RESEARCH PAPER

The Ultrasonic-Assisted Green Synthesis of Pyranopyrazoles Using Recyclable Brønsted-acidic ionic liquid [Hmim]TFA Catalyst

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ABSTRACT

The primary objective of this study was to explore an ultrasonic-assisted multicomponent strategy for synthesizing pyranopyrazole derivatives using the Brønsted-acidic ionic liquid [Hmim]TFA as a recyclable catalyst and reaction medium. It has carried out the condensation of substituted benzaldehydes, ethyl acetoacetate, hydrazine hydrate, and malononitrile to study the efficiency of [Hmim]TFA in promoting the cyclocondensation process. The plan has completed to recover the ionic liquid after product isolation and examine its reusability over multiple cycles. Through this investigation, we seek to determine the applicability of [Hmim]TFA as a sustainable catalytic system under mild ultrasonic conditions.

Keywords: Ultrasonic-assisted, Pyranopyrazole, Brønsted-acidic ionic liquid [Hmim]TFA, Eco-friendly synthesis, Recyclable catalyst and reaction medium.

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INTRODUCTION:

Organic solvents remain widely employed in synthetic chemistry and their extensive use poses environmental concerns and challenges related to safety, volatility, and waste generation.Ionic liquids (ILs) address this challenge through their strong solvating power, thermal stability, non-volatility, and recyclability, making them effective green media and catalysts [1, 2].Ultrasound-assisted methods further enhance IL-mediated reactions by accelerating transformations and reducing energy input. Brønsted acidic ILs such as 1-Methylimidazolium trifluoroacetate([Hmim]TFA) have shown excellent catalytic performance under these conditions [3].Beyond conventional roles, ILs are structurally tuned to create task-specific ionic liquids (TSILs), which combine solvent and reagent functions, offering efficient and sustainable alternatives for modern chemical processes [4–8].

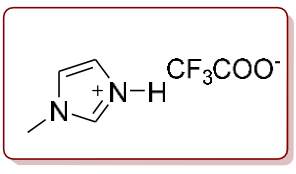


Fig-1: Structure Methylimidazoliumtrifluoroacetate ([Hmim]TFA)

Multi-component reactions (MCRs) involve the combination of three or more reactants in a single operation to form structurally complex products, thereby avoiding lengthy multi-step procedures [9]. Their convergent nature has made MCRs highly valuable in organic and medicinal chemistry, particularly for the rapid construction of heterocyclic scaffolds and for generating diverse libraries of drug-like molecules with strong combinatorial efficiency [10]. Alongside their synthetic power, MCRs are increasingly recognized for their environmental benefits, as they minimize waste, shorten reaction times, and simplify purification. These combined advantages efficiency, operational simplicity, and sustainability make MCRs superior to traditional stepwise synthetic routes and highly relevant to modern green chemistry practices [11].Pyranopyrazoles represent a versatile class of pharmacologically relevant heterocycles that have gained considerable prominence in synthetic and medicinal chemistry. These fused frameworks exhibit a broad range of biological properties, including fungicidal [12], bactericidal [13], and molluscicidal activities [14], along with important anticancer potential [15]. They are also known for their

effectiveness as cyclooxygenase (COX) inhibitors [16–18] and as cannabinoid inverse agonists useful in obesity management [19].In addition, pyranopyrazole derivatives demonstrate antihypertensive [20], antimicrobial [22], insecticidal [23], and antifungal activities [24]. Their medical relevance is underscored by the presence of pyrazole-based motifs in several approved drugs, including sulfaphenazole, celecoxib, rimonabant, and mepiprazole [25]. More recently, related scaffolds have shown promise as potent Chk1 kinase inhibitors, reinforcing their potential in anticancer agent development [26]. These attributes highlight pyranopyrazoles as valuable platforms for innovative drug discovery [27].

