient and reusable promoting medium for the synthesis of quinazolinones

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ABSTRACT

A highly efficient and environmental benign procedure for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones via the condensation of carbonyl compounds with 2-aminobenzamide using a glycerol based ionic liquid with a boron core as a new and reusable promoting medium is described. A broad range of substrates including aldehydes and ketone were condensed with 2-aminobenzamide. All reactions are completed in short times and the products are obtained in good to excellent yields. The reaction medium could be recycled and reused several times without any loss of efficiency. Moreover, presented procedure has been applied successfully for the synthesis of some novel bis(pyrazolinone) derivatives.

1. Introduction

One of the most important classes of nitrogen heterocycles is quinazoline and its derivatives that have recently been evaluated as antagonists of various biological receptors, such as 5-HT5A related diseases [1], calcitonin generelated peptide [2], and vasopressin V3 receptors [3]. Between various kinds of quinazoline derivatives, 2-substituted quinazolines show important biological activities such as anti-inflammatory [4], antihypertensive [5], anticancer [6], antitumor [7], and antibacterial [8]. Methaqualone (Fig. 1) is an anti-malarial drug [9a] and currently being used for the assessment of the abuse liability of sedative hypnotic drugs [9b]. Tiodazosin (Fig. 1), a hybrid of quinazoline and [1,3,4]-oxadiazole heterocycles has been marketed as antihypertensive agent [9c,d].

Based on the above facts, the synthesis of quinazoline derivatives is currently of great interest in organic synthesis. The most popular method for the synthesis of 2-sustituted quinazolines is based on the condensation of 2-aminobenzamide with aromatic aldehydes catalyzed by NH_4Cl, $AlCl_3/ZnCl_2$, *p*-TSA, other Lewis acids, and asymmetric Brönsted acids [10] or one-pot condensation of isatoic anhydride and amines with aldehydes in organic solvents [11].

Moreover, reductive condensation of 2-nitrobenzamide and aldehydes or ketones was also reported in the presence of low valence titanium [12].

Most of these methods have certain limitations such as tedious processes, long reaction times, harsh reaction conditions, application of volatile organic solvents, toxic reagents, non-reusability of the catalyst and low yields of products.

Nowadays, more severe legislation and restrictions are ordained in order to reduce of the environmental impact of man-made chemicals. In this context, the ability of ionic liquids (ILs) as new reaction media, to provide the requirements of environmental sustainability is remarkable. The most important advantage of ILs is their lack of vapor pressure relative to the traditional volatile molecular solvents, that corroborate their efficiency about the incorporation in a sustainable synthesis [17] (Green Chemistry principle 2) [13]. Moreover, their low toxicity and limited environmental persistence (Green Chemistry principles 3 and 10) [13] make them competent solvents for sustainable chemical processes. Another feature of ionic liquids is their ability to be reused many times. Over the last few years, there have been several reviews published in which ionic liquids occupied a central theme [14].

Glycerol, an organic liquid molecule that forms from the alkaline hydrolysis of fats has gained special attention because of its unique chemical and physical properties. Nowadays, this material is obtained as a by-product of biodiesel synthesis via the transesterification of seed oils with methanol. Glycerol is a green and biodegradable compound. Moreover, this material is non-volatile under normal atmospheric pressure and has a high boiling point. Besides, it is a nontoxic

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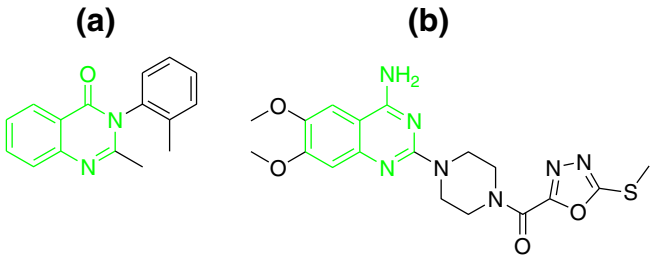


Fig. 1. Chemical structures of (a) Methaqualone and (b) Tiadazosin.

(LD_{50} (oral rat) = 12,600 mg kg^{-1}) material that is used widely in cosmetics such as face masks, skin creams, tooth paste etc.

