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ULTRASOUND-ASSISTED ONE-POT SYNTHESIS OF 3-AROYLIMIDAZO[1,2-A] PYRIDINES DERIVATIVES USING A THREE-COMPONENT METHOD IN A GREEN SOLVENT

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ABSTRACT
The integration of ultrasound technology with three-component synthesis strategies is a notable advancement in the formation of 3-aroylimidazo[1,2-a] pyridine derivatives. The utilisation of green solvents, such as glycerol and dimethyl carbonate, enhances the environmental sustainability of these processes while maintaining a high synthetic efficiency. The ongoing development of these methodologies holds promise as valuable tools for both academic research and industrial applications in the synthesis of bioactive molecules. Future research should focus on expanding the substrate scope, developing additional metal-free catalytic systems, and optimising reaction conditions for maximum sustainability and efficiency. This method employs aldehydes, acetophenone, and 2-aminopyridine under mild conditions, offering high yields, operational simplicity, and environmental sustainability. Structural characterisation was performed using NMR and mass spectrometry.
Keywords: One-pot Synthesis, MCR Reaction, 3-aroylimidazo[1,2-a] Pyridine, Sonochemistry, Green Chemistry
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INTRODUCTION

3-aroylimidazo[1,2-a] pyridines derivatives have gained attention owing to their wide range of pharmacological properties, including antimicrobial, anti-inflammatory, antioxidant, and anticancer activities. The presence of a heterocyclic ring in a single molecular framework enhances their bioactivity and makes them promising candidates for drug discovery. MCR has a hybrid tool for organic synthesis, with its improvement of atom economy, operational simplicity, and high efficiency. One-pot, three-component strategies are particularly attractive because they enable the rapid construction of complex molecular architectures using simple, readily available precursors. In recent years, green solvents with sonochemistry have gained prominence as a green and sustainable approach for facilitating organic transformations under mild conditions. The named sonochemical condition represents a novel advancement in this domain, enabling the synthesis of heterocyclic scaffolds with improved reaction efficiency and selectivity. The present study focuses on the development of a novel and efficient one-pot MCR synthetic approach for the construction of aroylimidazo derivatives via sonochemical reaction. This methodology utilizes Aldehyde, Acetophenone and 2-amino pyridine derivatives under reduce conditions, leading to superb yields of specific products. The reaction mechanism involves a Sonochemically driven cascade process, which enhances the efficiency and selectivity of the transformation. To ensure the structural integrity of the synthesised compounds, extensive characterisation was performed using spectroscopic techniques such as NMR and mass spectrometry. This research contributes to the ongoing efforts in sustainable heterocyclic synthesis by providing a practical and eco-friendly strategy for the development of bioactive molecules.

EXPERIMENTAL

Chemicals and Reagents

All chemicals and reagents used in this study were of analytical grade and were obtained from commercial suppliers without further purification. Aldehydes, Acetophenone and 2-Aminopyridine were purchased from Sigma-Aldrich and used as received. The solvents used, including ethanol, Ethyl acetate, n-hexane, DMC, and glycerol, were of high-purity grade.

Synthesized Compounds: Instrumentation and Characterization

The synthesised compounds were characterised using various spectroscopic techniques. NMR spectra

were obtained using a Bruker Avance 600 MHz spectrometer for both 1H and 13C NMR(Sathi-BHU), and a mass spectrometer (Private Testing Lab).

General Procedures

Synthesis of 3-aroylimidazo[1,2-a] pyridines Derivatives

Preparation of Green Solvent Composite

Take 100 ml RBF and take Glycerol and Dimethyl Carbonate (DMC) in the desired proportions. The amount of each solvent used depends on the desired viscosity, solubility, and application. Typically, a ratio of 1:1 (Glycerol: DMC) is the starting point, but adjustments can be made based on the nature of the materials used. The two solvents were then mixed by stirring at room temperature. If necessary, the mixture was gently heated to aid the dissolution process (maintaining the temperature below 60°C to avoid excessive evaporation of DMC). and make in various ratios like (3:7), (5:5), (7:3).

