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[BMIM][HSO_4] Ionic liquid catalyzed solvent-free microwave assisted one-pot multicomponent synthesis of tetrahydrobenzo[b]Pyran Derivatives

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ABSTRACT:
We present a simple and environmentally friendly protocol for synthesizing tetrahydrobenzo[b]pyran derivatives through multicomponent condensation of dimedon, malononitrile, and various aromatic aldehydes in the presence of 1-ethyl-3-methylimidazolium hydrogen sulphate [BMIM][HSO_4] as a catalyst under microwave irradiation. The one-pot synthesis, simple solvent-free conditions, and high isolated yield demonstrate the usefulness of this green technique. The structural features are determined utilizing analytical approaches like Fourier Transform Infrared Spectroscopy (FT-IR) and 1H and 13C Nuclear Magnetic Resonance (NMR) Spectroscopy. To maximize yield, electronic synthesis of tetrahydrobenzo[b]pyran derivatives was performed employing the catalytic activity of 1-ethyl-3-methylimidazolium hydrogen sulphate.

Keywords; tetrahydrobenzo[b]pyran, microwave irradiation, solvent free, ionic liquid, FT-IR, TLC.

I. INRTODUCTION

Chemistry is constantly working to develop sustainable and environmentally friendly reactions and methodologies. It is critical to consider green chemistry principles and put them into action by implementing strategies that shift the chemical industry toward sustainability.[1] In conclusion, there have been foremost concerns about waste minimization and sustainability, as there are considerable current issues involving environmental aspects.[2] As of now the multicomponent reactions are executed in ionic fluids which possess enormous advantages and environmentally benign.[3- 4] It's have been portrayed as a green medium which is safe for environment [5] and nowadays their utilization in chemical industry has become particularly important. [6] In recent times, the applications of ionic liquids to execute multicomponent reactions have been currently cited.

Ionic liquids are salts in liquid form with extremely low vapor pressure. Most ionic liquids have low combustibility, strong thermal stability, electrical conductivity, and solvating properties. [8] Many separation or catalytic procedures use ionic liquids as a reaction medium, given that a wide range of organic, inorganic, and polymeric molecules are soluble in ionic liquids. The solvating power of an ionic liquid is dependent on the smaller anion and the huge size of the organic cation. Ionic liquids contain cations such as ammonium, imidazolium, phosphonium, pyrrolidinium, pyridinium, as well as anions such as acetate, formate, benzoate, tetrafluoroborate, trifluromethane sulfonate, nitrate, phosphate, hexafluorophosphate, hydrogen sulphate, and so on.

For the synthesis of organic molecules use of microwave irradiation [9- 10] is a powerful dielectric heating tool which is fast, efficient, simple and clean method. [11- 17] For the synthesis of different heterocycles, the combination of microwave and ionic liquids is of great interest and proves to be a green protocol. [18-20] The 4H benzo[b]pyran derivatives possess broad spectrum of biological properties [21] like anticancer, diuretics, anti-coagulant, spasmolytic, anti-anaphylactic activity. [22- 25] Besides this they have other important applications in treating neurodegenerative disease, including Alzheimer disease, Parkinson's disease, Huntington's disease,

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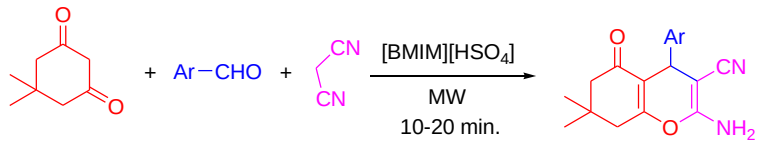
amyotrophic lateral sclerosis, Downs's syndrome, AID's associated dementia as well as for the management of Schizophrenia and myoclonus. [26]

