Volume 15, Issue 2, 152518 (1-7)

Iranian Journal of Catalysis (IJC)

https://doi.org/10.57647/j.ijc.2025.1502.18

Ionic liquid-mediated multi-component reaction towards the synthesis of dihydropyrimidones: An environmentally benign green protocol

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Original Research

Received:
8 March 2024
Revised:
10 July 2024
Accepted:
21 April 2025
Published online:
3 May 2025

Abstract:

Multi-component reaction (MCR) has notable benefits when compared to traditional linear-type synthetic methods, owing to its adaptable, convergent, and atom-efficient properties. Herein, this study reported the three-component reaction using ethylacetoacetate, aldehydes, and urea in one-step synthesis that provided the 3,4-dihydropyrimidin-2(1*H*)-ones in excellent yields, such as 5-(Ethoxycarbonyl)-4-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one obtained up to 94% of yield. The conversion was led by a catalytic amount of sulfuric acid and ionic liquids as a recyclable green solvent at 80 °C. The recyclable activity of the catalytic system up to three cycles shows excellent yields, ranging from 88% to 94%.

Keywords: Ionic liquid (ILs); Imidazolinium ionic liquids; Pyridinium ionic liquids; Dihydropyrimidinones

1. Introduction

MCRs are a major class of synthetic processes in which three or more different starting materials are combined in a one-pot procedure to create a desired single product [1–5]. Due to their large scope of use and their high productivity, ease of use, and convergent nature, MCRs have emerged as essential tools in modern organic synthesis, allowing for the development of diverse compounds for screening purposes [6]. This method provides an easy way to generate molecular complexity and a diverse variety of molecules by forming multiple new covalent bonds in a single reaction step [7–10].

Pyrimidines and their derivatives have received great attention due to their numerous applications in biology and pharmacology. As a result, in recent years, significant effort has been taken on the development of these structural frameworks. The pyrimidine moiety has a broad variety of biological activities [11–15]. The pyrimidine core is a part of various natural alkaloids as well as medicinally important substances [16–18]. Furthermore, it has been observed that dihydropyrimidinone cores with other heterocycles significantly increase the compound's biological activity [19, 20]. As dihydropyrimidinones play a vital role, several methods have been developed for the synthesis of dihydropyrimidone derivatives via multi-component reactions using a variety of catalysts [21–29].

At the start of the twentieth century, the scientific community increased its focus on finding more sustainable ways to produce a wide range of materials due to environmental concerns. This development is most noticeable in the growth of green chemistry [30, 31]. Green chemical methods have the potential to significantly reduce the production

of by-products and waste, as well as lower energy costs. Additionally, they can facilitate the development of new methodologies for obtaining materials that were hitherto difficult or impossible to obtain using existing methods [32]. In recent years, towards a green approach in organic synthesis, ionic liquids as solvents have received significant attention due to their unique properties, like Breslow's rediscovery of water as a solvent during the 1980s. The distinct properties of ionic liquids, such as low vapour pressure, high thermal stability, and tunable polarity, have made them extremely desirable in a wide range of diverse applications, including organic synthesis, catalysis, and separation science. The properties like non-volatility, non-flammability, and low toxicity make the ionic liquid a safer and more environment friendly alternative to traditional organic solvents.

2. Experimental

Materials and methods

All common reagents and solvents were purchased from commercial suppliers and used without further purification. The chemicals Pyridine and 1-Methylimidazole were purchased from Sigma-Aldrich and used without further purification. The 2-bromoethanol and 1-bromopropane were purchased from Aura Chemicals. Ionic liquids were synthesized using some modifications to previously reported procedures [33, 34]. All the experiments were conducted under neat conditions; Aluminium pre-coated silica gel TLC plate 60 F_{254} was used for thin-layer chromatography, and melting points (mp) were recorded using the Digital Analab Scientific Instrument (DASI). The FT-IR spectra were recorded using an Alpha II Bruker spectrophotometer. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker Advance Neo-500 MHz spectrometer in *DMSO-d6*. ESI-MS spectra were performed on a Waters Micromass® Q-ToF Micro™ LC-MS spectrometer.

