SRI VENKATESWARA INTERNSHIP PROGRAM
FOR RESEARCH IN ACADEMICS
(SRI-VIPRA)
Minor Research Project (Grant)

SRI-VIPRA

Project Report of 2025: SVPG-2501

"Ecofriendly synthesis of biologically significant 3,4-dihydropyridimidine-2(1H)-thiones using Deep Eutectic solvents as green media and catalyst"

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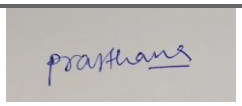
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Sri Venkateswara College
University of Delhi
Benito Juarez Road, Dhaula Kuan, New Delhi
New Delhi -110021

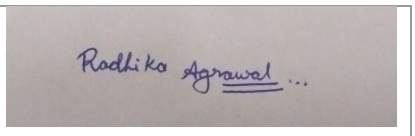
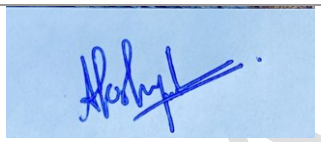
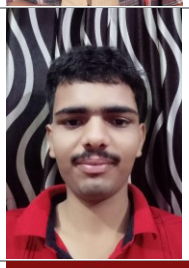
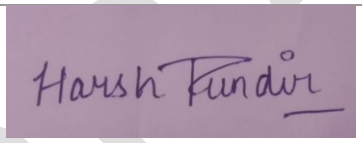
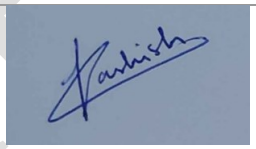
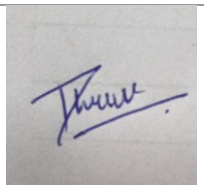
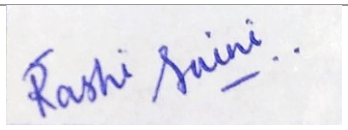
SRIVIPRA Minor Research Project (Grant) 2025

Title : ECO-FRIENDLY SYNTHESIS OF BIOLOGICALLY SIGNIFICANT 3,4-DIHYDROPYRIMIDINE-2(1H)-THIONES USING DEEP EUTECTIC SOLVENTS AS GREEN MEDIA AND CATALYST

Name of Mentor	**:**	**Prof. Sharda Pasricha**	
Name of Department	**:**	**Department of Chemistry**	
Designation	**:**	**Professor**	
Name of Mentor	**:**	**Dr. K. Murali Mohan Achari**	
Name of Department	**:**	**Department of Chemistry**	
Designation	**:**	**Assistant Professor**	

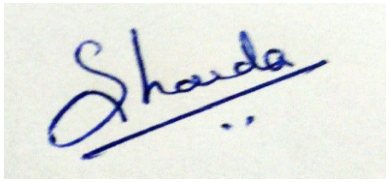
List of students under the SRIVIPRA Minor Research Project

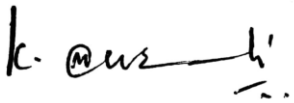
S.No	Photo	Name of the student	Roll number	Course	Signature
1		Prarthana Rohatgi	1122122	B.Sc. Life Sciences	

2		Radhika Agarwal	1122161	B.Sc. Life Sciences	
3		Akshayah M	1123165	B.Sc. Life Sciences	
4		Harsh Pundir	1523027	B.Sc. (Hons) Chemistry	
5		Kashish Mann	1523062	B.Sc. (Hons) Chemistry	
6		Pahi Talukdar	1523045	B.Sc. (Hons) Chemistry	
7		Dhruv Tiwari	1524068	B.Sc. (Hons) Chemistry	
8		Rashi Saini	1524040	B.Sc. (Hons) Chemistry	

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9		Vidhi Aggarwal	1524039	B.Sc. (Hons) Chemistry	