In addition, boron is present in many foods and drinking water supplies. Estimated human consumption of boron in the U.S. diet ranges from 0.02 mg boron/day to more than 9 mg boron/day with an estimated average intake of 1.17 mg boron/day for men and 0.96 mg boron/day for women. Recent evidence has suggested that boron may be an essential micronutrient. The US EPA considers boric acid to be low in acute toxicity based on studies in rats with an oral LD_{50} of 3450 mg kg^{-1} for male rats and 4080 mg kg^{-1} for female rats.

Considering the biological importance and pharmaceutical applications of quinazolines and green behaviors of glycerol and importance of its derivatives in the development of more environmental benign organic procedures, and along with our previous studies on the application of glycerol and its derivatives in organic synthesis [15], herein we report the application of a glycerol based ionic liquid with a boron core as a highly efficient biodegradable and reusable promoting medium for the synthesis of quinazolinone derivatives (Scheme 1).

2. Method and material

Reagents and solvents were purchased from Merck, Fluka or Aldrich. The IL was prepared according to the reported method [16]. Melting points were determined in capillary tubes in an electro-thermal C14500 apparatus. The progress of the reaction and the purity of compounds were monitored by TLC analytical silica gel plates (Merck 60 F250). All known compounds were identified by comparison of their melting points and ^{1}H NMR data with those in the authentic samples. The ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) were run on a Bruker Avance DPX-250, FT-NMR spectrometer. Chemical shifts are given as a δ value against tetramethylsylane as the internal standard and *J* values are given in Hz. Microanalysis was performed on a Perkin-Elmer 240-B microanalyzer. All experiments performed in this work were repeated three times. The yield reported represents the average of the values obtained for each reaction.

3. Experimental

3.1. General procedure for the synthesis of H[Gly_2B]

Boric acid (61.83 g, 1 mol) and glycerol (190 g, 2 mol) were added to a 1 L flask containing toluene (500 mL) and the mixture was stirred at 110 °C for 4 h. During this time, the byproduct water was removed by azeotropic distillation. After this time, toluene was evaporated under reduced pressure and glyceroboric acid was obtained as a colorless viscous liquid [16].

3.2. General procedure for the synthesis of quinazolinones

2-Aminobenzamide (1 mmol) were added to the mixture of carbonyl compound (1 mmol) in H[Gly_2B] (0.5 g) in a 25 mL pyrex flask connected to a condenser and the resulted mixture was stirred magnetically for the appropriate time (Table 1) at 60 °C. The reactions were followed by thin layer chromatography (TLC) using hexane/ethyl acetate (3:1) as a mobile phase. After completion of the reaction, water (20 mL) was added and stirred magnetically for 5 min. Insoluble crude products were filtered and recrystallized from EtOH. To recover the H[Gly_2B], after the isolation of insoluble products, water was evaporated, and the remaining viscous liquid was washed with methyl *tert*-butyl ether (5 mL) and dried under reduced pressure. As H[Gly_2B] is too hydrophilic, in order for the complete removal of water, an additional lyophilization step was run. For this, recovered H[Gly_2B] was frozen in liquid nitrogen and lyophilized to near-dryness over 2 days [20]. (H[Gly_2B] was recovered in 98% yield).

3.3. Selected spectral data

3.3.1. 1′H,2H-spiro[acenaphthylene-1,2′-quinazoline]-2,4′(3′H)-dione (compound 3x)

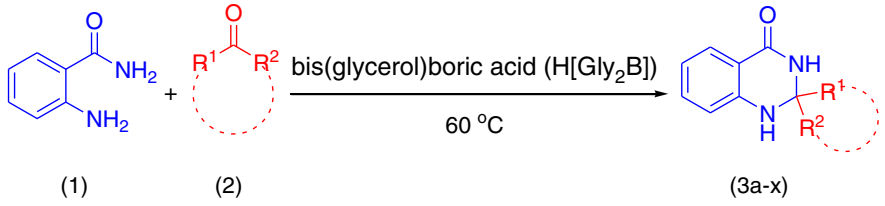
White powder (m.p. = 295–297 °C), υ_{max} (KBr):,3470, 3340, 3025, 2960, 1710 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 6.95–7.03 (m, 2H), 7.13 (d, *J* = 7.5 Hz, 1H), 7.47–7.51 (m, 3H), 7.71 (d, *J* = 7.3 Hz, 1H), 7.93 (d, *J* = 8.0 Hz, 2H), 8.09–8.18 (m, 2H), 9.5 (s, 1H), 10.8 (s, 1H). ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 104.0, 113.5, 115.8, 116.5, 120.9, 124.8, 125.9, 127.3, 128.8, 128.9, 129.3, 129.9, 131.8, 132.5, 133.1, 143.1, 147.5, 165.9, 198.8. Anal. Calcd for $C_{19}H_{12}N_2O_2$: C, 75.99; H, 4.03; N, 9.33%; found: C, 75.91; H, 4.09; N9.41%.