The 4H benzo[b]pyran derivatives are also employed to promote cognitive function. 4H pyrans are also a structural unit in many natural goods [27]. Different 2-amino-4H-pyrans have been used as photoactive materials. [28] Polyfunctionalized benzopyrans have numerous applications, including cosmetics, pigments, and biodegradable agrochemicals. [29] The synthesis of 4H benzo[b]pyran derivatives is important in medical and organic chemistry because of their wide range of uses. The easiest method for synthesizing these chemicals is a one-pot three-component condensation of an aldehyde, malononitrile, and dimedon under varying circumstances. A number of catalysts, including Na_2SeO_4, [30] hexadecyldimethylbenzyl ammonium bromide, [31] NaBr, [32] tetramethyl ammonium hydroxide $(CH_3)_4N^+OH^-$, [33] TEBA, [34] KF montmorillonite, [35] KF alumina, [36] Organocatalysts, [37] Acetic Acid, [38] Diammonium Phosphate [39] and hexadecyltrimethyl ammonium bromide [40] were utilized in synthesis.

In continuation of our inquiry to expand greener, sustainable methods [41–43], we present the microwave assisted synthesis of 4H benzo[b]pyrans employing 1-ethyl-3-methylimidazolium hydrogen sulphate as an effective catalyst. (Scheme 1).



1 **2(a-l)** **3** **4 (a-l)**

Scheme **1**: [BMIM][HSO_4] catalyzed microwave assisted synthesis of Tetrahydrobenzo [b] Pyrans (**4a-l**)

II. RESULTS AND DISCUSSIONS

2.1 Optimization of Reaction Conditions:

To optimize the reaction conditions, the model substrate benzaldehyde was used. The reaction was carried out at room temperature in an ethanol solvent without the addition of a catalyst, and the reaction profile revealed no progress or product production even after 24 hours (Table 1, entry 1). The same reaction was planned under reflux conditions, which resulted in trace product production after 24 hours as shown by the appearance of a new spot on TLC (Table 1, entry 2). In the following experiment, we performed two parallel reactions in ethanol with 150 mg [BMIM][HSO_4] catalyst at RT and reflux conditions. The former exhibited no significant improvement in 24 hours, but the latter showed 80% product production in 1 hour (Table 1, entries 3 and 4). In subsequent experiments, we explored reactions under microwave irradiation at different abilities under solvent-free circumstances.

In the absence of the [BMIM][HSO_4] catalyst, trace product production occurred (Table 1, entry 5). In the presence of the [BMIM][HSO_4] catalyst, the reaction produced 62, 68, and 78% of the product in 15 minutes at 140, 210, and 240 W, respectively (Table 1, entries 6, 7, and 8). The use of 150 mg of [[BMIM][HSO_4] catalyst at 240 W power under solvent-free conditions produced 84% of the isolated product in just 10 minutes, whereas the identical reaction conditions and ethanol solvent yielded only 62% of the product (Table 1, entries 10 and 11). Increasing the reaction duration and catalyst quantity had no meaningful influence on the reaction yield.

Table 1 Optimization of reaction conditions

Entry	Catalyst	Reaction condition	Time	Isolated Yields (%)
1	No catalyst	Stirring at RT	24 h	NR
2	No catalyst	Reflux	24 h	Trace

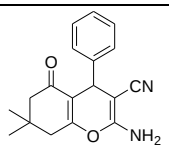
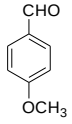
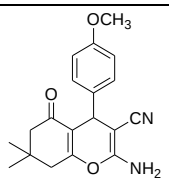
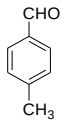
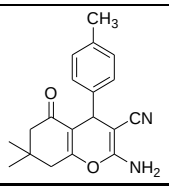
International Journal of Pharmaceutical Research and Applications
Volume 10, Issue 1 Jan - Feb 2025, pp: 1357-1363 www.ijprajournal.com ISSN: 2456-4494