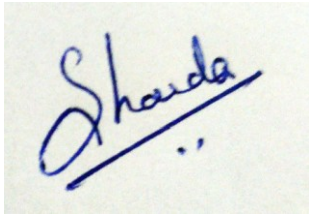
(Prof. Sharda Pasricha) **(Dr. K. Murali Mohan Achari)**

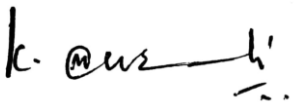
Signatures of Mentors

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Certificate of Originality

This is to certify that the aforementioned students from Sri Venkateswara College have participated in the summer project SVPG-2501 titled **"Eco-Friendly Synthesis of Biologically Significant 3,4-Dihydropyrimidin(thio)ones Using Deep Eutectic Solvents As Green Media And Catalyst"**.The participants have carried out the research project work under my guidance and supervision from 1st July 2025 to 15th February 2026. The work is original and conducted in an online/offline/hybrid mode.





(Prof. Sharda Pasricha) **(Dr. K. Murali Mohan Achari)**

Signature of Mentors

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Acknowledgements

I would like to express my sincere gratitude to my supervisors, **Prof. Sharda Pasricha** and **Dr K. Murali Mohan Achari,** for their invaluable guidance, encouragement, and constant support throughout this research.

I extend my heartfelt thanks to the Principal of Sri Venkateswara College, Prof Vajala Ravi, for their support and for providing the necessary resources to conduct this study. We are particularly grateful for the minor research grant provided, which was instrumental in carrying out this work and achieving our research objectives

I would also like to thank my fellow team members for their collaboration and collective efforts, which were essential to the successful completion of this project. Finally, I am thankful to Sri Venkateswara College for providing me with the opportunity and the necessary resources to conduct this research, providing a valuable platform to learn, grow and contribute to the field of science.

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TABLE OF CONTENTS

S.No	Topic	Page No.
1.	**Introduction** **1.1 Biginelli reaction: An introduction** **1.2 Mechanism** **1.3 Exploring DHPMs and DHPTs**	7-10
2.	**Review of Literature** **2.1 Advances in Biginelli Reaction** **2.2 Catalysts Used in Biginelli Reaction** **2.3 Deep Eutectic Solvents**	11-12
3.	**Materials and Methods** **3.1 Materials** **3.2 Preparation of DES** **3.3 Preparation of DHPTs**	13
4.	**Results and Discussion**	14
5.	**References**	15-16

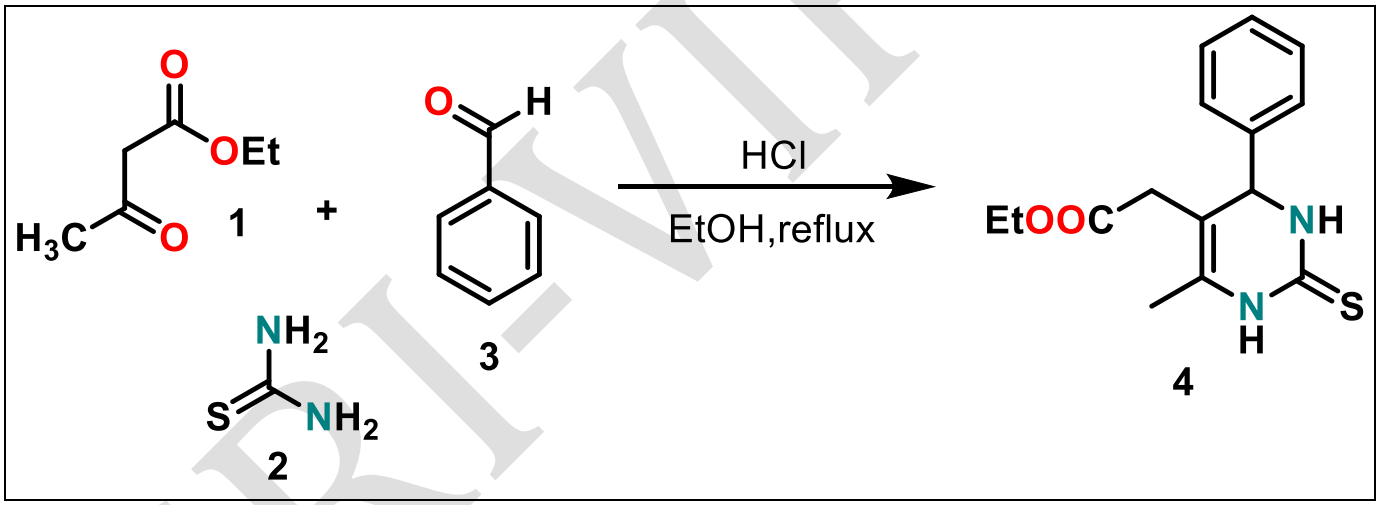
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1. INTRODUCTION