Synthesis of Ionic Liquids (ILs):

Synthesis of 1-propyl pyridinium bromide (PPBr):

Pyridine (1 mol) and 1-bromopropane (1.1 mol) were placed in a 50 mL two-necked round-bottomed flask. The flask was then placed in an oil bath, heated to 90 °C, and allowed to continue the reaction for 60 minutes. After completion of the reaction, the excess starting materials were removed by washing the reaction mixture with 5 mL of ethyl acetate, and the final product was dried in a vacuum oven at 80 °C for 4 hours. The synthesized ionic liquid was characterised and confirmed by ^{1}H-NMR, ^{13}C-NMR, and mass spectroscopy (scheme 1).

Synthesis of *N*-(2-hydroxyethyl)pyridinium bromide (HEPBr):

Pyridine (1 mol) and 2-bromoethanol (1.1 mol) were placed in a 50 mL two-necked round-bottomed flask. The flask was then placed in an oil bath and heated to 90 °C, and the reaction was continued for 60 minutes. After completion of the reaction, the excess starting materials were removed by washing the reaction mixture with 5 mL of ethyl acetate, and the final product was dried in a vacuum oven at 80 °C for 4 hours. The synthesized ionic liquid was characterised and confirmed by ^{1}H-NMR, ^{13}C-NMR, and mass spectroscopy.

Synthesis of 1-methyl-3-propyl imidazolium bromide (PMImBr):

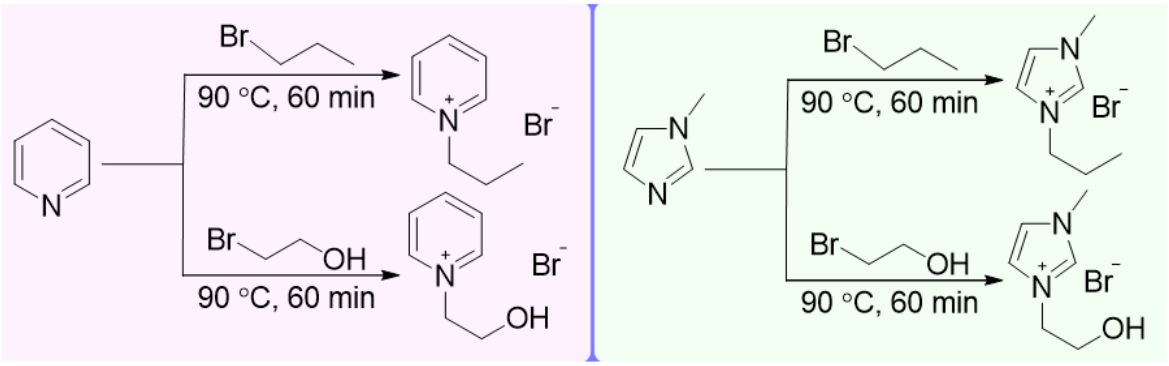
1-Methyl imidazole (1 mol) and 1-bromopropane (1.1 mol) were placed in a 50 mL two-necked round-bottomed flask. The flask was then placed in an oil bath, heated to 90 °C, and allowed to continue the reaction for 60 minutes. After completion of the reaction, the excess starting materials were removed by washing the reaction mixture with 5 mL of ethyl acetate, and the final product was dried in a vacuum oven at 80 °C for 4 hours. The synthesized ionic liquid was characterised and confirmed by ^{1}H-NMR, ^{13}C-NMR, and mass spectroscopy.

