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*A Reference Book of*

# RECENT TRENDS IN SCIENCE AND TECHNOLOGY

*Chief Editor*

**Dr. Siddheshwar D. Jadhav**

**Published by Deccan Education Society's Willingdon College, Sangli**

*Prarup Publication, Kolhapur*

*A Reference Book of*

# Recent Trends in Science and Technology

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**Chief Editor:** Dr. Siddheshwar Dnyaneshwar Jadhav
M.Sc., Ph.D. (Institute of Chemical Technology, Mumbai)
Associate Professor in Chemistry,
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# PREFACE

Science and Technology is undergoing a rapid expansion and need to review in publication form is very significant. During the last few years, global scenario in science and technology has changed significantly and this is a paradigm shift in the overall development and its speed. The scientific research in multidisciplinary areas is totally changing the nature of development of the countries. Also, huge volume of variety data is available in conferences, journals, magazines and on the Internet. There is an impending need to turn the huge volume of data into knowledge. In response to such a grand challenge, this book is committed to deepening the understanding on current issues and trends to develop knowledge in and around the Emerging Technologies for scientific development.

We would like to express our deepest appreciation to the authors, whose research contributions are submitted for ISBN Book publication as a Chapter. This book is a collection of the six chapters of Chemistry, ten chapters of Computer Science, twelve chapters of Electronics, five chapters of Environmental Science, eight chapters of Life Sciences, ten chapters of Physics and five chapters of Mathematics and Statistics total fifty-six accepted manuscripts based on recent trends in sciences and Technology. Each chapter is reviewed by respective subject experts and Co-editorial team followed by plagiarism check. Publishing research work in areas of science in the form of Book represents a step further towards contribution in development of human beings.

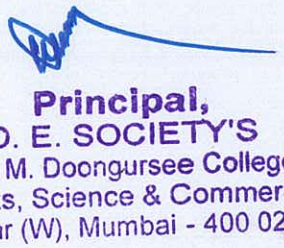
I would like to thank the Authorities of Deccan Education Society, Principal and Editorial team for their continuous support for Book Publication. I am also grateful to all Co-editors & reviewers, who have read each manuscript carefully, given critical comments on draft manuscripts, updating manuscripts into proper chapter scientific format and proofreading of this Book. This is time consuming, analytical and tedious task, without the contributions of these people, a book such as this would not have been possible.

I hope that this book will be very useful for scientific community to understand recent advances in Science and Technology.

**Dr. Siddheshwar D. Jadhav**

**Editor in Chief,**

**Recent Trends in Science and Technology (ISBN): 978-81-948061-9-6**

## MESSAGE FROM PRINCIPAL

Teaching is undoubtedly the main objective of collegiate institutions in India. The primary purpose of educational institution is to disseminate the accumulated fund of knowledge among the younger generations. Deccan Education Society's Willingdon College, Sangli occupies a special position due to its unique role, it has played in academic life of this city. We believe that a good teacher remains a keen student throughout his life. He/She excels not only in the classroom but also in the academic field of his choice. In our view, the colleges in India need teachers whose sense of dedication to learning will enable them to combine teaching and research.

It gives me an immense pleasure that, many more high-quality research articles have been received, which are selected for publication after plagiarism process through standard software & review by peer members. This edition records the scholarly combination from all over India and outside of India as well. The variety of submissions from such States have held the national and international initiative of the Book. I am also delighted that the researchers from Electronics, Physics, Life Sciences and Computer Science have demonstrated an interest to share their research with the readers of this Book.

This Book edition on Recent Trends in Science and Technology has been prepared under the esteemed guidance of number of people to whom I extend my sincere thanks. I also express my sincere thanks to Chief Editor Dr. Siddheshwar D. Jadhav and editorial team. This eBook edition on Recent Trends in Science and Technology will surely get a warm reception by all students, teachers & research scholars who want to be acquainted with the innovative ideas.

**Dr. Bhaskar V. Tamhankar,**
**Principal,**
**Deccan Education Society's Willingdon College, Sangli.**

D. E. Society's Kirti M. Doongursee College of Arts, Science & Commerce Mumbai-400028

**Principal,**
D. E. SOCIETY'S
Kirti M. Doongursee College
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Dadar (W), Mumbai - 400 028.