3.3.2. Bis(spiro-quinazoline) (compound 3y)

White powder, mp: > 300 °C, υ_{max} (KBr): 3450, 3390, 3020, 2975, 1700, 1650 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 1.09 (m, 4H), 1.47 (m, 4H), 6.8 (t, *J* = 7.5 Hz, 2H), 7.08 (d, *J* = 7.5 Hz, 2H), 7.51 (t, *J* = 7.5 Hz, 2H), 7.76 (d, *J* = 7.5 Hz, 2H), 9.8 (s, 2H), 10.7 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 21.8, 81.7, 112.5, 115.7, 116.8, 127.5, 132.9, 147.1, 166.6. Anal. Calcd for $C_{20}H_{20}N_4O_2$: C, 68.95; H, 5.79; N, 16.08%; found: C, 69.01; H, 5.83; N, 16.01%.

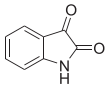
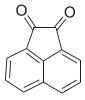
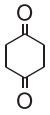
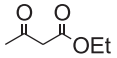
3.3.3. Ethyl 2-(2-methyl-4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)acetate (compound 11a)

White powder, m.p. = 227–229 °C, υ_{max} (KBr): 3420, 3395, 3017, 2950, 1705, 1675 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 0.97 (t, *J* = 7.8 Hz, 3H), 1.12 (s, 3H), 2.56 (d, *J* = 15.5 Hz, 1H), 2.68 (d, *J* = 15.5 Hz, 1H), 3.71 (m, 2H), 6.83 (t, *J* = 7.3 Hz, 1H), 7.03 (d, *J* = 7.3 Hz, 1H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.76 (d, *J* = 7.3 Hz, 1H), 9.15 (s, 1H), 10.31 (s, 1H). ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 14.6, 24.7, 45.5, 62.7, 73.4, 112.1, 116.3, 117.2, 128.5, 132.3, 147.3, 164.3, 171.9.



Scheme 1. Synthesis of quinazolinone derivatives using H[Gly_2B] as a highly efficient and reusable promoting medium.

Table 1
Synthesis of quinazolinone derivatives using H[Gly_2B] as a highly efficient, reusable glycerol based promoting medium.

Entry	R^1	R^2	Time (min)	Yield (%)[a]	M.P (°C)	
					Found	Reported
3a	C_6H_5	H	10	90	226–228	(225–226) [18]
3b	4-Cl-C_6H_4	H	10	91	205–207	(207–208) [18]
3c	4-Br-C_6H_4	H	10	91	198–199	(197–198) [18]
3d	3-Br-C_6H_4	H	15	90	227–228	(229–230) [18]
3e	4-F-C_6H_4	H	10	90	277–278	(279–280) [18]
3f	4-CH_3-C_6H_4	H	15	90	228–229	(229–231) [18]
3 g	4-OCH_3-C_6H_4	H	25	89	181–182	(183–184) [18]
3 h	2-OCH_3-C_6H_4	H	25	90	170–171	(173–174) [18]
3i	2-Cl-C_6H_4	H	15	91	207–209	(205–206) [19]
3j	4-NO_2-C_6H_4	H	5	92	>300	(310–313) [18]
3 k	3-NO_2- C_6H_4	H	5	91	182–184	(180–182) [18]
3 l	4-CN-C_6H_4	H	10	91	>300	(350–351) [18]
3 m	4-CF_3-C_6H_4	H	5	90	193–195	(194–196) [19]
3n	2-Furyl	H	15	89	165–166	(166–167) [11a]
3o	2-pyridyl	H	25	87	185–186	(187–188) [11e]
3p	$CH_3(CH_2)_3$	H	25	90	145–146	(143–144) [19]
3q	CH_3	CH_3	20	90	181–182	(183–184) [19]
3r	C_6H_5	CH_3	25	90	219–221	(222–224) [19]
3 s	$CH_3(CH_2)_2$	CH_3	20	91	191–193	(192–195) [19]
3 t	C_6H_5	C_6H_5	55	83	200–203	(203–205) [19]
3u	-$CH_2CH_2CH_2CH_2$-		30	89	260–261	258–259 [19]
3v	-$CH_2CH_2CH_2CH_2CH_2$-		30	90	219–221	216–218 [19]
3w			30	90		261–263 [21]
3x			30	91	295–297	–
3y[b]			50	87	>300	–
11a			180	85	227–229	–
11b[c]			180	71	220–221	–
11c[b]			360	78	>300	–