3	150 mg [BMIM][HSO_4]	Stirring at RT	24 h	Trace
4	150 mg [BMIM][HSO_4]	Reflux	1 h	80
5	No catalyst	MW irradiation at 240 W	20 min.	Trace
6	100 mg [BMIM][HSO_4]	MW irradiation at 140 W	15 min.	62
7	100 mg [BMIM][HSO_4]	MW irradiation at 210 W	15 min.	68
8	100 mg [BMIM][HSO_4]	MW irradiation at 240 W	15 min.	78
9	150 mg [BMIM][HSO_4]	MW irradiation at 210 W	15 min.	70
10	150 mg [BMIM][HSO_4]	MW irradiation at 240 W	10 min	84
11	150 mg [BMIM][HSO_4]	MW irradiation at 240 W	10 min	62

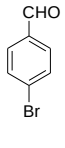
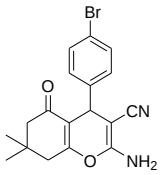
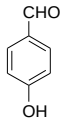
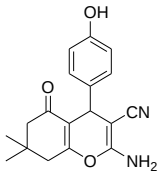
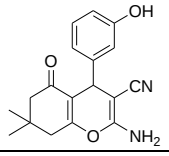
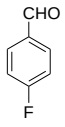
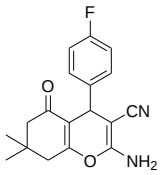
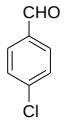
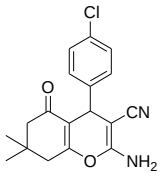
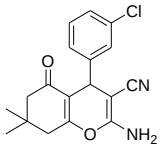
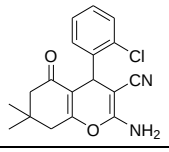
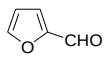
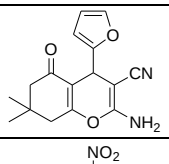
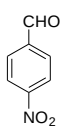
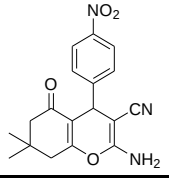
Reaction Condition: Bezaldehyde (1.0 mmol), malononitrile (1.0 mmol), dimedon (1.0 mmol) and [BMIM][HSO_4] 150 mg, solvent free, 240 W.

Following the optimization of reaction conditions, the substrate capacity was investigated further with a variety of aromatic aldehydes containing electron withdrawing and donating groups. (Table 2).

Table 2: Synthesis of Benzo[b]Pyran derivatives:

Entry	Aldehyde	Product	Time (Min.)	[a]Yield (%)	M. P. °C	
					In this work	Literature
4a			10	84	226- 228	228- 230
4b			20	87	196- 198	199- 201
4c			10	90	214- 215	214- 216

International Journal of Pharmaceutical Research and Applications
Volume 10, Issue 1 Jan - Feb 2025, pp: 1357-1363 www.ijprajournal.com ISSN: 2456-4494

4d			10	88	204- 207	196- 198
4e			15	84	204- 205	206- 208
4f			10	87	233-236	236- 238
4g			10	86	190- 192	192- 194
4h			10	92	210- 212	209- 211
4i			10	96	228- 230	226- 229
4j			10	90	217- 218	213- 215
4k			15	78	216- 218	218- 220
4l			20	82	177- 179	177- 178

Reaction Condition: aryl/hetero aryl aldehyde (1.0 mmol), malononitrile (1.0 mmol), dimedone (1.0 mmol) and [BMIM][HSO_4] 150 mg, solvent free, 240 W, a: Isolated yields.

In another modification, comparing the current ionic liquid catalyst to other known catalysts in the literature for the synthesis of Benzo[b]Pyran derivatives (Table 3) demonstrates

International Journal of Pharmaceutical Research and Applications
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that our current inquiry using the [BMIM][HSO_4] catalyst is more capable than existing approaches.