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## 1. Chemistry (CH)



## 2. Computer Science (CS)

# CH-01. Applicability of Deep Eutectic Solvent in Biocatalysis

**Dattatray B. Yedage*[a] and Dattatray V. Patil[b]**

[a] *Department of Chemistry, Willingdon College, Vishrambag, Sangli, India – 416415.*

[b]*Department of Chemistry, Kirti M. Doongursee College, V. S. Marg, Kashinath Dhuru Road, Near Agar Bazar, Dadar (w), Mumbai, India -400028.*

***Corresponding author E-mail:** dbyedagedb@gmail.com

## Abstract:

Deep Eutectic Solvents (DESs) or Low Transition Temperature Mixtures (LTTMs) have become attractive in recent years due to their unique physicochemical properties. Due to its inherent biological properties these eutectic solvents can be used as non-volatile biocompatible solvents with whole cells and isolated enzymes which has shown improved enzyme stability and activity translating to high conversion of product.

In this article we have reported Baker's yeast (saccharomyces cerevisiae) catalyzed one-pot three component condensation of aryl aldehydes, thioglycolic acid, and amines in deep eutectic solvent leading to 4-thiazolidinones successfully.

Keywords: Deep Eutectic Solvents, Biocatalysis, Thiazolidinones, Greener synthesis, Ecofriendly.

## 1. Introduction:

In the last few decades, biocatalysis has emerged as a powerful tool to carry out different chemical processes typically under eco-friendly reaction conditions with higher selectivity.[1], [2] Whole-cell biocatalysis by using yeast has shown its applicability in the field of pharmaceutical and medicinal chemistry. The whole-cells or enzymatic transformation have also been reported under water-free conditions, using ionic liquids[2] or neat substrates[3] among other several non-aqueous reaction media. In order to improve the solubility of the substrate(s) and product, the setup of biocatalysis under non-conventional reaction media makes available a practical framework, and at the same time offers enzyme-friendly conditions. Various ionic liquids are proved to be efficient in biocatalysis. Their toxicological properties as well as high price are important restrictions in their practical applications. To avoid these hurdles, recently, DESs have emerged as a new generation of ILs with its tailored and reduced toxicological properties.

Baker's yeast or Saccharomyces cerevisiae can carry out several organic transformations under mild reaction conditions. It can be available easily, economical, and non-hazardous. Baker's yeast and lipase can catalyze several condensation reactions to synthesize biologically active structurally diverse

heterocyclic compounds like polyhydroquinolines,[4] dihydropyridines,[5] dihydropyrimidines,[6] benzotriazoles,[7] benzimidazoles,[8] and quinoxaline.[9]

The biotransformation reactions using eutectic solvent studied so for involves aminolysis, lipase-catalyzed transesterification, epoxide hydrolysis Knoevenagel condensation, and N-alkylation of aromatic amines.[10] Baker's yeast was also explored for different enantioselective reductions of carbonyl compounds.[11]

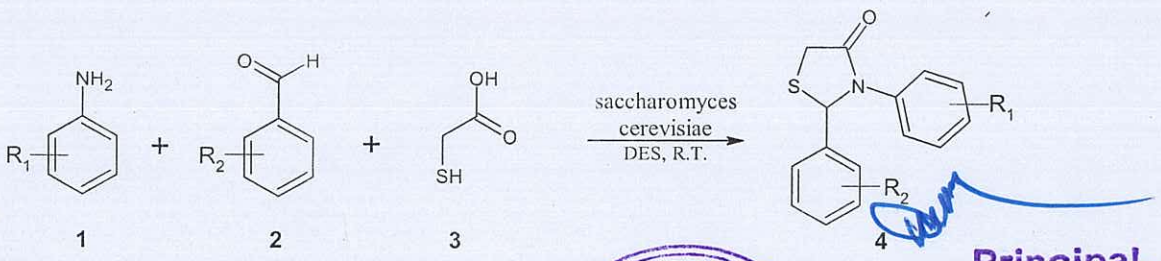
Considering the importance of thiazolidinones the applicability of choline chloride-based DES was explored as a better solvent system for the biocatalytic transformation. As a model reaction, the cyclocondensation of 4-thiazolidinones was selected (Scheme 1).