General Procedure for Preparation

Synthesis In a 25 mL round-bottom flask, a mixture of Acetophenone (0.288 g), benzaldehyde (0.216 g), 2-amino pyridine (0.242 g), and green solvent underwent ultrasonic irradiation at various temperatures, monitored by TLC. After completion and cooling to room temperature, the reaction mass either formed a solid (filtered and washed with chilled ethyl acetate) or remained liquid. The mixture was extracted with EtOAC (3 × 30 mL), separating the upper EtOAC layer from the green solvent layer. The EtOAC layer was washed with distilled water (3 x 10 mL), dried over Na2SO4, and concentrated via rotary evaporator. The product was purified by crystallization using hot ethanol and characterized using 1H, 13C NMR and Mass spectroscopy.

Recovery of Solvent

The reaction mass was filtered under vacuum conditions, and the receiver was chilled and then washed with n-hexane to recover the solvent and remove n-hexane at room temperature. If any impurity or product spot was not observed in the recovered solvent, then it was processed for the next cycle, and up to three cycles obtained a constant yield.

RESULTS AND DISCUSSION

DSC Graph

The DSC thermogram (Fig.-1) displays the thermal transitions of the analysed sample, revealing both endothermic and exothermic events.

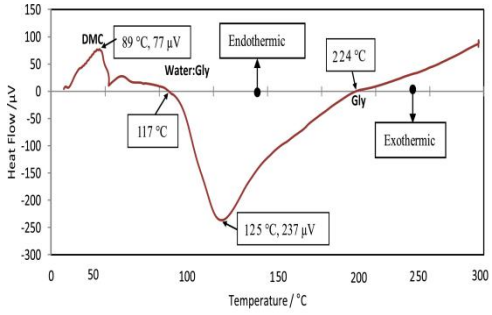


Fig.-1: (DSC graph of green solvent composite)

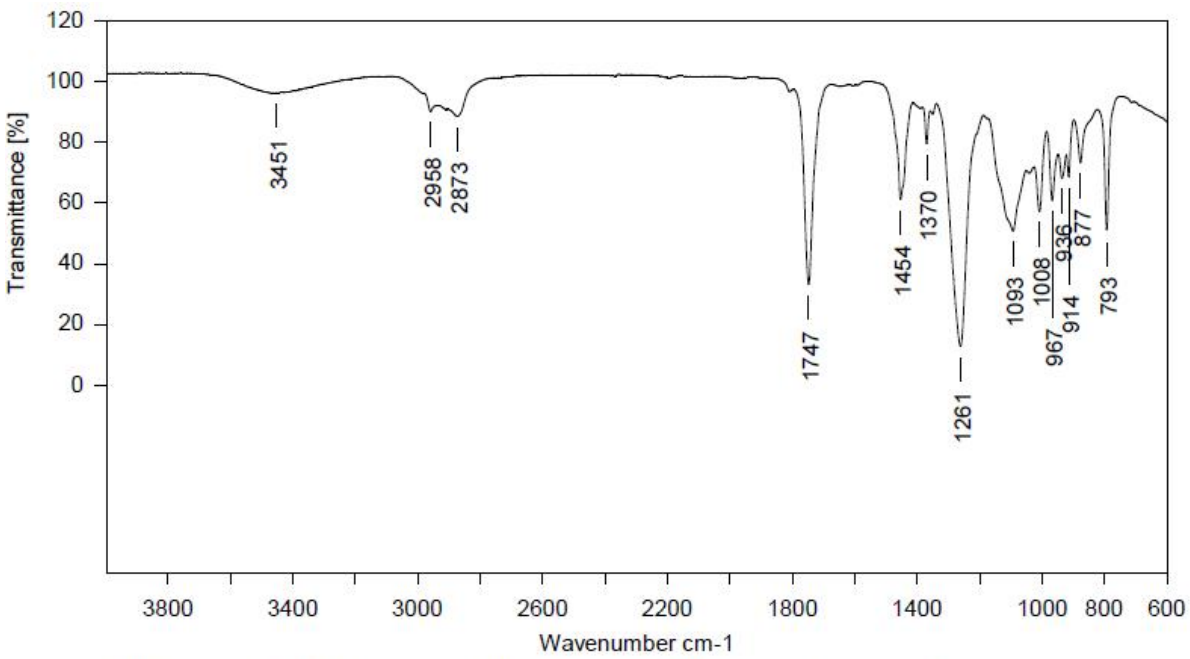
Initial Thermal Events (Up to ~100°C)

The first thermal transition occurs at 89°C (77 µV), corresponding to the desorption of DMC (Dimethyl Carbonate), which likely indicates the removal of residual solvent or physically adsorbed species. A second peak is observed at 117°C, attributed to the evaporation of water-glycerol mixtures, suggesting moisture loss or weakly bound solvent removal.