Table 3. Catalyst comparison result for scheme 1 using ionic liquid with other known catalyst in the literature

Entry	Catalyst	Conditions	Solvent	Yield (%)	Ref.
1	Na_2SeO_4	Reflux, 0.6- 3 h	EtOH:H_2O	80- 98	30
2	HDMBAB	80-90°C, 6 h	H_2O	84- 96	31
3	TMAH	RT, 0.5- 2 h	H_2O	79- 93	33
		50°C, 1.5- 5 h	H_2O:EtOH		
4	KF-Al_2O_3	RT, 1- 3 h	DMF	60- 89	36
5	N-Methylimidazole	RT, 60- 420 min.	EtOH 95%	83- 98	37
		RT, 90- 780 min.	H_2O	87- 97	
		Grinding	SF	87- 96	
6	$(NH_4)_2HPO_4$	RT, 0.5- 2 h	H_2O	78- 97	39
		50°C, 1.5- 5 h	EtOH:H_2O	78- 94	
7	[BMIM][HSO_4]	MW 10- 20 min.	SF	78- 96	Present work

2.2 Spectral Data:

4a. 2-amino-5,6,7,8-tetrahydro-7,7-dimethyl-5-oxo-4-phenyl-4H-chromene-3-carbonitrile

M.P. 226-228°C, FTIR (cm^{-1}): 3328, 3197, 2966, 2199, 1652, 1603, ^{1}HNMR (500 MHz, DMSO-d_6) : 0.96 (3H, s, CH_3), 1.04 (3H, s, CH_3), 2.09-2.12 (1H, d, J = 16 Hz, CH), 2.24-2.27 (1H, d, J = 16 Hz, CH), 2.48-2.56 (2H, m, CH_2), 4.18 (1H, s, CH), 7.01 (2H, bs, NH_2), 7.14-7.20 (3H, m, ArH), 7.27-7.30 (2H, m, ArH), ^{13}CNMR (100 MHz, DMSO-d_6) : 27.27, 28.87, 32.28, 36.05, 50.44, 58.77, 113.21, 120.20, 127.04, 127.62, 128.80, 145.22, 158.96, 162.96, 196.12.

4b. 2-amino-5,6,7,8-tetrahydro-4-(4-methoxyphenyl)-7,7-dimethyl-5-oxo-4H-chromene-3-carbonitrile

M.P. 196-198°C, FTIR (cm^{-1}): 3373, 3178, 2833, 2198, 1682, 1605, ^{1}HNMR (500 MHz, DMSO-d_6) : 0.95 (3H, s, CH_3), 1.03 (3H, s, CH_3), 2.08-2.11 (1H, d, J = 16 Hz, CH), 2.23-2.26 (1H, d, J = 16 Hz, CH), 2.46-2.54 (2H, m, CH_2), 3.71 (3H, s, OCH_3), 4.13 (1H, s, CH), 6.83-6.85 (2H, d, J = 8.4 Hz, ArH), 6.96 (2H, bs, -NH_2), 7.05-7.06 (2H, d, J = 8.4 Hz, ArH), ^{13}CNMR (100 MHz, DMSO-d_6) : 27.24, 28.89, 32.26, 35.23, 50.48, 55.46, 59.03, 113.47, 114.14, 120.28, 128.69, 137.33, 158.39, 158.88, 162.61, 196.13.

4e. 2-amino-5, 6, 7, 8-tetrahydro- 4-(4-hydroxypheny l)-7, 7-dimethyl-5-oxo-4H-chromene-3-carbonitrile

M.P. 204-205°C, FTIR (cm^{-1}): 3476, 3309, 2962, 2200, 1654, 1590, ^{1}HNMR (500 MHz, DMSO-d_6) : 0.96 (3H, s, CH_3), 1.04 (3H, s, CH_3), 2.09-2.12 (1H, d, J = 16 Hz, CH), 2.24-2.27 (1H, d, J = 16 Hz, CH), 2.48-2.53 (2H, m, CH_2), 4.18 (1H, s, CH), 7.01 (2H, bs, NH_2), 7.14 (1H, s, OH), 7.15-7.20 (2H, m, ArH), 7.27-7.30 (2H, m, ArH), ^{13}CNMR (100 MHz, DMSO-d_6) :27.22, 28.88, 32.25, 35.16, 50.50, 59.21, 113.65, 115.45, 120.36, 128.61, 135.64, 156.45, 158.86, 162.46, 196.13.

