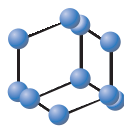


RESEARCH ARTICLE

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SCIENCE

New Crown Ether-Based Ionic Liquids as a Green and Versatile Organocatalyst for Three-Component Synthesis of 1,5-Dihydropyrano[2,3-c]chromene Derivatives



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Abstract: A simple and green method for the synthesis of 1,5-dihydropyrano[2,3-c]chromene derivatives has been reported by three-component reaction of aromatic aldehydes, malononitrile, and 3-hydroxycoumarin in the presence of a series of novel crown ether-based ionic liquids (CE-based ILs) in H₂O/EtOH (1:1), under the reflux conditions. The novel CE-based ILs have been synthesized by 18-crown-6 or dibenzo 18-crown-6 chelated with sodium benzenesulfinate derivatives and used as a green and environmental organocatalyst. This method has some advantages such as the aqueous reaction medium, stable catalysts, cleaner reaction profiles and high yield of products in short reaction time.

Keywords: Dihydropyrano[2,3-c]chromene, ionic liquids, sodium benzenesulfonates, crown ethers, three component reactions, aromatic aldehydes.

1. INTRODUCTION

An ionic liquid is defined as a salt with a melting point typically <100°C, although there is a more general definition of ionic liquids, which have a low melting point. There are many alternatives which have been used as ionic liquids. The terms room temperature ionic liquid, non-aqueous ionic liquid, molten salt, liquid organic salt, and fused salt have all been used to describe these salts in the liquid phase. The unique properties of ILs, such as polarity, high stability and density, conductivity, negligible vapour pressure, hydrophilicity/hydrophobicity, and high viscosity distinguish them from traditional organic solvents [1, 2]. ILs have been used as solvents or co-catalysts in many organic reactions, such as organic catalysis [3], three-component reactions [4, 5], Diles-Alder reactions [6], Michael addition [7], Friedel-Crafts acylation and alkylation reactions [8, 9], and Knoevenagel condensation [10].

Multi-component reactions (MCRs) represent an attractive synthetic strategy for generating molecular complexity in a single synthetic operation since they are performed without a need to isolate any intermediate during their processes. This reduces time and saves both energy and raw materials [11-13]. In fact, the development of MCRs can lead to a new diverse synthetic approach to afford many small organic

molecules in the field of new organic, bioorganic, and medicinal chemistry [14, 15].

Hence, MCRs are now considered as a vital technique in the synthesis of many important heterocyclic compounds such as pyrano[3,2-c]chromene derivatives.

Chromene and its derivatives have not only attracted considerable interest due to their biological activity, but have also found wide application in medicinal chemistry as well [16]. For example, these compounds have antihyperglycemic and antidyslipidemic [17], cytotoxic [18], anti-inflammatory [19], antifungal [20], anticancer [21] and antimalarial activities [4]. Chromene and its derivatives are also widely employed as cosmetics, pigments [5], and potential biodegradable agrochemicals [7]. In addition, chromenes are found in plants [22] and used as valuable leads for the design and synthesis of new pharmacophores for medicinal chemistry and drug development. Therefore, we can proceed with synthesizing 1,5-dihydropyrano[3,2-c]chromene derivatives using the method mentioned below.

In this report, we synthesized a series of novel crown ether-based ionic liquids (CE-based ILs) by chelating 18-crown-6 or dibenzo 18-crown-6 with sodium benzenesulfinate derivatives for the first time. They are then used as green and environmental organocatalysts for the synthesis of 1,5-dihydropyrano[2,3-c]chromene derivatives by three-component reaction of aromatic aldehydes, 3-hydroxycoumarin and malononitrile in H₂O/EtOH (1:1), under reflux conditions.