Synthesis of 1-(hydroxymethyl)-3-methyl-1*H*-imidazole-3-ium bromide (HEMImBr):

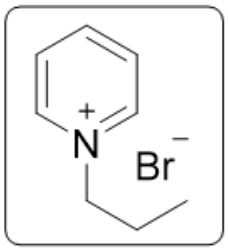
1-Methyl imidazole (1 mol) and 2-bromoethanol (1.1 mol) were placed in a 50 mL two-necked round-bottomed flask. The flask was then placed in an oil bath, heated to 90 °C, and allowed to continue the reaction for 60 minutes. After completion of the reaction, the excess starting materials were removed by washing the reaction mixture with 5 mL of ethyl acetate, and the final product was dried in a vacuum oven at 80 °C for 4 hours. The synthesized ionic liquid was characterised and confirmed by ^{1}H-NMR, ^{13}C-NMR, and mass spectroscopy.

Spectral data

1-Propylpyridinium bromide (PPBr):



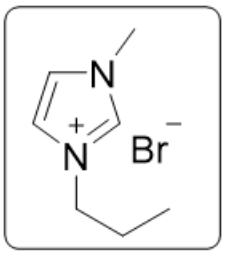
Scheme 1. Ionic liquids (ILs) synthetic pathway.



^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 0.23 – 0.26 (t, 3H), 1.34 – 1.39 (m, 2H), 4.22 – 4.25 (t, 2H), 8.88 – 8.90 (d, 2H), 7.93 – 7.97 (t, 2H), 8.88 – 8.90 (t, 1H). ^{13}C-NMR (125 MHz, *DMSO-d6*): 10.11, 28.90, 62.42, 128.15, 144.72, 145.24.
ESI-MS (m/z): 202.16.

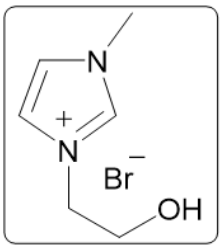
***N*-(2-hydroxyethyl)pyridinium bromide (HEPBr):**
^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 3.93 – 3.97 (q, 2H), 4.85 – 4.87 (t, 2H), 5.31 (s, 1H), 8.26 – 8.29 (q, 2H), 8.72 –8.75 (t, 1H), 9.22 – 9.23 (d, 2H). ^{13}C-NMR (125 MHz, *DMSO-d6*): 35.69, 51.92, 59.20, 122.53, 123.20, 136.66.
ESI-MS (m/z): 207.13.

1-methyl-3-propylimidazolium bromide (PMImBr):



^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 0.11 – 0.14 (t, 3H), 1.11 – 1.15 (m, 2H), 3.30 (s, 3H), 3.49 – 3.52 (t, 2H), 6.95 – 6.96 (d, 1H), 6.99 – 7.00 (d, 1H), 9.23 (s, 1H). ^{13}C-NMR (125 MHz, *DMSO-d6*): 39.71, 111.13, 119.12, 119.39, 119.83, 119.94, 122.11, 123.62, 126.89, 130.52, 131.33, 136.71, 143.11.
ESI-MS (m/z): 205.07.

1-(hydroxyethyl)-3-methyl-1*H*-imidazol-3-ium bromide (HEMImBr):

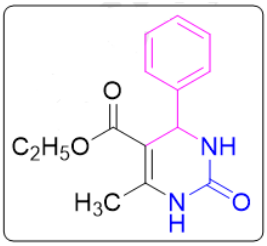


^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 3.70 – 3.71(t, 2H), 3.87 (s, 3H), 4.23 – 4.25 (t, 2H), 5.08 (s, 1H), 7.75 – 7.78 (d, 2H), 9.22 (s, 1H).
^{13}C-NMR (125 MHz, *DMSO-d6*): 35.69, 51.92, 59.20, 122.53, 123.20, 136.66.
ESI-MS (m/z): 192.14.