1.1. Biginelli reaction: An introduction

Named after the Italian chemist Pietro Biginelli, the Biginelli reaction is a well-studied multicomponent reaction (MCR) that has attracted significant interest from scientists for its synthetic versatility and potential applications in drug development.[1] MCRs are selective, exhibit synthetic convergence, and are atom-economic, making them instrumental for advancements in synthetic methodology for pharmaceutical and drug discovery research. They offer simple and critical tools to produce drugs with the fewest possible steps.[1]

Biginelli reaction produces dihydropyrimidinones (DHPMs) or Dihydropyrimidin(thio)ones (DHPTs) (**4**) by condensing an aldehyde (**3**), a β-keto ester (**1**), and urea or thiourea (**2**) in the presence of a Bronsted acid catalyst (**Scheme 1**).[2]

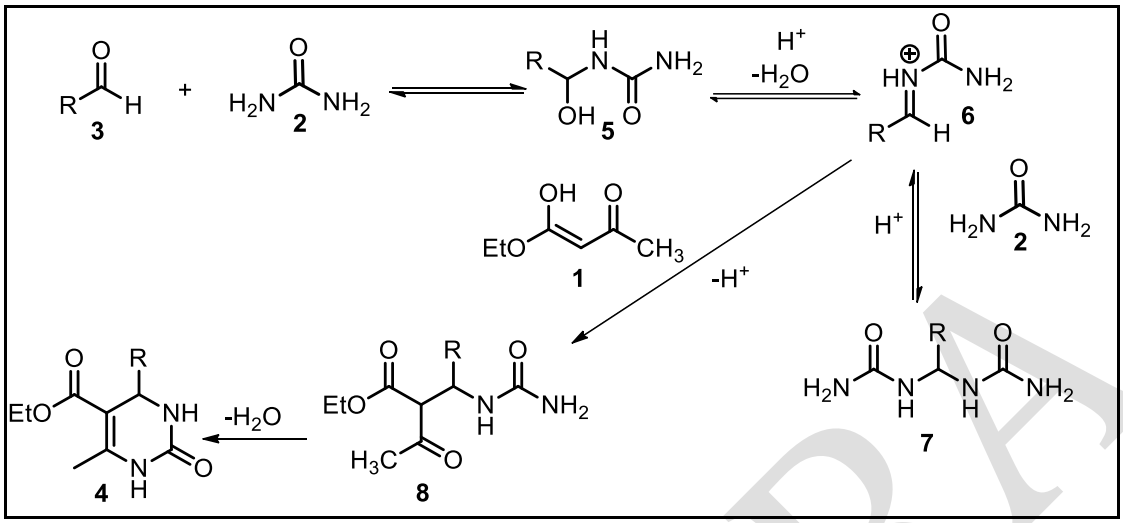


Scheme 1. Classical Biginelli Reaction

1.2. Mechanism

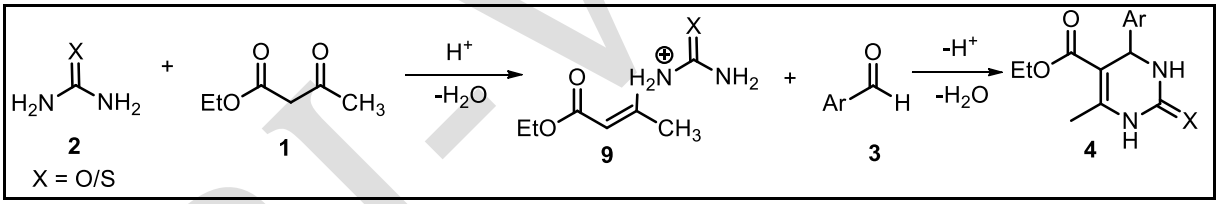