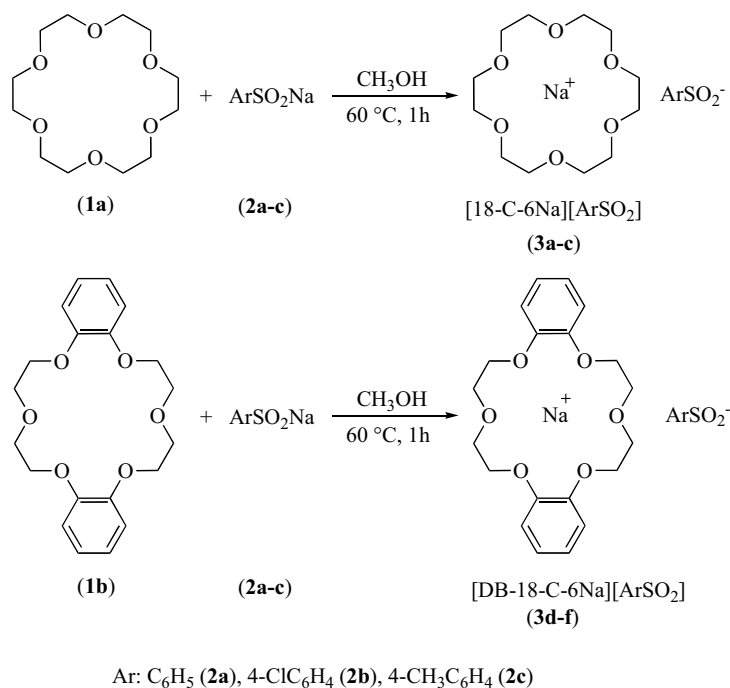
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2. RESULTS AND DISCUSSION

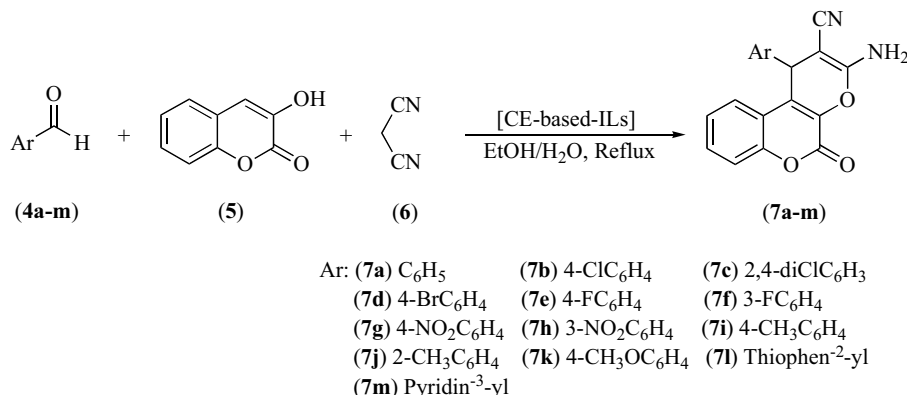
In the beginning of our research, we synthesized 1,5-dihydropyrano[3,2-*c*]chromene derivatives (7a-m) in two steps, (i) a series of novel crown ether-based ionic liquids (CE-based ILs) were synthesized in 100% yield, by treating an equimolar ratio of sodium benzenesulfinate derivatives (2a-c) with crown ethers (18-C-6 (1a) or dibenzo 18-C-6 (1b)) in methanol as solvent at 60°C for 1h (Scheme 1). Then, CE-based ILs were characterized with ¹H NMR, ¹³C NMR and elemental analyses techniques. (ii) After formation of the expected CE-based ILs (3a-f) and in connection with our previous researches on environmentally friendly multi-component reactions [23-28], we used CE-based ILs as green and environmentally organocatalysts, for the synthesis of 1,5-dihydropyrano[2,3-*c*]chromene (7a-m) derivatives by three-component reaction of aromatic aldehydes (4a-m), 3-hydroxycoumarin (5) and malononitrile (6) in H₂O/EtOH (1:1), under reflux conditions (Scheme 2). The mixture of

EtOH and H₂O in a 1:1 ratio was not found only to be more effective than other pure solvents such as EtOH, H₂O, CH₃OH, or CH₃CN but also showed more efficiency than other ratios of mixed EtOH and H₂O [10,29]. Afterwards, to optimize the reaction conditions, a methodological study considering different variables affecting the reaction time and yield, was carried out on the three-component reaction of benzaldehyde (4a), 3-hydroxycoumarin (5) and malononitrile (6) in the presence of CE-based ILs as catalysts for preparing a compound of 3-amino-5-oxo-1-phenyl-1,5-dihydropyrano[2,3-*c*]chromene-2-carbonitrile (7a). The results are shown in Table 1.