General procedure

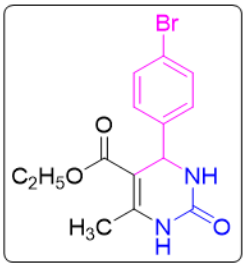
Synthesis of 3,4-Dihydropyrimidin-2(1*H*)-ones:

In a 50 mL round-bottom flask, a mixture of ethyl acetoacetate (1 mmol), aromatic aldehyde (1 mmol), urea (1.5 mmol), and HEMImBr (2 mL) was heated at 80 °C in the presence of sulfuric acid (0.5 mmol) for 25 – 35 minutes. The progress of the reaction was monitored using thin-layer chromatography (TLC). After complete consumption of the starting material, the reaction mixture is allowed to cool to room temperature. Then, adding approximately 10 g of crushed ice to the reaction mixture, the precipitate separated out was filtered, washed thoroughly with ice-cold water, and dried under vacuum. Subsequently, the crude product was purified by recrystallization using hot ethanol. The obtained filtrate was concentrated under reduced pressure to recover the HEMImBr ionic liquid. The recovered ionic liquid was then dried in a vacuum oven for 2 hours and subsequently used for the next experimental cycle.



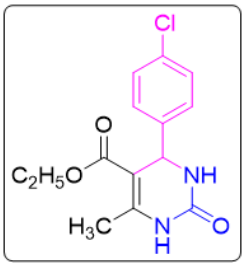
IR (KBr, cm^{-1}): 3427 and 3328 (N–H *str.*), 1685 (> C=O *str.*). ^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 1.08 – 1.11 (t, 3H), 2.25 (s, 3H), 3.96 – 4.01 (q, 2H), 5.15 (s, 1H), 7.23 – 7.25 (m, 3H, Ar-H), 7.31 – 7.34 (m, 2H, Ar-H), 7.72 – 7.73 (d, 1H, NH), 9.18 (s, 1H, NH). ^{13}C-NMR (500 MHz, *DMSO-d6*): 13.96, 17.66, 53.85, 59.06, 99.15, 126.13, 127.14, 128.27, 144.75, 148.25, 152.02, 159.44, 165.23.
ESI-MS (m/z): 261.15.

5-(Ethoxycarbonyl)-4-(4-bromophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4b):



IR (KBr, cm^{-1}): 3460 and 3320 (N–H *str.*), 1658 (>C=O *str.*). ^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 1.08 – 1.11 (t, 3H), 2.25 (s, 3H), 3.96 – 4.01 (q, 2H), 5.45 (s, 1H), 6.80 – 6.81 (d, 2H, Ar-H), 7.84 – 7.85 (d, 2H, Ar-H), 9.26 (d, 1H, NH), 10.00 (s, 1H, NH). ^{13}C-NMR (500 MHz, *DMSO-d6*): 14.00, 17.72, 53.41, 59.19, 98.71, 128.61, 131.23, 135.04, 144.10, 148.63, 151.89, 155.79, 159.59, 165.12.
ESI-MS (m/z): 338.37.

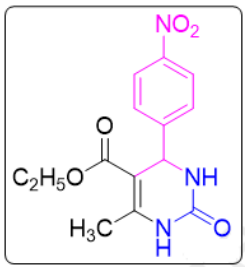
5-(Ethoxycarbonyl)-4-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4c):



IR (KBr, cm^{-1}): 3154 (N–H *str.*), 1683 (>C=O *str.*). ^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 0.98 – 1.01 (t, 3H), 2.30 (s, 3H), 3.87 – 3.92 (q, 2H), 5.41 (s, 1H), 7.27 – 7.35 (m, 4H, Ar-H), 9.26 (s, 1H, NH), 10.35 (s, 1H, NH). ^{13}C-NMR (500 MHz, *DMSO-d6*): 13.80, 17.55, 51.38, 58.95, 97.78, 128.86, 131.57, 135.69, 141.61, 149.17, 151.21, 157.19, 164.85.

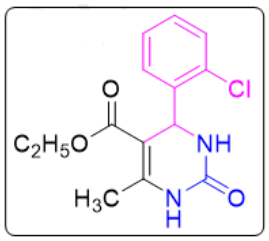
ESI-MS (m/z): 295.11.