## 2. Results and discussion:

Initially, a multicomponent reaction of benzaldehyde (10 mmol), thioglycolic acid (10 mmol) and aniline (10 mmol) was carried out by using 20 $cm^3$ of different eutectic solvents which are made up of thiourea, urea, oxalic acid, succinic acid, glycerol, malonic acid with choline chloride quaternary ammonium salt in a different ratio. Choline Chloride:Glycerol ChCl:Gly (1:2) was found to be excellent eutectic solvent at room temperature with 2.0 g baker's yeast.

When the model reaction was carried out by using DES ChCl:Gly without baker's yeast as a blank reaction at normal room temperature low yield of desired product was obtained. However, when the same reaction was carried out by using baker's yeast 70 % yield of the product was found in 26 hours. It indicates that the baker's yeast plays a key role in the cyclocondensation of thiazolidinone in the presence of a eutectic solvent. The enzyme lipase which exists in baker's yeast may be responsible for accelerating the reaction and formation of the product. It improves the rate of imine formation as well as cyclisation.

The model reaction was carried out using the variable quantity of baker's yeast from 1 g to 3 g. 2 g of baker's yeast was found to be sufficient to carry out the reaction successfully giving a 70 % yield of the desired product.




1 2 3 4

Scheme 1 Baker's yeast catalyzed the synthesis of 4-thiazolidinones.

2.0 g of baker's yeast with 20 $cm^3$ of eutectic solvent was found to be sufficient to carry out the reaction at normal room temperature. Under these optimized reaction conditions, a set of substituted thiazolidinone derivatives were synthesized.

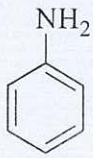
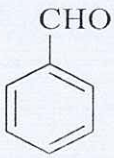
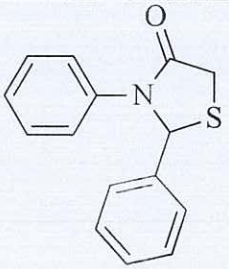
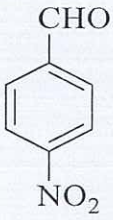
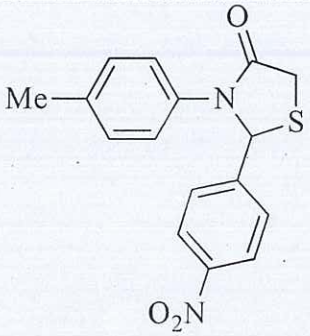
The choline chloride based eutectic solvents is well known to offer enzyme stability and activity. These results validate that the eutectic solvents may truly become promising non-conventional reaction media for whole-cell biocatalysis, in which enzymes are used for organic transformations may be active and stable.

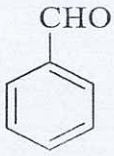
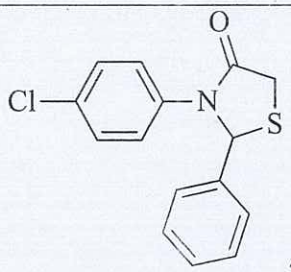
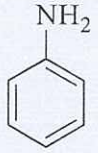
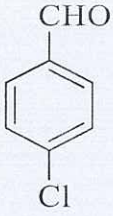
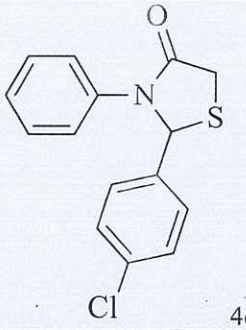
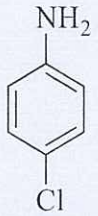
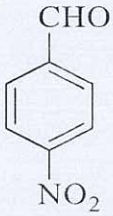
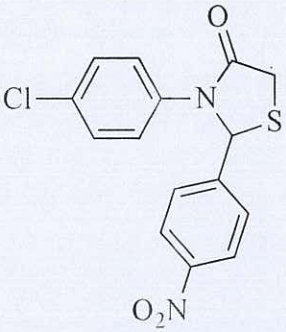
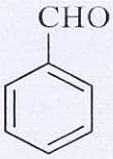
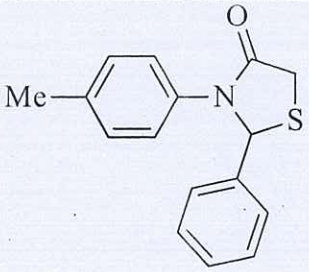
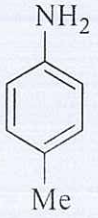
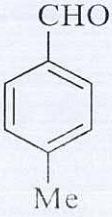
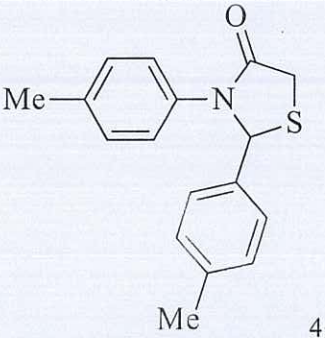
The enzyme lipase available in baker's yeast may be responsible for the formation of the imine intermediate as well as cyclocondensation of the desired product, 4-thiazolidinone.