Endothermic Transition (~125°C)

A significant endothermic peak appears at 125°C (237 µV), indicating a possible phase transition, melting, or decomposition of a particular component. This is likely associated with the breakdown of an

intermediate phase. High-Temperature Transitions (Above 200°C): At 224°C, another thermal event is recorded, which can be related to the degradation of glycerol -based components. An exothermic event is observed beyond 224°C, signifying a possible crystallisation or structural reorganisation of the sample. The DSC analysis reveals multiple thermal transitions associated with solvent evaporation, phase changes, and decomposition. The strong endothermic peak at 125°C suggests a crucial transformation, while the exothermic event at higher temperatures indicates further material modifications. These findings provide valuable insight into the thermal stability and composition of the sample.



FTIR spectrum of GLY-01 provides valuable information about the functional groups present in the sample, helping to identify the potential components of the material.

1. 3451cm-1 (Broad Peak): This is a broad peak, often indicative of O-H stretching vibrations. This suggests the presence of hydroxyl groups.(New peak observed, it's not present in DMC and Glycerol)
2. 2958 cm-1 and 2873 cm-1 (Sharp Peaks): These peaks are likely due to C-H stretching vibrations in alkanes or alkyl groups.
3. 1747 cm-1 (Sharp Peak): This peak suggests a C=O stretching vibration, possibly indicating the presence of a carbonyl group (Same as DMC Graph peak value 1746cm-1).
This all above FT-IR and DSC graph indicates the new type solvent formation it's high boiler than DMC and low boiler than glycerol .

Fig.-2: Represent FTIR Graph of GLY+DMC

FTIR spectroscopy successfully identified key functional groups within the Glycerol sample. The presence of O-H, C-H, and C=O bonds suggests a complex organic structure.

Table-1: Solvent Optimisation Study

Entry	Glycerol	Other solvent	*T* [ºC]	Sonication Method		Conventional Method	
				Sonication yield	(%) Recovery of solvent	Conventional yield	(%) Recovery of solvent
1	glycerol	--	120	45	95	25	90
2	glycerol	Water (5:5)	90	62	93	27	85
3	glycerol	DMC (5:5)	80	89	80	75	80
4	glycerol	Ethanol (5:5)	75	67	83	60	79
5	glycerol	Methanol (5:5)	60	70	82	62	75

6	glycerol	PEG-400	110	45	97	29	90
7	glycerol	IPA	75	49	81	41	72
8	glycerol	Diethyl carbonate	80	62	80	42	76
9	glycerol	DMC (6:4)	70	62	80	41	74
10	glycerol	DMC (7:3)	70	65	80	54	76
11	glycerol	DMC (8:2)	90	66	72	57	69
12	glycerol	DMC (4:6)	65	64	75	55	72
13	glycerol	DMC (3:7)	72	69	76	67	71
14	glycerol	DMC (1:9)	75	70	75	65	69
15	glycerol	DMC (5:5) (10 vol)	60	89	85	75	80
16	glycerol	DMC (5:5) (1 vol)	80	10	80	7	75
17	glycerol	DMC (5:5) (4 vol)	60	91	87	79	85
18	glycerol	DMC (5:5) (7 vol)	60	90	86	74	80
19	glycerol	DMC (5:5) (9 vol)	60	91	87	79	85
20	glycerol	DMC (5:5) (4 vol)	70	90	80	78	72
21	glycerol	DMC (5:5) (4 vol)	60	91	86	80	85

The optimisation study focuses on evaluating the effect of different solvent systems on reaction yield and solvent recovery using Glycerol as the primary medium. The study systematically compares the efficiency of the sonochemical method with conventional heating to determine the best reaction conditions. The results indicate that glycerol provides moderate yields, but the addition of co-solvents significantly enhances reaction efficiency. Among the various solvent combinations, glycerol with dimethyl carbonate (DMC) in a 5:5 ratio at 80°C under sonication yielded the highest conversion (89%) with good solvent recovery (80%) (Table-1, entry 03). Further optimisation of the solvent ratio and temperature selection confirmed at 600 °C temperature, and 4 volume solvent optimisation was finalised for the reaction. (Table-1, entry 17,21). If temperature parameters increase, then the same yield is obtained, but there is an impact on solvent recovery (Table-1, Entry 20). The improved performance in sonication is attributed to cavitation effects, which enhance mass transfer, accelerate reaction rates, and facilitate better interaction between reactants, leading to further optimisation of the reaction. Temperature optimisation further revealed that reactions conducted between 60–65°C offered the best balance between high yield and solvent stability, while higher temperatures led to decreased solvent recovery. A comparative analysis of the two methods demonstrated that sonochemical synthesis not only improves yield but also reduces reaction time and energy consumption, making it a more sustainable and efficient alternative. The findings support the potential of using Glycerol -based solvent systems for green and high-yielding reactions, particularly when combined with optimised sonication parameters.