[a] Isolated yields.
[b] Reaction conditions: diketone (1 equivalent), 2-aminobenzamide (2 equivalents).
[c] Reaction conditions: diketone (1 equivalent), 2-aminobenzamide (1 equivalents).

Anal. Calcd for $C_{13}H_{16}N_2O_3$: C, 62.89; H, 6.50; N, 11.28%; Found: C, 63.95; H, 6.55; N, 11.38%.

3.3.4. 2-Methyl-2-(2-oxopropyl)-2,3-dihydroquinazolin-4(1H)-one (compound 11b)

White powder, m.p. = 220–221 °C, υ_{max} (KBr): 3425, 3395, 3020, 2980, 1710, 1685 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ (ppm) 1.25 (s, 3H), 2.46 (s, 3H), 2.76 (d, J = 16.0 Hz, 1H), 2.99 (d, J = 16.0 Hz, 1H), 6.87 (t, J = 7.5 Hz, 1H), 7.03 (d, J = 7.5 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.81 (d, J = 7.5 Hz, 1H), 9.31 (s, 1H), 10.04 (s, 1H). ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 23.5, 27.8, 57.9, 68.3, 112.8, 116.6, 117.9, 127.5, 133.1, 146.4, 196.02. Anal. Calcd for $C_{12}H_{14}N_2O_2$: C, 66.04; H, 6.47; N, 12.84%; Found: C, 66.11; H, 6.58; N, 12.75%.

3.3.5. 2,2′-Methylenebis(2-methyl-2,3-dihydroquinazolin-4(1H)-one) (compound 11c)

White powder, m.p. = >300 °C, υ_{max} (KBr): 3440, 3410, 3010, 2950, 1710, 1670 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6): δ (ppm) 1.89 (s, 6H), 2.08 (s, 2H), 6.87 (t, J = 7.6 Hz, 2H), 7.08 (d, J = 7.5 Hz, 2H), 7.55 (t, J = 7.7 Hz, 2H), 7.89 (d, J = 7.6 Hz, 2H), 9.16 (s, 2H), 10.23 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 24.8, 61.3, 67.8, 112.6, 116.5, 117.7, 128.3, 132.9, 146.1, 165.9. Anal. Calcd for $C_{19}H_{20}N_4O_2$: C, 67.84; H, 5.99; N, 16.66%; Found: C, 67.95; H, 6.07; N, 16.72%.

4. Results and discussion

In the first step, bis(glycerol)boric acid, H[Gly_2B], was synthesized using the synthetic route described in a recent patent [16]. For this purpose, glycerol was added to the appropriate quantity of boric acid in toluene, and the reaction was allowed to proceed at reflux temperature. The byproduct water was continuously removed from the reaction by azeotropic distillation, obtaining a highly viscous uncolored liquid. The structure of the resulted ionic liquid has been demonstrated with microanalysis, electrospray ionization mass spectroscopy (ESI-MS) and IR and its thermal stability is investigated by TG analysis. The mass spectrum of H[Gly_2B] dissolved in acetonitrile contains a strong peak at *m/e* 191 attributable to a bis(glycerol)borate, which is shown to be in good agreement with the results obtained from TG analysis and conventional elemental analysis. Carbon content of the H[Gly_2B] was 29.41% by conventional elemental analysis that is in appropriate agreement with a 1:2 adduct of glycerol and boric acid. The thermal stability of H[Gly_2B] was also investigated using a self-made TG analysis instrument, and the results are shown in Fig. 2. Fig. 2 shows the thermogram of the sample at an air flow of 3 mL min^{-1} and temperature ramp of 2 °C min^{-1}. Following the thermogram, the decrease observed in the slope of the diagram, starting at around 260 °C can be related to the loss of the covalently bound organic groups.