## 3. Recyclability of DES ChCl:Gly:

Recovery and recyclability of the choline chloride based deep eutectic solvent used in this biocatalysis were also studied. We used DES ChCl:Gly as a reusable solvent with Baker's yeast to synthesize thiazolidinones. The recyclability was studied using the model reaction of aniline, benzaldehyde and thioglycolic acid under optimized conditions. After the completion of the reaction, the reaction mixture was neutralized, and the product was separated. After 26 hrs the above reaction mixture was filtered by the Celite bed to remove the baker's yeast. Water soluble eutectic solvent was recovered from the previous reaction and was reused for the next batch without purification. There was no significant loss in the yield of the expected product observed (Fig. 1, Table 2). The DES was reused for next five cycles successfully.

**Table 1:** Synthesis of 1,3-thiazolidin-4-ones using Baker's yeast.[a]

| ENTRY | ANILINE | ALDEHYDE | PRODUCT | MELTING POINT (°C) | YIELD (%)[b] |
|---|---|---|---|---|---|
| 1. |  |  |  4a | 130-132 (lit[12] 129-131) | 70 |
| 2. |  |  |  4b | 158-160 (lit[13] 158-160) | 81 |

| ENTRY | ANILINE | ALDEHYDE | PRODUCT | MELTING POINT (°C) | YIELD (%)[b] |
|---|---|---|---|---|---|
| 3. |  |  |  4c | 120- 122 (lit[12] 121-123) | 68 |
| 4. |  |  |  4d | 124-126 (lit[12]122-126) | 81 |
| 5. |  |  |  4e | 112-114 (lit[14] 112-115) | 80 |
| 6. |  |  |  4f | 105-107 (lit[12] 105-107) | 75 |
| 7. |  |  |  4g | 122-125 (lit[12] 121-123) | 90 |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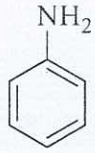
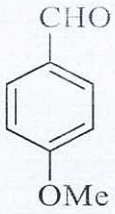
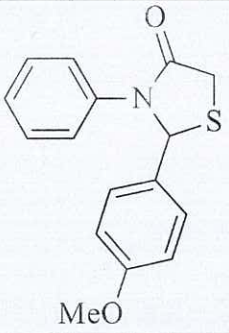
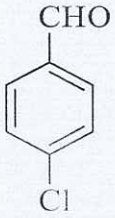
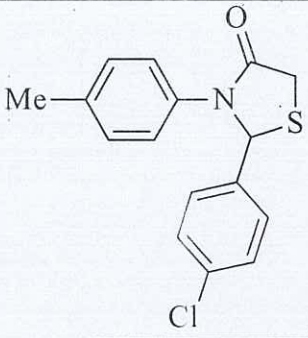
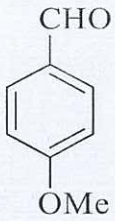
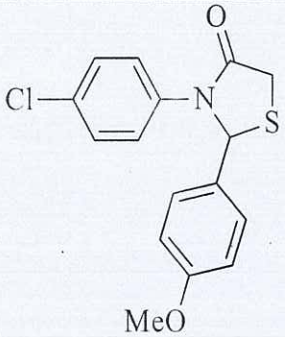
|---|---|---|---|---|---|
| 8. |  |  |  4h | 98-100 (lit[13] 97-99) | 72 |
| 9. |  |  |  4i | 161-163 (lit[12] 162-164) | 81 |
| 10. |  |  |  4j | 104-106 (lit[13] 104-106) | 65 |