Under ultrasonic conditions, the reaction yields are based on the nature of the substituted aromatic groups (R1, R2, R3), indicating the influence of electronic and steric effects on product formation. The highest yield (88%) was observed when both R_2 and R_3 were unsubstituted phenyl groups, suggesting that sterically unhindered systems favour better conversion. Electron-withdrawing substituents, such as p-nitrophenyl (-NO_2), facilitated high yields (86%), likely due to increased electrophilicity, which enhances reactivity.

Table-2: The Comparison of Green Solvent Under Ultrasound Method (Glycerol: DMC) (1:1) with Previously Reported Catalyst and Conditions

Entry	Reagent	Conditions	Time (min)	Yield (%)[a]	Ref.
1	$FeBr_3$	Toluene,110°C (Inert condition)	960	89	25
2	I2,AlCl3	Chlorobenzene	960	82	28

3	$CuCl2.2H_2O$	Toluene /Reflux	720	86	26
4	TFA	120/CHCl3	840	98	27
5	$RuCl_3·H_2O/I_2$	110/Toluene	720	86	11
6	CuI/BF3Et2O	Neat/40°C	1440	70	30
7	Sno2/I2	110/Tolune	720	84	29
8	$CuFe_2O_4/I_2$	140/1,4-Dioxane	420	75	33
9	Catalyst free	RT/DCM	240	80	31
10	Green solvent composite	60/Sonication	70	91	This work

[a] Isolated yield.

Similarly, the presence of electron-donating groups like p-methoxy ($-OCH_3$) or p-hydroxy (-OH) resulted in moderate to high yields (73–82%), indicating their role in stabilising reaction intermediates.

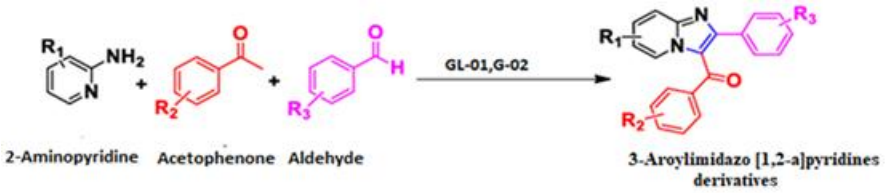


Table-3: Substrate Scope of Green Solvent Mixture Under Ultrasound Method Reaction of Aroylimidazo Derivatives

Code	R2 Acetophenone	R1 pyridine	R3 aldehyde	Yield (%)/ Time(Min)
PP-01	Acetophenone	2-amino Pyridine	Benzaldehyde	88/60
PP-02	Acetophenone	2-amino Pyridine	4-Nitro Benzaldehyde	86/45
PP-03	Acetophenone	2-amino Pyridine	3,4 Di-Methoxy Benzaldehyde	78/70
PP-04	Acetophenone	2-amino Pyridine	4-methoxy Benzaldehyde	73/70
PP-05	Acetophenone	2-amino Pyridine	4-Hydroxy Benzaldehyde	82/45
PP-06	4-Chloro Acetophenone	2-amino Pyridine	Benzaldehyde	68/70
PP-07	4-Fluoro Acetophenone	2-amino Pyridine	Benzaldehyde	62/70
PP-08	4-Methoxy Acetophenone	2-amino Pyridine	Benzaldehyde	57/70
PP-09	Acetophenone	2-amino Pyridine	4-Chloro Benzaldehyde	65/70
PP-10	Acetophenone	2-amino Pyridine	4-Fluro Benzaldehyde	59/70
PP-11	Acetophenone	2-amino Pyridine	Furanaldehyde	61/70
PP-12	Acetophenone	2-amino Pyridine	4-Bromo Benzaldehyde	83/45
PP-13	Acetophenone	2-amino Pyridine	4-ethoxy Benzaldehyde	78/45
PP-14	Acetophenone	2-amino Pyridine	4-ethoxy,3-hydroxy Benzaldehyde	65/70