In addition, several environmentally benign four-component synthetic methodologies have been introduced employing a diverse set of catalytic systems. Among these, DABCO [28], γ-alumina [29], Amberlyst A21 [30], triethylamine [31], and hexadecyl dimethyl benzyl ammonium chloride [32] have demonstrated notable efficiency in promoting the desired transformations under mild and sustainable conditions. More recently, the catalytic utility of per-6-amino-β-cyclodextrin [33], basic ionic liquids [34], and piperidine [35] has further expanded the scope of these green multicomponent reactions, underscoring continued advancements in environmentally responsible synthetic strategies. A broader examination of the literature reveals that numerous organocatalysts, including baker's yeast [36], L-proline [37], chitosan [38], meglumine [39], and thiamine hydrochloride [40] have been successfully applied across a variety of organic transformations, thereby highlighting their versatility and operational simplicity. Alongside these, a range of heterogeneous catalytic systems such as BSA [41], ZnO [42], $FeNi_3$@SiO_2–HPG [43], SBA-$PrNH_2$ [44], and TrCl [45] have also been reported, offering advantages in terms of catalyst separation and potential reusability. Even, many of these catalytic protocols still encounter major drawbacks, including limited recyclability, suboptimal product yields, extended reaction durations, and persistent dependence on conventional thermal heating. These limitations collectively underscore the continuing need for the development of more robust, efficient, and sustainable catalytic methodologies. With the considerations of this view, in this report a clean and efficient synthesis of pyranopyrazole derivatives via a one-pot, four-component condensation protocol catalyzed by 1-Methylimidazolium trifluoroacetate([Hmim]TFA). This eco-friendly and inexpensive ionic-liquid catalyst which also acts as reaction medium enables the transformation to proceed under ultrasonic irradiation at 80 °C, affording the desired products in good to excellent yields.

EXPERIMENTAL:

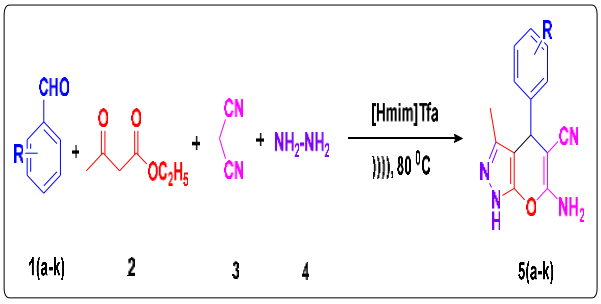
Material and method

All chemicals and solvents used for the preparation of Pyranopyrazole derivatives were of analytical grade (≥99% purity) and were obtained from reputable suppliers, including Sigma-Aldrich. When necessary, reagents were purified prior to their application in the reaction. The progression of each reaction was routinely assessed using thin-layer chromatography (TLC) on silica gel plates. Melting points of the synthesized products were determined with a capillary melting point apparatus and are presented as uncorrected values. Structural confirmation of the final compounds was achieved through infrared (IR) and nuclear magnetic resonance (NMR) spectroscopic analyses, employing tetramethylsilane (TMS) as the internal reference standard.

General Procedure for the synthesis of Pyranopyrazole derivative

A reaction mixture containing ethyl acetoacetate (1 mmol), hydrazine hydrate (1 mmol), the desired aldehyde (1 mmol), malononitrile (1 mmol), and 1-methylimidazolium trifluoroacetate ([Hmim]TFA, 7.5 mol%) as the catalyst and also which acts as a reaction medium

and mixed thoroughly. The mixture was then exposed to ultrasonic irradiation at 80 °C for the required duration, with the progress monitored by TLC.After completion, the reaction mass was allowed to cool to ambient temperature and was subsequently poured onto crushed ice. The precipitated product was isolated by filtration, washed with water, and dried. Final purification was achieved by recrystallization inethanol, yielding the corresponding pyranopyrazole derivative.The aqueous filtrate containing the ionic liquid catalyst was concentrated under reduced pressure to remove the solvent. The resulting residue, corresponding to the recovered [Hmim]TFA, was directly reused in the next cycle without any additional purification.



Scheme 1: Synthesis of pyranopyrazole catalyzed by [Hmim]TFA under ultrasonic irradiation.