5-(Ethoxycarbonyl)-4-(4-nitrophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4d):



IR (KBr, cm^{-1}): 3360 (N–H *str.*), 1661 (>C=O *str.*). ^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 1.08 – 1.11 (t, 3H), 2.27 (s, 3H), 3.97 – 4.01 (q, 2H), 5.28 (s, 1H), 7.88 – 8.23 (m, 4H, Ar-H), 9.34 (s, 1H, NH), 10.17 (s, 1H, NH). ^{13}C-NMR (500 MHz, *DMSO-d6*): 13.94, 17.76, 53.58, 59.58, 98.08, 127.55, 130.53, 139.97, 146.61, 149.28, 150.53, 152.89, 164.95.
ESI-MS (m/z): 306.13.

5-(Ethoxycarbonyl)-4-(2-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4e):



IR (KBr, cm^{-1}): 3334.25 and 3244.60 (N–H *str.*), 1679.32 (>C=O *str.*). ^{1}H-NMR (500 MHz, *DMSO-d6*): δ (ppm) 1.08 – 1.11 (t, 3H), 2.26 (s, 3H), 3.97 – 4.01 (q, 2H), 5.44 (s, 1H), 7.25 – 7.45 (m, 4H, Ar-H), 9.25 (s, 1H, NH), 10.01 (s, 1H, NH). ^{13}C-NMR (500 MHz, *DMSO-d6*): 13.98, 17.72, 53.35, 58.55, 98.77, 128.10, 131.51, 134.76, 139.30, 143.70, 148.62, 151.89, 155.79, 159.57, 165.12.
ESI-MS (m/z): 295.11.

3. Result and discussion

Herein, this manuscript presents the synthesis of derivatives of 3,4-dihydropyrimidin-2(1*H*)-one by the reaction of appropriate aldehydes, ethyl acetoacetate, and urea in the presence of 0.5 mmol of sulfuric acid in the ionic liquid HEMImBr (scheme 2).

In this reaction, after the addition of Conc. H_2SO_4 yield of the product increases, this might be due to the bromine (Br^-) ion converted into the most acidic hydrogen sulphate (HSO_4^-) ions (scheme 3), which increases the acidity, and the [HEMIm] $^+$ [Br]$^-$ is converted to [HEMIm] $^+$ [HSO_4]$^-$ [35].

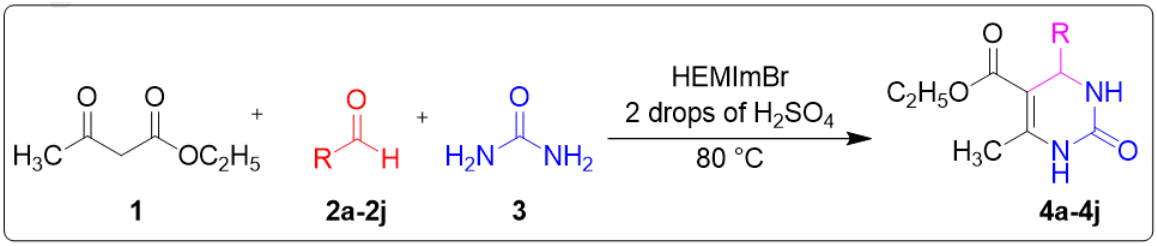
In initial findings, 4-chlorobenzaldehyde, ethyl acetoacetate, and urea were reacted in the presence of a catalytic amount (0.5 mmol) of Conc. H_2SO_4 and four different ionic liquids such as 1-propylpyridinium bromide (PPBr), 1-propyl-3-methylimidazolium bromide (PMImBr), 1-(2-hydroxyethyl)pyridinium bromide (HEPBr), and 1-ethanol-3-methyl-imidazolium bromide (HEMImBr). During optimisation of the reaction condition, the reaction was performed with and without conc. H_2SO_4 in (Table 1).