When this three-component reaction was performed in the presence of [18-C-6Na][4-ClC₆H₄SO₂], a compound (7a) was obtained in 93% yield within 25 min. We also optimized the quantity of the catalysts. At first, the reaction was carried out in a catalyst-free condition. It was observed that no considerable product was obtained. Also the reaction time increased



Scheme (1). Synthesis of crown ether-based ionic liquids (CE-based ILs).



Scheme (2). Three-component reaction of aromatic aldehydes (4a-m), 3-hydroxycoumarin (5) and malononitrile (6) in the presence of catalytic amounts of crown ether-based ionic liquids (CE-based ILs).

Table 1. Optimization of reaction condition.

Entry	Catalyst	Catalyst (mol%)	Time (min)	Yield (%)
1	[18-C-6Na][C ₆ H ₅ SO ₂]	40	35	91
2	[18-C-6Na][4-ClC ₆ H ₄ SO ₂]	40	25	93
3	[18-C-6Na][4-CH ₃ C ₆ H ₄ SO ₂]	40	40	90
4	[DB-18-C-6Na][C ₆ H ₅ SO ₂]	40	35	85
5	[DB-18-C-6Na][4-ClC ₆ H ₄ SO ₂]	40	30	88
6	[DB-18-C-6Na][4-CH ₃ C ₆ H ₄ SO ₂]	40	40	80
7	[18-C-6Na][4-ClC ₆ H ₄ SO ₂]	20	35	88
8	[18-C-6Na][4-ClC ₆ H ₄ SO ₂]	30	30	90
9	[18-C-6Na][4-ClC ₆ H ₄ SO ₂]	50	25	93
10	[18-C-6Na][4-ClC ₆ H ₄ SO ₂]	-	300	trace

to several hours (entry 10). An increase in the amount of catalyst from 20 to 50 mol% improved the yield of the compound 7a. The best results were obtained when the reactions were carried out in the presence of 40 mol% [18-C-6Na][4-ClC₆H₄SO₂] (Table 1, entries 7-9).

On the basis of our collected data, we decided to apply this method for the synthesis of 1,5-dihydropyrano[2,3-*c*]chromene derivatives (7a-m) by three-component reaction of aromatic aldehydes (4a-m), 3-hydroxycoumarin (5), and malononitrile (6) in H₂O/EtOH (1:1), in the presence of [18-C-6Na][4-ClC₆H₄SO₂] (40 mol%) under reflux condition. The results are summarized in Table 2.

3. EXPERIMENTAL

Solvents and reagents were purchased from commercial sources and were used without further purification. Melting points were measured on an Electrothermal-9100 apparatus and were uncorrected. IR spectra were recorded on a Bruker FTIR Tensor 27 infrared spectrophotometer. ¹H NMR spectra were recorded on a Bruker Avance III 300 MHz spectrometer. ¹³C NMR spectra were recorded on the same instruments at 75 MHz, using TMS as an internal standard. Elemental analyses were performed using a Heraeus CHN-O Rapid analyzer.

3.1. General Procedure for the Synthesis of Crown Ether-based ILs (3a-f)

A mixture of 18-crown-6 or dibenzo 18-crown-6 (**1a**, **1b**) (2 mmole) and sodium benzenesulfinate derivatives (**2a-c**) (2 mmole) in methanol (15 mL) was stirred at 60°C for 1h. Then, the solvent was evaporated under reduced pressure. The residue was vacuum dried to generate the desired CE-based ILs in 100% yield.

3.1.1. [18-C-6Na][C₆H₅SO₂] (**3a**)

Liquid; ¹H NMR (300 MHz, D₂O) δ_{ppm}: 7.54 (d, 2H, *J* = 6 Hz, CH-Ar), 7.45-7.39 (m, 3H, CH-Ar), 3.52 (s, 24H, OCH₂); ¹³C NMR (75 MHz, D₂O): δ_{ppm}: 153.5, 130.4, 129.0, 123.4, 69.3 (OCH₂). Anal. calcd. for C₁₈H₂₉NaO₈S: C, 50.46; H, 6.82; S, 7.48%. Found: C, 50.29; H, 6.65; S, 7.32%.