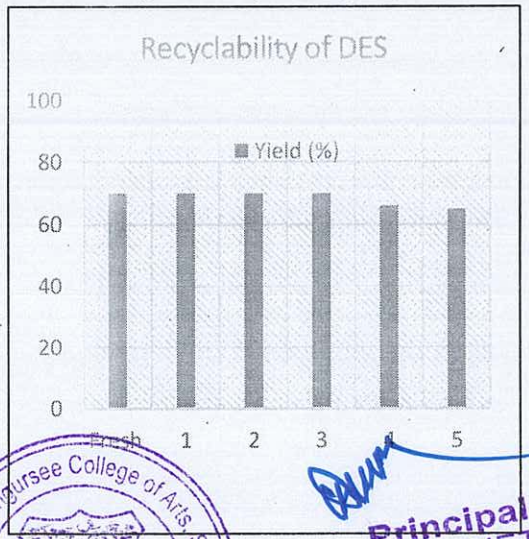
Reaction conditions: aryl amine (10 mmol), aryl aldehyde (10 mmol), thioglycolic acid (10 mmol), baker's yeast 2.0 g in 20 $cm^3$ DES stir, 26 hrs at r.t.; [b] Isolated yield.

Table 2 Recyclability of DES ChCl:Gly.[a]

| ENTRY | RUN | TIME (Hrs) | YIELD (%) [b] |
|---|---|---|---|
| 1 | Fresh | 26 | 70 |
| 2 | 1 | 26 | 70 |
| 3 | 2 | 26 | 70 |
| 4 | 3 | 26 | 70 |
| 5 | 4 | 26 | 66 |
| 6 | 5 | 26 | 65 |

Reaction conditions: aryl amine (10 mmol), aryl aldehyde (10 mmol), thioglycolic acid (10 mmol), baker's yeast 2.0 g in DES 20 $cm^3$ at r.t.; [b] Isolated yield.

Figure 1 Recyclability of DES.



## 4. Conclusions:

A simple and energy-efficient pathway for the development of biologically important functionalized thiazolidinones like 1,3-thiazolidinon-4-one has been reported using Baker's yeast with ChCl:Gly cost effective, and eco-friendly solvent. This methodology is catalyst-free and proceeds under mild reaction conditions and produced the products in good yields. The recovered solvent could be recycled for up to five runs.

5. Experimental Section:

5.1. Preparation of ChCl:Gly (1:2) DES

As per the literature,[15] 50 mmol of choline chloride (ChCl) and 100 mmol of glycerol were mixed together in a round-bottom flask. The reaction mixture was stirred and heated at 72 °C until a clear liquid was formed. The atom efficiency of this reaction is 100 %.

5.2. Preparation of 2, 3-diaryl 4-thiazolidinones using Baker's yeast

The mixture of aryl aldehyde (10 mmol), aromatic amine (10 mmol), thioglycolic acid (10 mmol) and baker's yeast (active & dry, 2 g) added in ChCl:Glycerol (1:2) deep eutectic solvent (20 $cm^3$) and stirred continuously at normal room temperature. The progress of the reaction was observed by TLC (EtOAc:Hexane solvent system). After 26 hours the above reaction mixture was filtered by the Celite bed to remove the baker's yeast. Under reduced pressure, the obtained filtrate was concentrated and the resulting crude product thiazolidinone was recrystallized.

6. Physical constants and characterization of compounds

Spectral and physical data ($^1$H NMR, $^{13}$C NMR, MP, etc.) of all these thiazolidinones are in agreement with those discussed in the literature[16-19]. Representative spectral data is as follow.

4a: *2,3-diphenylthiazolidin-4-one*: 4a was synthesized by cyclocondensation between aniline 1a, benzaldehyde 2a and thioglycolic acid 3 as a white solid; Yield: 95 %; Melting point: 128-130 °C (lit[12]); $^1$H NMR (600 MHz, $CDCl_3$) δ 7.30-7.34 (dd, 7H $J$ = 9.6, 4.9 Hz,), 7.14-7.17 (d, 3H, $J$ = 7.9 Hz), 6.18 (s, 1H), 3.83-3.97 (dd, $J$ = 50.1, 2H, 15.8 Hz); FTIR ($cm^{-1}$): 2893, 1678, 1588, 1488, 1338, 610; $^{13}$C NMR (400 MHz, $CDCl_3$) δ 34.35, 66.06, 124.70, 126.18, 127.22, 130.10, 130.27, 130.88, 137.66, 139.35, 171.41; LC MS (m/z): 256.09 $[M+H]^+$.