The general mechanism of the Biginelli reaction involves the addition of urea (**2**) to an aldehyde (**3**), leading to the formation of an *N*-(1-hydroxybenzyl) urea intermediate (**6**) through nucleophilic addition (**Scheme 2**). The intermediate then undergoes acid-catalysed dehydration, generating a highly reactive *N*-acyliminium species. Next, a β-dicarbonyl compound (**1**) reacts with the *N*-acyliminium species and followed by cyclisation, resulting in the formation of a dihydropyrimidinone product (**4**) known as the Biginelli product. This concludes the iminium route of Biginelli Reaction.[3]

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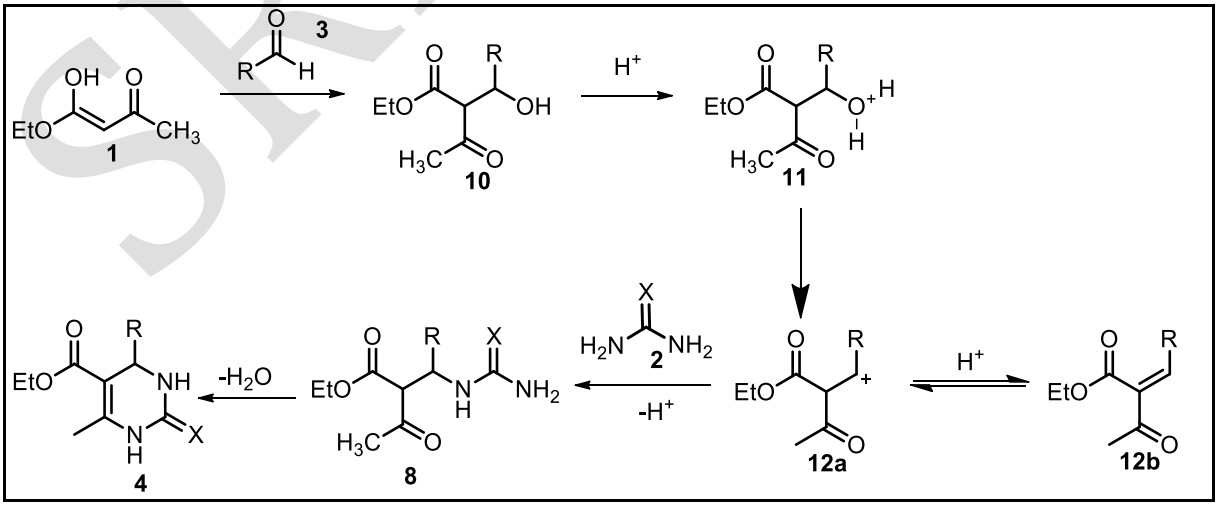


Scheme 2. Iminium Route for the synthesis of DHPM

The enamine route (**Scheme 3**) is another mechanism that involves the condensation of urea (2) and a β-keto ester (1), forming an enamine intermediate (**9**), which further reacts with an aldehyde (**3**) to give rise to the DHPM (**4**). The third route (**Scheme 4**) involves the reaction between an aldehyde (**3**) and a β-keto ester (**1**), yielding a carbenium ion intermediate (**12a**), which reacts with urea (**2**) to form DHPM (**4**). It involves a Knoevenagel-type reaction mechanism.[1]