4k. 2-amino-4-(furan-2-yl)-5,6,7,8-tetrahydro-7,7-dimethyl-5-oxo-4H-chromene-3-carbonitrile

International Journal of Pharmaceutical Research and Applications
Volume 10, Issue 1 Jan - Feb 2025, pp: 1357-1363 www.ijprajournal.com ISSN: 2456-4494

M.P. 216-218°C, FTIR (cm^{-1}): 3392, 3214, 2879, 2195, 1656, 1602, ^{1}HNMR (500 MHz, DMSO-d_6) : 0.99 (3H, s, CH_3), 1.05 (3H, s, CH_3), 2.16-2.19 (1H, d, J = 16 Hz, CH), 2.27-2.30 (1H, d, J = 16 Hz, CH), 2.43-2.55 (2H, m, CH_2), 4.33 (1H, s, CH), 6.05-6.06 (2H, d, J = 3 Hz, ArH), 6.32-6.33 (2H, dd, J = 1.85 & 3 Hz), 7.08 (2H, bs, NH_2), 7.48-7.49 (1H, m, ArH), ^{13}CNMR (100 MHz, DMSO-d_6) : 27.02, 28.90, 29.45, 32.29, 50.36, 55.85, 105.53, 110.83, 110.92, 120.03, 142.22, 156.19, 159.78, 163.73, 195.89.

III. EXPERIMENTAL

3.1 Chemicals and Apparatus:

MW-assisted reactions were performed in a RAGA scientific microwave (700W) synthesis apparatus. Melting points were measured in open capillaries and are uncorrected. IR spectra were obtained using a PerkinElmer Fourier-transform infrared (FT-IR) spectrophotometer. 1H NMR spectra were recorded on a Bruker Avance II 500-MHz instrument, whereas 13C NMR spectra were obtained at 100 MHz. To ensure compound purity, precoated silica gel aluminum plates were subjected to thin layer chromatography (TLC). [BMIM][HSO4] catalyst from Sigma-Aldrich was utilized.

3.2 Microwave Assisted Synthesis of tetrahydrobenzo[b]pyran derivatives, 4(a-l):-

A 50 ml RBF included a combination of aldehyde (1 mmol), malononitrile (1 mmol), dimedon (1 mmol), and 150 mg [BMIM][HSO4] catalyst. The reaction mixture was exposed to MW irradiation at 240 W for the appropriate time (Table 2) under solvent-free conditions. The development of the reaction was tracked using TLC. After the reaction, the mixture was cooled to room temperature, the reaction content was poured over crushed ice, and the solid result was filtered. The crude product was dried and recrystallized from ethanol, yielding pure crystals of tetrahydrobenzo [b] pyran derivatives in good quantities.

IV. CONCLUSIONS

We created a variety of various substituted tetrahydrobenzo[b]pyran derivatives utilizing [BMIM][HSO4] as a catalyst in a solvent-free cyclocondensation of aldehyde, dimedon, and malononitrile. It has been discovered that [BMIM][HSO4] is the most effective catalyst for MW aided synthesis, providing various advantages. This green approach has a simple workup procedure, a rapid reaction time, and great yields. Furthermore, the appealing MW-assisted synthesis makes this protocol more ecologically friendly and contributes significantly to green chemistry.

SUPPLEMENTARY MATERIAL

Supporting documentation is available: spectral analysis data (MS-Word).

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