In contrast, halogen-substituted systems exhibited varied yields, with p-chlorophenyl and p-bromophenyl derivatives producing 65% and 83% yields, respectively. The slightly lower yield for p-fluorophenyl (59%) suggests that the electronegativity of fluorine may slightly hinder the reaction progression. Pyridine ($-C_5H_4N$) substitution was also found to influence the reaction efficiency, with yields ranging from 57% to 79%, depending on the nature of the co-substituent. The lowest yield (57%) was observed for p-methoxyphenyl combined with pyridine, potentially due to steric hindrance or electronic effects

affecting nucleophilic attack. Overall, ultrasonic irradiation significantly enhanced reaction rates and yields by promoting better mass transfer and energy input, facilitating efficient bond formation. The results suggest that electron-withdrawing groups generally improve reaction efficiency, while steric hindrance and electronic factors play a crucial role in determining the final product yield.

Structure Confirmation by Spectroscopy Analysis

PP-01: ^{1}H NMR: δ 7.26 (1H), 7.37-7.81 (11H,), 7.98 (1H), 8.86 (1H). ^{13}C NMR: δ 114.0, 117.2, 123.8, 125.8, 127.7 (2C), 128.0, 128.3-128.5 (3C), 128.6 (2C), 129.1 (2C), 131.0, 131.9, 138.3, 141.6, 145.8, 184.9. Mass spectra:301.21

PP-02: ^{1}H NMR: δ 7.41-7.82 (7H), 7.94-8.10 (3H), 8.22 (2H), 8.98 (1H).^{13}C NMR: 62.9,72.9,110.4,115.5,123.6-123.8(3C),128.1(2C),128.4-128.5(4C),130.0,131.9,137.0,137.2,142.4,147.3,157.5, Mass spectra:345.14

PP-03: ^{1}H NMR: δ ^{1}H NMR: δ 3.67-3.83 (6H), 6.93 (1H), 7.11-7.25 (2H), 7.41-7.74 (7H), 7.86 (1H), 8.91 (1H).^{13}C NMR: δ 55.9(2C), 110.9,114.0 , 114.5 (2C), 117.2 , 123.8 , 125.8 , 128.0 , 128.3 (2C), 130.1 (2C), 131.2 , 131.0 , 138.3 , 141.6 , 145.8 ,148.3 ,152.9,164.9 ,.Mass Spectra:359.13

PP-04:^{1}H NMR: δ 3.86 (3H), 6.96 (2H), 7.25 (1H), 7.37-7.67 (7H),7.74 (1H), 7.97 (1H), 8.85 (1H).^{13}C NMR: δ 55.3, 113.7 (2C), 114.0, 117.2, 123.8, 125.8, 127.3, 127.7 (2C), 128.4, 128.6 (2C), 131.0, 131.6 (2C), 133.3, 141.6, 145.8, 162.4, 164.9.

PP-05:^{1}H NMR: δ 6.88 (2H), 7.25 (1H), 7.37-7.67 (7H),7.74 (1H), 7.97 (1H), 8.85 (1H)^{13}C NMR: δ 114.0, 115.3 (2C), 117.2, 123.8, 125.8, 127.3, 127.7 (2C), 128.4, 128.6 (2C), 130.4 (2C), 131.0, 133.3, 141.6, 145.8, 160.9, 164.9.

PP-06:^{1}H NMR: δ 7.25 (1H), 7.37-7.67 (7H), 7.68-7.88 (3H), 7.97 (1H), 8.86 (1H).^{13}C NMR: δ 114.0, 117.2, 123.8, 125.8, 127.3, 127.7 (2C), 128.4, 128.5-128.8 (4C),130.4 (2C), 131.0, 133.3, 138.5, 141.6, 145.8, 164.9.

PP-07:^{1}H NMR: ^{1}H NMR: δ 7.25 (1H), 7.35-7.67 (7H), 7.68-7.84 (3H), 7.97 (1H), 8.85 (1H).^{13}C NMR: δ 114., 117.2, 123.8, 125.8, 127.3, 127.7 (2C), 128.4, 128.5-128.8 (4C), 130.4 (2C), 131.0, 133.3, 138.5, 141.6, 145.8, 164.9.