Scheme 3. Enamine Route



Scheme 4. Knoevenagel Route

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1.3. Exploring DHPMs and DHPTs

3,4-dihydropyrimidin-2(1*H*)-ones (DHPMs) and 3,4-dihydropyrimidin-2(1*H*)-thiones (DHPTs), their sulfur analogues, are versatile heterocyclic scaffolds and the primary product of Biginelli reactions. They are extremely adaptable substances, making them desirable targets for drug development and discovery. [4] Pyrimidine derivatives are involved in several vitamins and are necessary building blocks of DNA and RNA. Since 1980, when it was discovered that DHPMs shared structural similarities with the well-known Hantzsch-type dihydropyridine calcium channel modulators, the Biginelli reaction has undergone significant advancements. Numerous adjustments were made to get around the restrictions, and new techniques were documented in this field.[5]

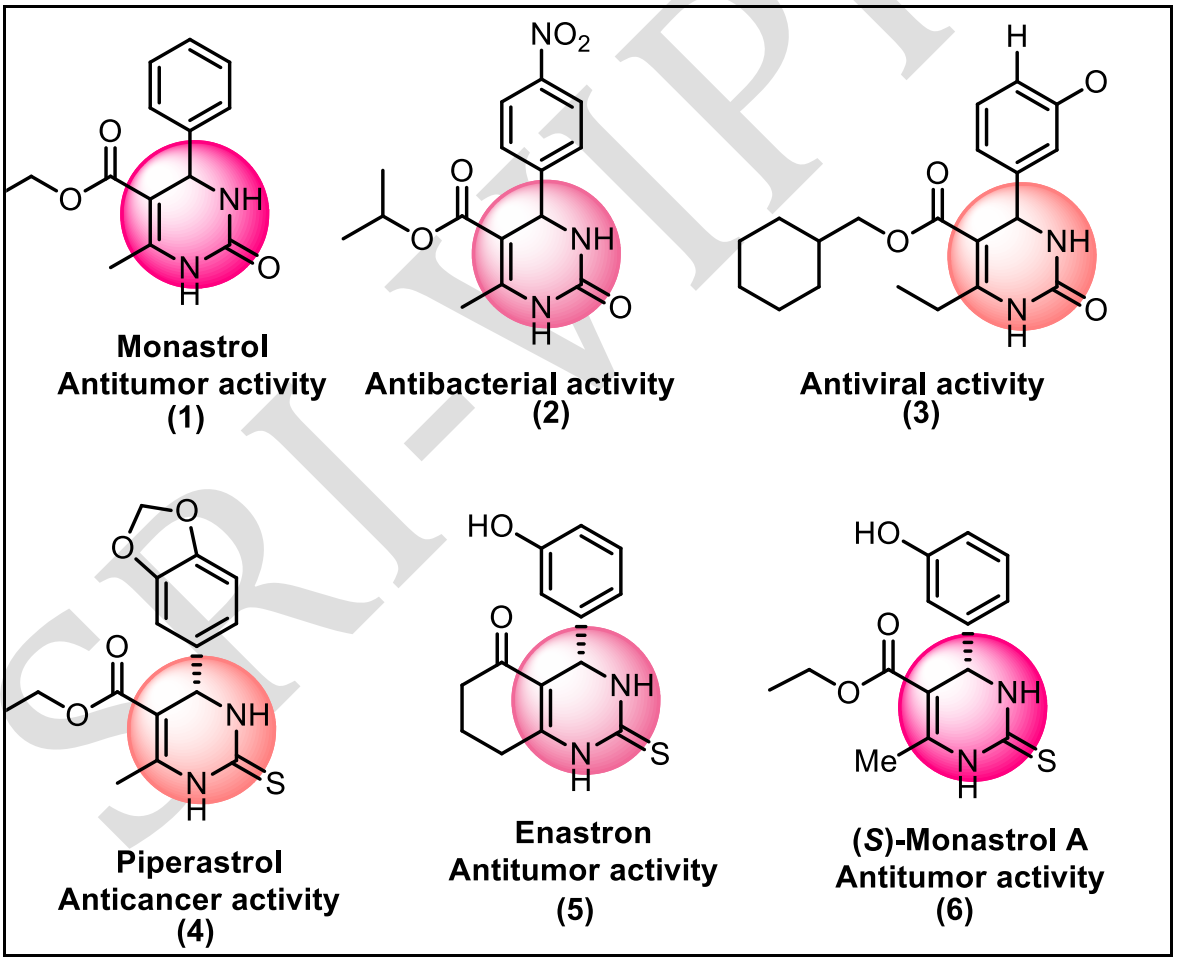


Figure 1. Library of DHPMs and DHPTs

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DHPMs and DHPTs show remarkable biological activity. For example, Monastrol, a DHPT derivative that acts as a mitotic inhibitor, is a lead target for anticancer drug development. Various derivatives also exhibit potent cytotoxicity against several cancer cell lines.[6] Many DHPMs/DHPTs have been reported to exhibit antiviral activity. Some derivatives are potential leads for AIDS therapy. They are also potent against the Trachoma group viruses[4] and show activity against HSV[7] and Hepatitis B Virus HBV.[1] They also show antibacterial and antifungal activity. They also show anti-Alzheimer's, anti-inflammatory, and antioxidant activity (**Fig. 1**).[6]

Therefore, the scientific community has long been drawn to the diverse biological activities of DHPMs and DHPTs and has been inspired to develop more effective and ecologically friendly Biginelli reaction methods.[5]

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2. Review of Literature