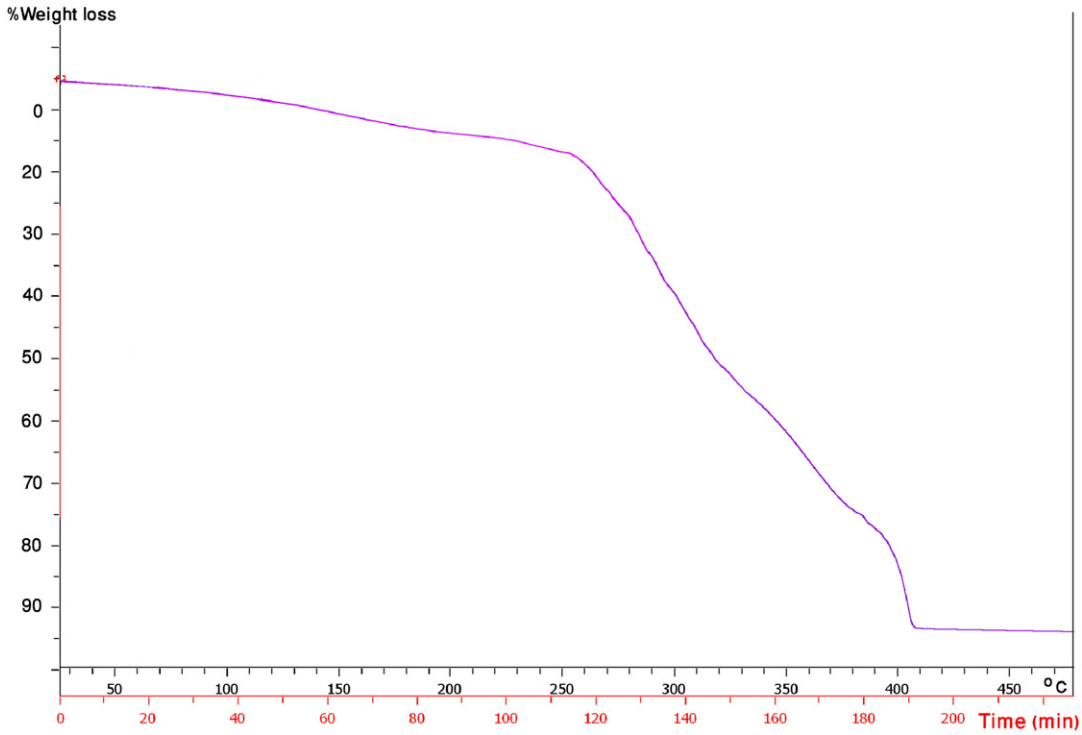
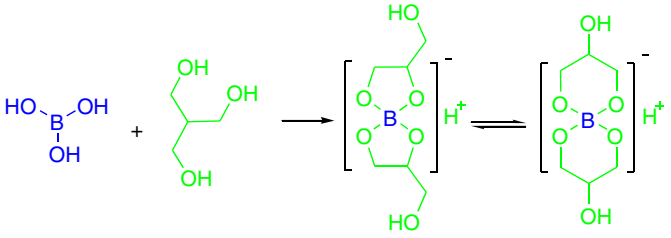


Fig. 2. TG analysis of H[Gly_2B].

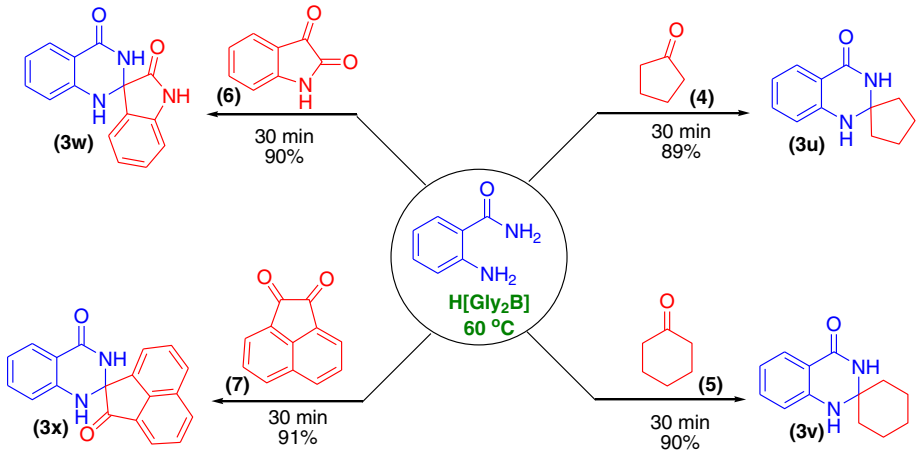


Scheme 2. Two possible structures of H[Gly_2B] (for more information see Ref. [17]).