RESULT AND DISCUSSION:

Catalytic activity

The reaction parameters for the ultrasonic-assisted synthesis of the pyranopyrazole derivative were optimized using benzaldehyde as the model substrate. Optimization of the reaction conditions was systematically carried out using [Hmim]TFA as a dual-function medium, serving both as catalyst and solvent under ultrasonic irradiation. In the absence of catalyst (Table 1, Entries 1 and 2), the reaction proceeded very sluggishly, giving only trace to 6% yield even after prolonged sonication (240–480 min), confirming the essential catalytic role of the ionic liquid.

Table 1: Optimization of the reaction condition for the synthesis of pyranopyrazole.

Entry	Catalyst	Amount (mol %)	Solvent	Time (Min.)	Temp. (^{0}C)	Yield (%)^X
1	-	-	-	240	rt	Trace
2	-	-	-	480	rt	6
3	[Hmim]TFA	15	-	60	rt	68
4	[Hmim]TFA	12.5	-	60	rt	68
5	[Hmim]TFA	10	-	55	rt	66
6	[Hmim]TFA	7.5	-	50	rt	65
7	[Hmim]TFA	5	-	80	rt	53
8	[Hmim]TFA	2.5	-	100	rt	45
9	[Hmim]TFA	7.5	-	50	60	68
10	[Hmim]TFA	7.5	-	30	80	85

X is the isolated yield of the product.
All reaction performed under ultrasonic irradiation.

Varying the catalyst loading from 15 mol% to 2.5 mol% (Table II-1, Entries 3–8) showed that 7.5 mol% of [Hmim]TFA offered the best balance between efficiency and economy, providing a 65% yield in 50 minutes at room temperature. Higher loadings (10–15 mol%) gave only marginal improvements (66–68%), while lower loadings (5–2.5 mol%) resulted in a noticeable decline in product formation (53–45%).

Temperature optimization at the optimal loading of 7.5 mol% (Table 1, Entries 6, 9-10) revealed that increasing the temperature from room temperature to 40 °C and 80 °C enhanced the yield from 65% to 68% and 85%, respectively. The important improvement at elevated temperatures highlights the synergistic effect of mild heating and ultrasonic energy, which likely accelerates molecular collisions and improves catalytic turnover in the ionic liquid environment. The study confirms that 7.5 mol% [Hmim]TFA, used without any additional solvent, combined with ultrasonic irradiation and mild heating (80 °C), delivers the maximum isolated yield of 85%, establishing these conditions as optimal for the efficient synthesis of pyranopyrazole derivatives.

Table 2: Comparison of [Hmim]TFA with commercially available catalyst.

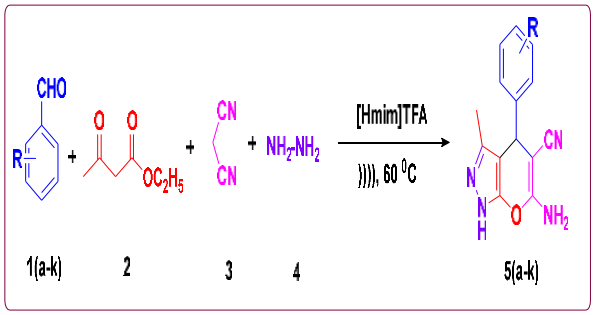
Entry	Catalyst (mol%)	Time (Min.)	Conversion (%)	Yield (%)^X
1	$ZnCl_2$	135	35	15
2	$AlCl_3$	120	42	25
3	$SnCl_4$	95	54	32
4	Na_2S	90	56	35
5	TFA	60	66	48
6	Al_2O_3	45	88	65
7	Pyridine	120	48	30
8	ZrO_2	40	95	72
9	**[Hmim]TFA**	**30**	**100**	**85**