2.1. Advances in Biginelli Reaction

Despite the utility of Biginelli reactions, they suffer from several drawbacks, including harsh reaction conditions, the use of volatile organic solvents, lengthy reaction times, and prolonged reflux. This approach is also limited to aromatic aldehydes and yields only low to moderate results.[8] Many alternative strategies, in addition to traditional Bignelli condensation, have been developed to synthesise DHPMs. Atwal et al. developed a novel strategy for the synthesis of DHPMs using the Biginelli reaction in the late 1980s. Alkyl 2-methylene-3-oxobutanoates (or equivalent 2-arylmethylene-1,3-diketones) react with iso(thio)urea analogues in a two-step process; the adducts are presumed to be intermediates. The DHPMs are obtained by deprotection after a subsequent cyclisation. It should be mentioned that this method initially requires simple media.[9] Bakibaeva and Filimonov discovered yet another base-catalysed method. They discovered that piperidine can facilitate the formation of matching DHPMs and their byproducts, Hantzsch-1,4-dihydropyridines, which were likely generated despite the breakdown of urea, leading to ammonia release.[10]

2.2. Catalysts Used in Biginelli Reaction

In traditional Biginelli reactions, HCl is used as a classical catalyst, but different catalysts have been explored, including acids and non-acidic substances.[10] Some of the catalysts used in previous literature are listed below (**Table 1**)

Table 1. Catalysts used in Biginelli Reaction

Catalyst	Reference
AcOH/EtOH	7, 14
AcOH	15, 16
H_3BO_3	17, 18
TsOH/THF	19
TMSOTf/MeCN/rt	4
TsOH	20
Ionic Liquids	21-23
Bakers'yeast	24
Graphite	25
I_2/ultrasound	26
$CuBr_2$/EtOH	27
$InBr_3$	28, 29

2.3. Deep Eutectic Solvents

Deep Eutectic Solvents have garnered attention as a new class of sustainable solvents. They have drawn notice as more environmentally friendly substitutes for traditional organic solvents.