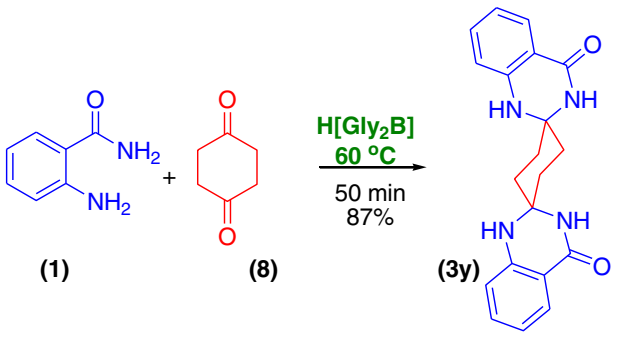
As it is shown in Scheme 2 there are two possible coordination forms between the glycerol and boric acid. Recently Chiappe et al. investigated the structure of H[Gly_2B] based on its ^{11}B and ^{13}C NMR spectra [17]. The ^{11}B NMR spectrum of H[Gly_2B] in dried CD_3CN contains two peaks in ca. 1:4 ratio at 28.05 and 23.8 ppm respectively. Based on their illustrations, we suggest that H[Gly_2B] is presented in two forms that are in equilibrium (Scheme 2).

In the next step, to evaluate the efficiency of H[Gly_2B] for the synthesis of quinazolinone derivatives, various kinds of carbonyl compounds were condensed with 2-aminobenzamide in the presence of H[Gly_2B] as a promoting medium (Scheme 1). For this purpose, 2-aminobenzamide (1 mmol) were added to the mixture of carbonyl compound (1 mmol) and H[Gly_2B] (0.5 g) and the resulting mixture was stirred at 60 °C. The obtained results are summarized in Table 1.

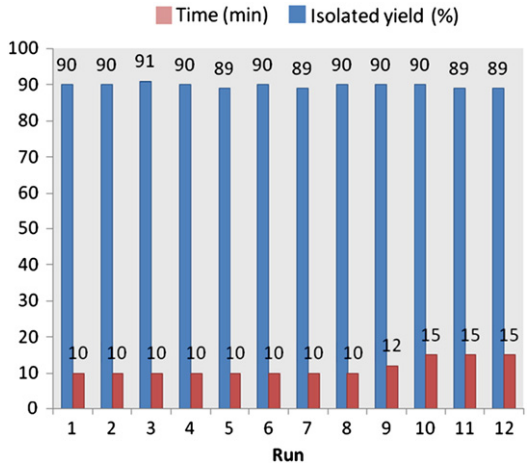
As it can be seen from Table 1, all reactions proceeded efficiently and the desired products were produced in good to excellent yields in relatively short reaction times without formation of any side products. Aromatic aldehydes having electron withdrawing groups (Table 1, entries 3j, 3k, 3l and 3m) reacted at faster rate compared with those that were substituted with electron releasing groups (Table 1, entries 3f,



Scheme 3. The synthesis of spiro-quinazolinones using H[Gly_2B] as a highly efficient promoting medium.



Scheme 4. The condensation of 2-aminobenzamide (1) and cyclohexane-1,4-dione (8) in the presence of H[Gly_2B] as a highly efficient glycerol based promoting medium.



Graph 1. Reusability of H[Gly_2B] in the synthesis of compound (3a).

3g and 3h). Besides, our methodology has been used successfully for heteroaromatic aldehydes, and corresponding product was obtained in excellent yields and without any byproduct (Table 1, entries 3n and 3o). As it is clear from the obtained results, presented methodology can be use in order of aliphatic and aromatic aldehydes as well as ketones.