3.1.2. [18-C-6Na][4-ClC₆H₄SO₂] (**3b**)

White powder; mp 60-62°C; ¹H NMR (300 MHz, D₂O) δ_{ppm}: 7.49 (d, 2H, *J* = 6 Hz, CH-Ar), 7.42 (d, 2H, *J* = 6 Hz, CH-Ar), 3.54 (s, 24H, OCH₂); ¹³C NMR (75 MHz, D₂O): δ_{ppm}: 152.3, 135.5, 128.9, 125.2, 69.3 (OCH₂). Anal. calcd. for C₁₈H₂₈ClNaO₈S: C, 46.70; H, 6.10; S, 6.93%. Found: C, 46.53; H, 5.94; S, 6.76%.

3.1.3. [18-C-6Na][4-CH₃C₆H₄SO₂] (**3c**)

White powder; mp 36-37°C; ¹H NMR (300 MHz, D₂O) δ_{ppm}: 7.42 (d, 2H, *J* = 6 Hz, CH-Ar), 7.23 (d, 2H, *J* = 6 Hz, CH-Ar), 3.53 (s, 24H, OCH₂), 2.26 (s, 3H, CH₃); ¹³C NMR (75 MHz, D₂O): δ_{ppm}: 150.5, 140.9, 129.4, 123.4, 69.3 (OCH₂), 20.4 (CH₃). Anal. calcd. for C₁₉H₃₁NaO₈S: C, 51.57; H, 7.06; S, 7.25%. Found: C, 51.41; H, 6.89; S, 7.09%.

3.1.4. [Dibenzo 18-C-6Na][C₆H₅SO₂] (**3d**)

White powder; mp > 300°C; ¹H NMR (300 MHz, DMSO-*d*₆) δ_{ppm}: 7.54 (d, 2H, *J* = 6 Hz, CH-Ar), 7.35-7.22 (m, 3H, CH-Ar), 6.99-6.89 (m, 8H, CH-Ar), 4.08 (t, 8H, *J* = 3 Hz, OCH₂), 3.90 (t, 8H, *J* = 3 Hz, OCH₂); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{ppm}: 160.6, 147.7, 128.0, 127.9, 124.7, 121.1, 111.7, 68.9 (OCH₂), 67.3 (OCH₂). Anal. calcd. for C₂₆H₂₉NaO₈S: C, 59.53; H, 5.57; S, 6.11%. Found: C, 59.36; H, 5.41; S, 5.95%.

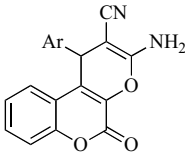
3.1.5. [Dibenzo 18-C-6Na][4-ClC₆H₄SO₂] (**3e**)

White powder; mp > 300°C; ¹H NMR (300 MHz, DMSO-*d*₆) δ_{ppm}: 7.52 (d, 2H, *J* = 6 Hz, CH-Ar), 7.36 (d, 2H, *J* = 6 Hz, CH-Ar), 6.99-6.89 (m, 8H, CH-Ar), 4.08 (t, 8H, *J* = 3 Hz, OCH₂), 3.90 (t, 8H, *J* = 3 Hz, OCH₂); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{ppm}: 159.8, 147.7, 132.4, 127.9, 126.8, 121.1, 111.8, 68.9 (OCH₂), 67.3 (OCH₂). Anal. calcd. for C₂₆H₂₈ClNaO₈S: C, 55.86; H, 5.05; S, 5.74%. Found: C, 55.69; H, 4.89; S, 5.59%.

3.1.6. [Dibenzo 18-C-6Na][4-CH₃C₆H₄SO₂] (**3f**)

White powder; mp > 300°C; ¹H NMR (300 MHz, DMSO-*d*₆) δ_{ppm}: 7.39 (d, 2H, *J* = 6 Hz, CH-Ar), 7.12 (d, 2H,

Table 2. Synthesis of 1,5-dihydropyrano[2,3-*c*]chromene derivatives.