They are binary or ternary mixtures of hydrogen bond donors (HBDs) and hydrogen bond acceptors (HBAs) in predetermined mole ratios. By stabilising the liquid structures and interacting with one another through hydrogen bonding, these mixtures lower their melting temperature. So far, 33 DESs have been prepared using easy-to-follow procedures with an atom economy of nearly 100% and typically using basic substrates.[11,12]

They are distinguished by remarkable solvent properties, including high thermal stability, low volatility, low vapour pressure, and tunable polarity. They aid in the solubilization of the solutes by forming hydrogen bonds with them. Their non-toxic, affordable, and biodegradable qualities make them superior to structurally comparable ionic liquids (ILs).[11,12] Recent research has examined the diverse functions of these innovative solvent systems, which can serve as catalysts, solvents, or even reagents, and can also promote the Biginelli condensation.

Sreekumar and coworkers identified DES, composed of zirconium oxychloride octahydrate ($ZrOCl_2 \cdot 8H_2O$) and ethylene glycol (in a 1:2 molar ratio), as a powerful catalyst for the Biginelli reaction. This DES was efficient at room temperature, exhibiting strong hydrogen bonding capability and low viscosity. The catalyst proved recyclable for at least four cycles.[12]

Zheng and colleagues proposed a DES comprising choline chloride (ChCl) and ethylene glycol in a 1:2 molar ratio as a solvent for the Biginelli reaction using *p*-TSA as a catalyst under mild conditions (50 ℃). This approach yielded high yields and could be reused for up to 8 cycles.[13] Another innovative application of DES was reported by Pasricha and co-workers, who produced room-temperature DES from choline chloride and thiourea for the synthesis of DHPTs. This research highlighted the dual role of DES as both a solvent and a source of thiourea.[11]. Given the versatility, sustainability, and benign nature of DES, this project was designed to further explore the role of DESs as a medium and a catalyst for the Biginelli reaction

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3. Materials and Methods

3.1. Materials

All reactions took place in a 5 mL single-neck round-bottom flask. Starting materials and other chemicals came from Sigma-Aldrich or local suppliers. Reaction progress was checked by thin-layer chromatography (TLC) on pre-coated silica gel plates (Merck silica gel 60, F254) and visualised under UV light.

4.1. Preparation of DES

A 100 mL single-neck round-bottom flask was fitted with a magnetic stir bar. Suitable HBD and HBA were transferred to the flask, which was placed in a preheated oil bath at 75 °C and stirred for 1 hour to produce DES as a clear, colourless, viscous liquid. DES is a liquid at room temperature.

4.2. Preparation of DHPTs

A 5.0 mL single-neck round-bottom flask was filled with the substrates, aldehyde and β-keto ester, DES, and a magnetic stir bar. The flask was placed in a preheated oil bath at 75 ^{o}C and stirred until all the starting materials had been consumed, as confirmed by TLC. After the reaction was complete, the mixture was poured into cold water, and the solid formed was filtered and dried thoroughly. No further purification was needed.

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4. Results and Discussion

Initially, the model reaction was performed with an aldehyde and a β-keto ester in the presence of thiourea, without any base, using a choline chloride-based DES, yielding products in low yield. This product was confirmed in ^{1}HNMR, establishing that the reaction proceeds as expected. This indicates that the choline chloride-based deep eutectic solvents (DES) can activate the carbonyl oxygen through hydrogen bonding. To achieve optimal yields of the product, further optimisation of reaction parameters, including temperature, time, catalyst, and solvent, was undertaken. The optimisation is now complete, and the best reaction parameters have been identified. The substrate scope for preparing the desired DHPTs is currently in progress.

Future Scope: Upon completion of the substrate scope, the manuscript shall be prepared and submitted to a suitable journal. In the future, the synthesis and characterization of novel DES will be undertaken. The scope and utility of the synthesized DES will then be explored for suitable reactions.

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