An investigation began with optimizing the reaction with the ionic liquid 1-propylpyridinium bromide (PPBr). In this investigation, after extensive experimentation, it was found that PPBr as a solvent without a catalyst resulted in a yield of 67% of 5-(Ethoxycarbonyl)-4-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one in 67% of the yield with 45 45-minute reaction time. The same reaction was performed by adding 0.5 mmol of conc. H_2SO_4 acid as a catalyst with the PPBr, the yield increased slightly from 67% to 74%. Also, the early consumption of the starting material, confirmed by TLC analysis, indicates the reaction time decreased from 45 minutes to 40 minutes.

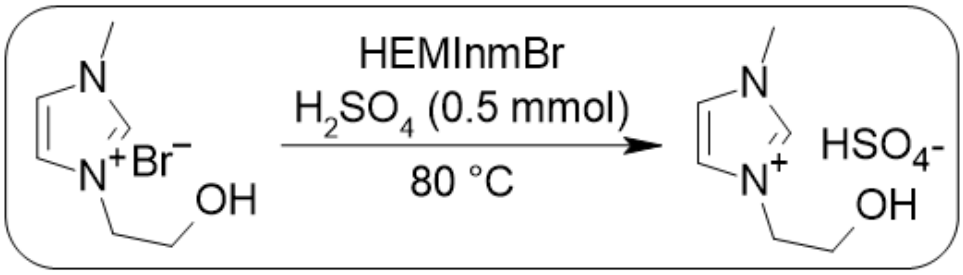
Then screen the above reaction using ionic liquids PMImBr, HEPBr, and HEMImBr with conc. H_2SO_4 to exchange the Br^- ions to HSO_4^- ion and find that the reaction works well with moderate to excellent yields, such as 75%, 77%, and 94%, respectively, with a reduced reaction time (Table 1). In the reaction condition optimisation, it was found that the ionic liquid HEMImBr acts as an excellent solvent with the combination of 0.5 mmol Conc. H_2SO_4.

The efficacy of the reaction condition using 0.5 mmol Conc. H_2SO_4 and solvent (HEMImBr) were further checked for the various benzaldehydes (1 mmol) with ethyl acetoacetate (1 mmol) and urea (1.5 mmol), heated at 80 °C. The progress of the reactions was monitored by TLC analysis. After completion, the reaction mixture was diluted with cold water, and the products were isolated by filtration. The filtrate obtained after the product was isolated was evaporated under reduced pressure to recycle the ionic liquid, which was dried in a vacuum oven for 2 hours and reused for the next run (Table 2). The purified product was characterised and identified using IR, ^{1}H/^{13}C-NMR, and mass spectral analysis.

The reusability of the catalytic system of ionic liquids (ILs) with 0.5 mmol Conc. H_2SO_4 was evaluated by three cycles of the reactions of ethyl acetoacetate (1 mmol), 4-chlorobenzaldehyde (1 mmol), and urea (1.5 mmol) at 80 °C, and the progress of the reaction was monitored by TLC analysis. The product, being insoluble in water, was easily separated after filtration. The remaining filtrate containing



Scheme 2. Synthetic pathway of 3,4-Dihydropyrimidin-2(1*H*)-one.



Scheme 3. Ionic liquid HEMImBr *in situ* converted to $HEMImHSO_4$.

Table 1. Optimization of reaction conditions with various parameters for the model reaction.

Entry	Solvent[a]	Catalyst[b]	Temp °C	Time[c]	Yield (%)[d]
1	PPBr	-	80	45	67
2	PPBr	H_2SO_4	80	40	74
3	PMImBr	-	80	38	70
4	PMImBr	H_2SO_4	80	35	75
5	HEPBr	-	80	40	70
6	HEPBr	H_2SO_4	80	35	77
7	HEMImBr	-	80	32	85
8	HEMImBr	H_2SO_4	80	28	94

(a) Ionic liquids 2 mL for all reactions; (b) 0.5 mmol H_2SO_4 for all reactions; (c) Time in minutes; (d) Isolated yields.