PP-08:^{1}H NMR: δ 3.86 (3H), 6.96 (2H), 7.25 (1H), 7.37-7.67 (7H),7.74 (1H), 7.97 (1H), 8.85 (1H)
^{13}C NMR: δ 55.3, 113.7 (2C), 114.0, 117.2, 123.8, 125.8, 127.3, 127.7 (2C), 128.4, 128.6 (2C), 131.0, 131.6 (2C), 133.3, 141.6, 145.8, 162.4, 168.9.

PP-09:^{1}H NMR: δ 7.23 (1H), 7.38-7.78 (10H), 7.97 (1H), 8.85 (1H).^{13}C NMR: δ 114.0, 117.2, 123.8, 125.8, 127.3, 128.3 (2C), 128.9 (2C), 129.1 (2C), 129.4 (2C), 131.0, 131.9, 134.6, 138.3, 141.6, 145.8, 184.9 (1C, s).

PP-10:^{1}H NMR: δ 7.16-7.40 (3H),7.41-7.77 (8H), 7.97 (1H),8.85 (1H).^{13}C NMR: δ 114.0, 116.2 (2C), 117.2, 123.8, 125.8, 127.3, 128.3-128.5 (4C), 129.1 (2C), 131.0, 131.9, 138.3, 141.6, 145.8, 162.3, 168.9.

PP-11:^{1}H NMR: δ 6.51 (1H), 6.89 (1H), 7.11 (1H), 7.41-7.74 (6H), 7.79-7.92 (2H),8.86 (1H).^{13}C NMR: δ 25.7, 28.9, 62.9, 68.2, 69.6, 79.9, 110.4, 115.5, 123.7, 128.4-128.5 (4C), 130.0, 131.9, 137.0, 142.4, 178.5.

PP-12:^{1}H NMR: ^{1}H NMR: δ 7.17-7.34 (3H), 7.41-7.78 (8H), 7.97 (1H), 8.85 (1H).^{13}C NMR: δ 62.9, 72.9, 110.4, 115.5, 121.9, 123.7, 128.1 (2C), 128.4-128.5 (4C),130.0, 131.7 (2C), 131.9, 137.0, 137.2, 142.4, 2015

PP-13:^{1}H NMR: δ 1.29 (3H), 4.09 (2H), 7.05-7.26 (3H), 7.41-7.74 (8H),7.90 (1H), 8.96 (1H).^{13}C NMR: δ 14.7, 62.9, 63.7, 72.9, 110.4, 114.6 (2C), 115.5, 123.7, 128.1 (2C), 128.4-128.5 (4C), 130.0, 131.9, 137.0, 137, 142.0, 158.7, 171.5.

PP-14: ^{1}H NMR: δ 1.25 (3H), 4.07 (2H), 4.80 (2H), 5.011(1H), 5.79 (1H), 5.96 (1H), 6.65-6.85 (2H), 6.94-7.10 (2H), 7.24 (1H), 7.49-7.72 (3H),7.95 (2H).^{13}C NMR: δ 14.8, 62.9, 604.0, 72.9, 110.4, 111.8, 115.5-115.6 (2C), 123.7, 128.1, 128.4-128.5 (4C), 130.0, 131.9, 132.5, 137.0, 142.4, 146.1, 146.7, 189.5.

CONCLUSION

The one-pot, three-component synthesis of 3-aroylimidazo[1,2-a] pyridines represents a significant advancement in green chemistry and sustainable synthetic methodologies. Utilising a green solvent composite and ultrasonic irradiation, the ultrasonic technique accelerates reaction rates and enhances yields through acoustic cavitation, which creates localised high-temperature and high-pressure environments conducive to chemical transformations. The method's operational simplicity, coupled with its ability to produce high yields under mild conditions, makes it particularly attractive for both academic research and industrial applications. The use of NMR and mass spectrometry for compound characterisation ensures precise structural elucidation and purity assessment. Furthermore, the high recovery rate and low toxicity of the solvent system contribute to the overall sustainability of the process, thereby reducing environmental impact and potentially lowering production costs in scaled-up applications. This synthesis method exemplifies the potential for developing more environmentally friendly and efficient processes within the pharmaceutical and fine chemical industries.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-12: Responsible Consumption and Production*. This green chemistry approach promotes sustainable manufacturing by minimizing hazardous solvents, reducing energy via ultrasound, and enabling efficient one-pot reactions. The research profile of the authors can be verified from their ORCID ids, given below:

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