 (7a-m)					
Compd. No.	Ar	Time (min)	Yield (%)	M.P. Observed (°C)	M.P. Reported (°C) [Ref.]
7a	C ₆ H ₅	25	93	246-247	248 [30]
7b	4-ClC ₆ H ₄	20	94	217	218 [31]
7c	2,4-diClC ₆ H ₃	18	94	289-290	-
7d	4-BrC ₆ H ₄	25	92	207-209	210 [30]
7e	4-FC ₆ H ₄	20	93	201-202	203 [30]
7f	3-FC ₆ H ₄	23	92	274-275	-
7g	4-NO ₂ C ₆ H ₄	18	94	255	256 [31]
7h	3-NO ₂ C ₆ H ₄	20	91	234-236	237 [30]
7i	4-CH ₃ C ₆ H ₄	25	90	229-230	231 [30]
7j	2-CH ₃ C ₆ H ₄	30	89	297-298	-
7k	4-CH ₃ OC ₆ H ₄	25	90	232-234	234 [30]
7l	Thiophen-2-yl	23	90	203-205	205 [30]
7m	Pyridin-3-yl	27	89	253-254	-

$J = 6$ Hz, CH-Ar), 6.99-6.89 (m, 8H, CH-Ar), 4.09 (t, 8H, $J = 3$ Hz, OCH₂), 3.90 (t, 8H, $J = 3$ Hz, OCH₂), 2.29 (s, 3H, CH₃); ¹³C NMR (75 MHz, DMSO-*d*₆): δ_{ppm} : 157.8, 147.7, 136.9, 128.5, 124.8, 121.1, 111.8, 68.9 (OCH₂), 67.3 (OCH₂), 21.3 (CH₃). Anal. calcd. for C₂₇H₃₁NaO₈S: C, 60.21; H, 5.80; S, 5.95%. Found: C, 60.04; H, 5.86; S, 5.79%.

3.2. General Procedure for the Preparation of 1,5-Dihydropyrano[2,3-*c*]chromene derivatives (7a-m)

A mixture of aromatic aldehydes (**4a-m**) (2 mmol), 3-hydroxycoumarin (**5**) (2 mmol) and malononitrile (**6**) (2 mmol), and [18-C-6Na][4-ClC₆H₄SO₂] (40 mol%) in H₂O/EtOH (1:1) (10 mL) was refluxed in the time reported in Table 2 (the progress of the reaction being has been monitored by TLC (silica gel, hexane/ethyl acetate 3:1 as an eluent)). The reaction mixture was cooled after completion of the reaction, and the crude product was filtered and dried. The collected NMR spectra for **7a**, **7d**, **7e**, **7h**, **7i**, **7k** **7l** [30]; **7b** and **7g** [31], were consistent with the previously reported NMR data for these components.

3.2.1. 3-Amino-1-(2,4-dichlorophenyl)-5-oxo-1,5-dihydropyrano[2,3-*c*]chromene-2-carbonitrile (7c)

Pale green powder; mp 289-290°C; IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 3447, 3308 (NH₂), 2196 (CN), 1739 (C=O), 1662, 1630, 1587 (C=C); ¹H NMR (300 MHz, DMSO-*d*₆) δ_{ppm} : 7.69-7.68 (d, 1H, $J = 3$ Hz, CH-Ar), 7.53-7.17 (m, 8H, CH-Ar, NH₂), 5.46 (s, 1H, CH); ¹³C NMR (75 MHz, DMSO-*d*₆) δ_{ppm} : 159.5 (C=O), 154.2 (C3), 150.5, 139.5, 135.4, 133.4, 132.8, 132.5,

130.8, 129.6, 129.1, 125.4, 124.9, 124.3, 119.3, 117.2, 117.1 (CN), 55.3 (C2), 35.0 (C1). Anal. calcd. For C₁₉H₁₀Cl₂N₂O₃: C, 59.24; H, 2.62; N, 7.27. Found: C, 59.06; H, 2.45; N, 7.09%.