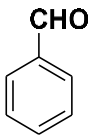
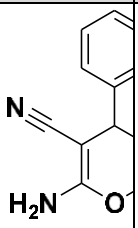
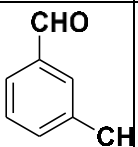
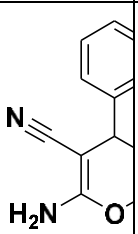
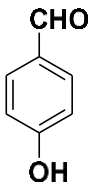
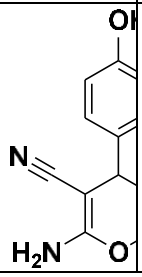
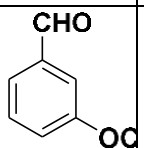
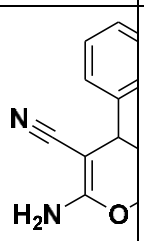
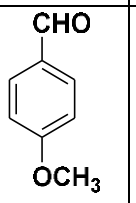
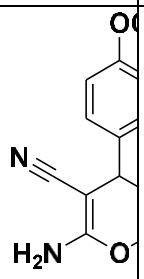
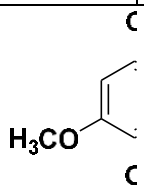
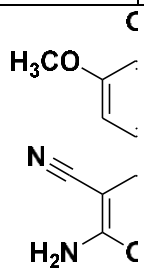
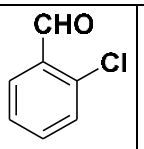
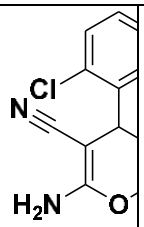
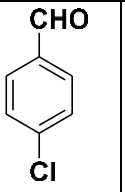
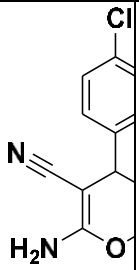
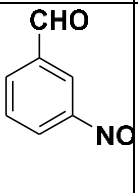
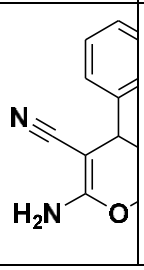
X is the isolated yield of the product.
All Catalyst taken in an amount of 7.5 mol%.
All Reaction carried under ultrasonic irradiation.

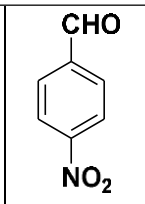
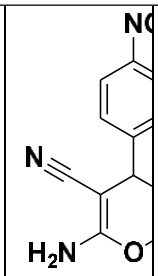
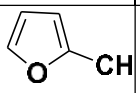
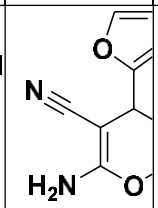
A comparative catalyst screening study was undertaken using a fixed loading of 7.5 mol% under ultrasonic irradiation to identify an efficient promoter for the synthesis of the pyranopyrazole derivative. Traditional Lewis acids such as $ZnCl_2$, $AlCl_3$, and $SnCl_4$ displayed limited efficiency, providing conversions of 35–54% and isolated yields of only 15–32%, indicating insufficient activation of the carbonyl substrates under these conditions (Table-2, Entries 1-3). Mild bases and inorganic materials, including Na_2S and pyridine, also performed poorly, delivering modest yields of around 30–35%. (Table-2, Entry4 and 8), whereas TFA showed moderate catalytic behaviour with 48% yield in 60 minutes (Table-2, Entry 5). Notably, solid supports such as Al_2O_3 and ZrO_2 exhibited improved activity, achieving 65% and 72% yields, respectively Table-2, Entries 6, 7), likely due to their surface acidity and enhanced cavitation effects under ultrasonication. Among all catalysts tested, the Brønsted-acidic ionic liquid [Hmim]TFA demonstrated superior catalytic performance, delivering complete conversion and the

highest isolated yield of 85% within only 30 minutes(Table-2 Entries 9). This exceptional efficiency is attributed to its strong yet tunable acidity, ability to stabilize charged intermediates, and excellent compatibility with ultrasonic conditions, which collectively accelerate each step of the multicomponent process.

Table 3.Substrate Electronic Effects in the Ultrasonic [Hmim]TFACatalyzed Synthesis of Pyranopyrazoles.



Entry	Aromatic aldehyde	Product	Time (Min.)	Yield (%)[X]	M.P. Obs.	M.P. Rep.[46-49]
5a			30	85	245-246	246-248
5b			45	81	200-202	200-202
5c			50	78	220-222	223-223
5d			30	83	226-228	227-230
5e			50	76	210-212	210-211
5f			50	78	220-221	219-222
5g			35	87	246-247	245-246
5h			25	90	234-235	233-234
5i			25	92	190-191	190-192

5j			20	94	255-2256	253-2254
5k			25	90	214-2166	216-2188

X is the isolated yield of the product.
All Reaction carried under ultrasonic irradiation.