We also applied our method for the synthesis of some spiro-quinozalinones via the condensation of cyclic ketones such as cyclopentanone (4), cyclohexanone (5), isatin (6) and acenaphthenequinone (7) with 2-aminobenzamide (1) in the presence of H[Gly_2B] at 60 °C (Scheme 3).

Moreover bis(spiro-quinazolinone) (3y) were synthesized via the condensation reaction of cyclohexane-1,4-dione (8) and 2-aminobenzamide (1) in the presence of H[Gly_2B] for the first time (Scheme 4).

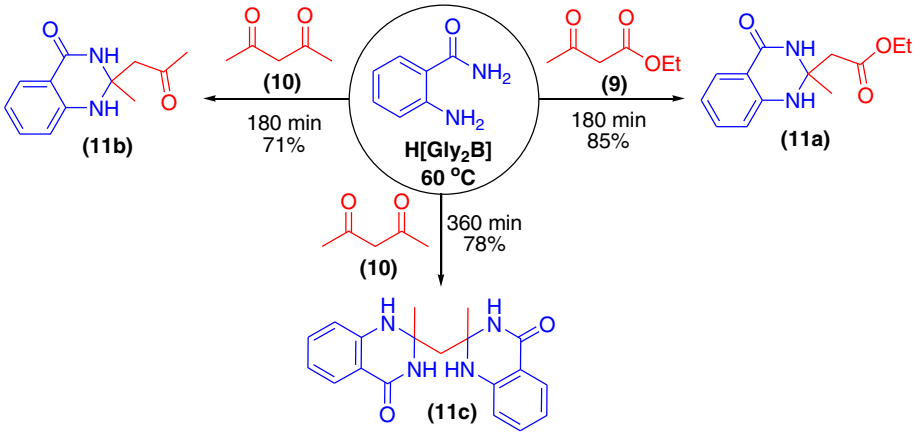
Besides, we examined our methodology for the condensation of 2-aminobenzamide with 1,3-dicarbonyl compounds such as ethylacetoacetate (9) and acetylacetone (10) and the obtained results are shown in Scheme 5.

As it is shown in Scheme 5, the condensation reaction between 2-aminobenzamide and enolizable 1,3-dicarbonyl compounds is more difficult and relatively lower yields were obtained in these cases. Only product (11b) and trace amounts of (11c) were obtained when acetylacetone (10) was treated with 1 equivalent of 2-aminobenzamide, whereas product 11c was the major product when 2 equivalents of 2-aminobenzamide were treated with acetylacetone (10) in the presence of H[Gly_2B] at 60 °C.

The possibility of recycling the reaction medium was examined for the synthesis of compound (3a) using the reaction between benzaldehyde (1 mmol) and 2-aminobenzamide (1) (1 mmol) in H[Gly_2B] (0.5 g) at 60 °C. After completion of the reaction, water (20 mL) was added and stirred magnetically for 5 min. Insoluble crude products were filtered and recrystallized from EtOH. To recover the H[Gly_2B], after the isolation of insoluble products, water was evaporated, and the remaining viscous liquid was washed with methyl *tert*-butyl ether (5 mL) and dried under reduced pressure. As H[Gly_2B] is too hydrophilic, in order for the complete removal of water, an additional lyophilization step was run. For this, recovered H[Gly_2B] was frozen in liquid nitrogen and lyophilized to near-dryness over 2 days.[20] The recovered H[Gly_2B] was reused twelve times for the synthesis of compound (3a) and no loss of efficiency was observed (Graph 1).

5. Conclusion

In summary, we have reported a highly efficient and green method for the synthesis of quinazolinone derivatives using H[Gly_2B] as a highly efficient and reusable glycerol based reaction medium. The method presented, avoids the use of hazardous catalysts or solvents. The promising points for the presented methodology are efficiency, generality, high yield, short reaction time, cleaner reaction profile, ease of product isolation, simplicity, potential for recycling of the reaction medium, and finally agreement with the green chemistry protocols, which all make it a useful and attractive process for the synthesis of quinazolinone derivatives. Moreover, the presented method is applicable for the synthesis of more complex structures such as spiro-quinazolinones and bis(spiro-quinazolinone)s.



Scheme 5. The condensation of 2-aminobenzamide with 1,3-dicarbonyl compounds in the presence of H[Gly_2B] as a highly efficient promoting medium.

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