ILs was isolated under reduced pressure at 80 °C, dried for two hours in a vacuum oven at 70 °C, and then reused. Both the ILs and the catalyst exhibited reusability with only a slight decrease in their activity, as presented in (Table 3). Upon comparison with the results obtained from the previously reported method, as mentioned in Table 4, it is evident that the optimized approach requires 29 minutes to achieve a remarkable 94% yield. This noteworthy improvement highlights the effectiveness and efficiency of the modified method in the synthesis process.

4. Conclusion

3-(2-Hydroxyethyl)-1-methylimidazolium bromide (HEMImBr) proved to be an incredibly effective and user-friendly green solvent for the synthesis of dihydropyrimidone. Employing a mere 0.5 mmol of Conc. H_2SO_4 as a catalyst, we successfully achieved high yields from ethyl acetoacetate, urea, and a diverse range of aldehydes,

Table 2. Synthesis of derivatives of 3,4-Dihydropyrimidin-2(1*H*)-one (4a–j) by catalytic amount of H_2SO_4 in HEMImBr as solvent.

Entry	Carbonyl compound (2a-j)	Product[a]	Time[b]	Yield (%)[c]	MP °C [Ref.]
1	C_6H_5-CHO (2a)	4a	29	92	198-199 [3]
2	p-BrC_6H_4-CHO (2b)	4b	28	90	213-214 [3]
3	p-ClC_6H_4-CHO (2c)	4c	28	94	215-216 [3]
4	p-$NO_2C_6H_4$-CHO (2d)	4d	26	89	209-210 [17]
5	o-ClC_6H_4-CHO (2e)	4e	24	90	221-222 [11]
6	p-$OCH_3C_6H_4$-CHO (2f)	4f	30	88	204-205 [11]
7	o-$NO_2C_6H_4$-CHO (2g)	4g	26	90	220-221 [11]
8	p-OHC_6H_4-CHO (2h)	4h	29	87	228-229 [11]
9	p-FC_6H_4-CHO (2i)	4i	31	84	173-174 [11]
10	m-BrC_6H_4-CHO (2j)	4j	34	86	194-195 [11]

(a) Ratio of starting materials: Benzaldehydes (1 mmol), ethyl acetoacetate (1 mmol), and urea (1.5 mmol), and 0.5 mmol H_2SO_4 used for all reactions; (b) Time in minutes; (c) Isolated yields.

Table 3. Reusability of Ionic liquid and catalyst.

No. of Cycles	Fresh	Run 1	Run 2	Run 3
Yield[a]	94	93	90	88
Time (Min)	28	28	29	29

Reaction conditions: Ethyl acetoacetate (1 mmol), 4-chlorobenzaldehyde (1 mmol) and urea (1.5 mmol); ILs and catalyst; temp: 80 °C; [a]isolated yields.

Table 4. Comparison of some reported methods for the synthesis of dihydropyrimidinone derivatives through Biginelli reaction.

Entry	Catalytic system	Condition	Time	Yield %	Ref.
1	[CEMIM][MSA]	75 °C	85 min	95	[22]
2	[MImH][OAc]	100 °C	45 min	87	[36]
3	Mag@Morph-AIL	80 °C	64 hr	86	[37]
4	[BMIM][HSO_4]	80 °C	1.5 hr	83	[38]
5	DES	50 °C	10 hr	94	[39]
7	HEMImBr/H_2SO_4	80 °C	29 min	94	This Work

thereby significantly reducing the utilization of organic volatile solvents towards an environment-friendly approach. This one-pot synthetic approach offers several compelling advantages, including rapid reaction time, the utilization of inexpensive and easily synthesized solvents to achieve high yields due to facile product separation, and the reusability of both the ionic liquid and catalyst. Consequently, this research provides a promising, efficient, and cost-effective approach for researchers to explore such frameworks.

Acknowledgement
We are grateful to the Principal, Arts and Science College, Bhalod for providing laboratory facilities and encouragement, and SAIF, Punjab University, Chandigarh for the NMR analysis.

Authors contributions
Authors have contributed equally in preparing and writing the manuscript.

Availability of data and materials
The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflict of interests
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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