After establishing the optimal ultrasonic-assisted reaction conditions for synthesizing pyranopyrazole derivatives from benzaldehyde, ethyl acetoacetate, malononitrile, and hydrazine hydrate, the electronic influence of substituents on benzaldehydes was systematically evaluated. The parent benzaldehyde, when subjected to the standardized parameters in the presence of the Brønsted-acidic ionic liquid [Hmim]TFA, afforded the corresponding product (Table-3, 5a) in an excellent 85% yield within 30 minutes. Here, [Hmim]TFA plays a vital catalytic role by protonating and activating the carbonyl groups, thereby enhancing electrophilicity, stabilizing charged intermediates, and promoting rapid proton-transfer steps essential for efficient cyclization, all of which are further accelerated by ultrasonic irradiation.

To investigate electron-releasing effects, benzaldehydes bearing 2-methyl, 4-hydroxy, 3-methoxy, 4-methoxy, and 3,4,5-trimethoxy substituents were explored, giving the respective pyranopyrazoles (Table-3, 5b–5g) in 81%, 78%, 83%, 76%, and 78% yields, requiring 30–50 minutes for completion. The modest reduction in yield and longer reaction times observed for these substrates originate from the electron-donating nature of the substituents, which decreases the electrophilicity of the aldehyde carbonyl carbon and renders nucleophilic addition less favourable, thus slowing the key condensation and cyclization steps despite catalytic activation by [Hmim]TFA.

In contrast, benzaldehydes containing electron-withdrawing substituents such as 2-chloro, 4-chloro, 3-nitro, 4-nitro, and furfuraldehyde produced the corresponding derivatives (Table-3, 5h–5k) in markedly higher yields of 87%, 90%, 92%, 94%, and 90%, respectively, within shorter reaction times of 20–35 minutes. This enhanced performance is attributed to the strong electron-withdrawing effects that increase the electrophilic character of the carbonyl carbon, enabling faster nucleophilic attack, smoother intermediate formation, and more efficient cyclization under the synergistic influence of [Hmim][TFA] and ultrasonic irradiation.

Recyclability study of [Hmim]TFA:

Furthermore, the reaction work-up was simple, as the catalyst could be easily separated from the crude product and reused, enhancing the environmental compatibility of the process. After completion, the reaction mixture was cooled to room temperature and poured onto crushed ice to precipitate the product, which was collected by suction filtration and washed with water. The aqueous filtrate containing the ionic liquid catalyst was concentrated under reduced pressure, and the recovered catalyst was reused directly without further purification.

Recyclability studies demonstrated that the catalyst showed excellent efficiency in the first cycle, affording a 98% yield, and retained its activity for up to four consecutive runs. However, a sharp decline in performance was observed in the fifth cycle, with the yield decreasing to 65%, indicating reduced catalytic efficiency upon extended reuse.

Recyclability study of [Hmim]TFA.

Efficiency of catalyst (%)	98	96	95	90	65
No. of Cycle	1	2	3	4	5

Spectral data of some synthesized compounds

The structure of the synthesized compounds was validated through spectral analysis, which corresponded well with their physical properties and previously reported literature data. [46-49].

6-amino-3-methyl-4-phenyl-2,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile (5a)

FT-IR (KBr, cm^{-1}):744, 888, 1044, 1071, 1158,1428, 1428, 1595, 2191, 3170, 3311, 3372; **^{1}H NMR (DMSO-d$_6$, δ ppm):** 1.75 (s, 3H), 4.56 (s, 1H), 6.82 (s, 2H), 7.12-7.30(m, 5H), 12.05 (s, 1H); **^{13}C NMR (DMSO-d$_6$, δ ppm):** 10.4, 37.2, 58.4, 97.8, 121.3, 127.5, 127.5, 127.8, 129.1, 136.2, 144.5, 155.5,161.8; **MS (ESI^{+}): m/z** 253 (M+1)$^{+}$.