3.2.2. 3-Amino-1-(3-fluorophenyl)-5-oxo-1,5-dihydropyrano[2,3-*c*]chromene-2-carbonitrile (7f)

White powder; mp 274-275°C; IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 3440, 3334 (NH₂), 2189 (CN), 1734 (C=O), 1660, 1629 (C=C); ¹H NMR (300 MHz, DMSO-*d*₆) δ_{ppm} : 7.51-7.05 (m, 10H, CH-Ar, NH₂), 5.18 (s, 1H, CH); ¹³C NMR (75 MHz, DMSO-*d*₆) δ_{ppm} : 164.4 (C-F), 161.2 (C=O), 159.4 (C3), 154.5, 150.6, 146.3, 146.2, 135.0, 131.4, 131.3, 130.7, 125.8, 125.4, 125.1, 124.1, 124.0, 119.9, 117.4, 116.8 (CN), 115.0, 114.9, 114.7, 114.6, 56.9 (C2), 37.5 (C1). Anal. calcd. For C₁₉H₁₁FN₂O₃: C, 68.26; H, 3.32; N, 8.38. Found: C, 68.09; H, 3.15; N, 8.21%.

3.2.3. 3-Amino-5-oxo-1-(*o*-tolyl)-1,5-dihydropyrano[2,3-*c*]chromene-2-carbonitrile (7j)

Pale yellow powder; mp 297-298°C; IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 3435, 3303 (NH₂), 2202 (CN), 1726 (C=O), 1665, 1633, 1595 (C=C); ¹H NMR (300 MHz, DMSO-*d*₆) δ_{ppm} : 7.50-7.39 (m, 2H, CH-Ar), 7.26-7.09 (m, 8H, CH-Ar, NH₂), 5.29 (s, 1H, CH), 2.64 (s, 3H, CH₃); ¹³C NMR (75 MHz, DMSO-*d*₆) δ_{ppm} : 159.2 (C=O), 154.4 (C3), 150.5, 141.7, 135.1, 134.8, 131.3, 130.6, 128.7, 127.7, 127.6, 127.0, 125.3, 125.0, 120.1, 117.4, 116.9 (CN), 56.6 (C2), 34.5 (C1), 19.6 (CH₃). Anal. calcd. For C₂₀H₁₄N₂O₃: C, 72.72; H, 4.27; N, 8.48. Found: C, 72.54; H, 4.10; N, 8.32%.

3.2.4. 3-Amino-5-oxo-1-(pyridin-3-yl)-1,5-dihydropyrano[2,3-*c*]chromene-2-carbonitrile (7m)

Cream powder; mp 253-254°C; IR (KBr, $\nu_{\max}/\text{cm}^{-1}$) 3452, 3340 (NH₂), 2195 (CN), 1744 (C=O), 1658, 1614 (C=C); ¹H NMR (300 MHz, DMSO-*d*₆) δ_{ppm} : 8.71 (d, 1H, *J*=3 Hz, CH-Ar), 8.47 (dd, 1H, *J*=6 Hz, 3 Hz, CH-Ar), 7.70-7.08 (m, 8H, CH-Ar, NH₂), 5.47 (s, 1H, CH); ¹³C NMR (75 MHz, DMSO-*d*₆) δ_{ppm} : 159.5 (C=O), 154.4 (C3), 150.7, 149.2, 149.1, 139.0, 135.6, 135.2, 130.7, 125.5, 125.4, 125.1, 124.6, 119.8, 117.2, 116.9 (CN), 56.6 (C2), 35.5 (C1). Anal. calcd. For C₁₈H₁₁N₃O₃: C, 68.14; H, 3.49; N, 13.24. Found: C, 67.97; H, 3.32; N, 13.08%.

CONCLUSION

In summary, we reported the use of crown ether-based ionic liquids (CE-based ILs), in three-component reaction of aromatic aldehydes, 3-hydroxycoumarin, and malononitrile for the synthesis of 1,5-dihydropyrano[2,3-*c*]chromene derivatives. These reactions were carried out in H₂O/EtOH under reflux. High yields, operational simplicity, and clean reaction conditions are advantages of this procedure that make it a useful practical process for the synthesis of these compounds.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIALS

The authors confirm that the data supporting the findings of this research are available within the article.

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CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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AUTHOR CONTRIBUTIONS

Iman Mohammadzadeh, Mehdi Abaszadeh, Designed and performed experiments, analysed data and wrote the paper. Ali Asadipour, Abbas Pardakhty, Designed experiments and co-wrote the paper. All authors were involved in revising the content, agree to take accountability for the integrity and accuracy of the work, and have read and approved the final manuscript.

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