6-amino-3-methyl-4(4-methylphenyl)-2,4-dihydropyrano[2, 3-c]pyrazole-5-carbonitrile (5b)

FT-IR (KBr, cm^{-1}): 1385, 1594, 1649, 2184, 3186, 3398; **^{1}H NMR (DMSO-d$_6$, δ ppm):** 1.60 (s, 3H), 2.18 (s, 3H), 4.29 (s, 1H), 6.72 (s, 2H), 7.14 (m, 4H), 11.88 (s, 1H); **^{13}C NMR (DMSO-d$_6$, δ ppm):** 9.1,

20.0, 35.0, 58.0, 97.0, 118.0, 120.0, 127.0, 129.0, 135.0, 140.0, 155.0, 160.0; **MS (ESI$^+$): m/z** 266 (M^+).

6-Amino-1,4-dihydro-4-(4-hydroxyphenyl)-3-methylpyrano[2,3-c]pyrazole-5-carbonitrile (5c)

FT-IR (KBr, cm^{-1}): 1579, 1646, 2215, 3052, 3347, 3408; **^{1}H NMR (DMSO-d$_6$, δ ppm):** 1.92 (s, 3H), 4.58 (s, 1H), 6.03 (d, 2H), 6.36 (s, 2H), 7.07 (d, 2H), 11.05 (s, 1H), 12.15 (s, 1H); **^{13}C NMR (DMSO-d$_6$, δ ppm):** 10.5, 27.8, 69.5, 116.0, 121.9, 124.9, 134.2, 137.6, 146.3, 149.0, 158.0, 163.2; **MS (ESI$^+$): m/z** 269 (M+1).

Amino-3-methyl-4-(3-methoxyphenyl)-2,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile (5d)

IR (KBr, cm^{-1}):1047, 1400, 1601, 1651, 2194, 3117, 3225, 3473; **^{1}H NMR (DMSO-d$_6$, δ ppm)**: 1.80 (s, 3H), 3.71 (s, 3H), 4.56 (s, 1H), 6.71 (s, 1H), 6.71–6.72 (d, 1H), 6.78–6.80 (d, 1H), 6.88 (s, 2H), 7.20–7.24 (t, 1H), 12.10 (s, 1H); **^{13}C NMR (DMSO-d$_6$, δ ppm):**12.6, 55.9, 114.2, 115.6, 118.9, 121.5, 125.7, 129.8, 131.6, 136.4, 142.1, 147.8, 152.3, 156.9, 162.4; **MS (ESI$^+$): m/z** 282 (M+).

6-Amino-1,4-dihydro-4-(4-methoxyphenyl)-3-methylpyrano[2,3-c]pyrazole-5-carbonitrile (5e)

FT-IR (KBr, cm^{-1}):871,1009, 1172, 1257, 1506, 2190, 2206, 3109, 3256, 3483; **^{1}H NMR (DMSO-d$_6$, δ ppm):** 1.80 (s, 3H), 3.74 (s, 3H), 4.55 (s, 1H), 6.82 (s, 2H), 6.88–7.10 (m, 4H), 12,08 (s, 1H); **^{13}C NMR (DMSO-d$_6$, δ ppm):**10.2, 35.9, 55.4, 58.1, 98.3, 114.2121.2, 128.9, 136.0, 136.9, 155.2, 158.4, 161.1 ; **MS (ESI$^+$): m/z** 283 (M+1).

CONCLUSION:

This work presents an environmentally benign and ultrasonically assisted protocol for the efficient synthesis of pyranopyrazole derivatives using the Brønsted-acidic ionic liquid [Hmim]TFA, which functions simultaneously as a recyclable catalyst and reaction medium. The optimized four-component condensation of substituted benzaldehydes, ethyl acetoacetate, malononitrile, and hydrazine hydrate proceeded smoothly, affording the desired products in 76–94% yields within short reaction times. After product isolation, the aqueous filtrate was concentrated under reduced pressure to recover [Hmim]TFA, which retained its catalytic activity over four consecutive cycles without appreciable loss in efficiency. The combination of ultrasonic activation, high yields, operational simplicity, and catalyst reusability underscores the sustainability and synthetic value of this methodology for the construction of pyranopyrazole scaffolds.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts ofinterest.

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