4b: *2-(4-nitrophenyl)-3-(p-tolyl) thiazolidin-4-one*: 4b was synthesized by cyclocondensation between 4-methylaniline 1b, 4-nitrobenzaldehyde 2b and thioglycolic acid 3 as a Yellow solid; Yield: 97 %; Melting point: 158-160 °C (lit[13]); $^1$H NMR (600 MHz, $CDCl_3$) δ 8.15-8.17 (d, 2H, $J$ = 8.5 Hz), 7.40-7.43 (d, 2H, $J$ = 8.2 Hz), 7.02 – 7.12 (m, 4H), 6.0 (s, 1H), 3.87-4.09 (dd, 2H, $J$ = 36.7, 15.9 Hz), 2.34 (s, 3H); FTIR ($cm^{-1}$): 2929, 1688, 1513, 1393, 1295, 630; $^{13}$C NMR (400 MHz, $CDCl_3$) δ 21.07, 34.32, 64.50, 128.28, 125.21, 128.00, 131.14, 134.88, 137.68, 145.98, 148.87, 170.11; LC MS (m/z): 315.06 $[M+H]^+$

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D. E. Society's Kirti M. Doongursee College of Arts, Science & Commerce Mumbai-400028

Principal,
D. E. SOCIETY'S
Kirti M. Doongursee College
of Arts, Science & Commerce
Dadar (W), Mumbai - 400 028.

# CH-02. Ecofriendly Eutectic Solvent Assisted One-Pot Three Component Synthesis of Functionalized Thiazolidinones

**Dattatray B. Yedage*[a] and Dattatray V. Patil[b]**

[a] *Department of Chemistry, Willingdon College, Vishrambag, Sangli-416415, India.*

[b] *Department of Chemistry, Kirti M. Doongursee College, V. S. Marg, Kashinath Dhuru Road, Near Agar Bazar, Dadar (w), Mumbai-400028, India.*

***Corresponding author E-mail:** dbyedagedb@gmail.com

## Abstract:

A simple and environment-friendly protocol was developed to synthesize functionalized thiazolidinones via a one-pot three-component reaction of readily available aryl aldehydes, diamines or 2-aminobenzimidazole and thioglycolic acid using choline chloride-based deep eutectic solvent. The advantages of this protocol are easy work-up, shorter reaction time, catalyst-free, high atom-economical reaction, and higher yields of the desired products. The eutectic solvent could be recycled up to five runs without a significant decrease in the yield of the desired products.

**Keywords**: Thioglycolic acid, Eutectic solvents, One-pot synthesis, Ecofriendly synthesis.

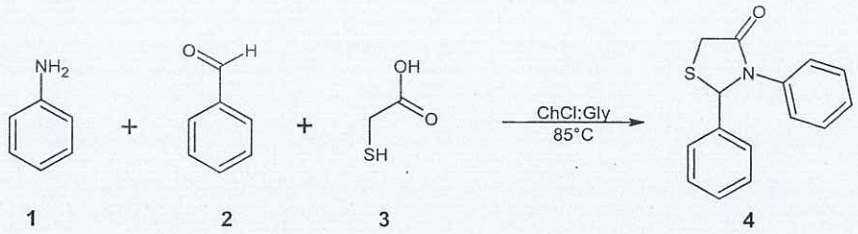
## 1. Introduction:

Owing to the high potential pharmacological activities thiazolidinone and its derivatives are one of the most extensively studied classes of organic compounds.[1] They show diverse therapeutic activities such as anti-bacterial,[2] anti-cancer,[3] anti-convulsant,[4] anti-inflammatory,[3] anti-tubercular,[5] antifungal,[6] antioxidants[7] and antihyperglycemic and antidyslipidemic agents.[8]

In recent years, many synthetic methods for the synthesis of thiazolidine-4-one ring system in the presence of catalysts or activators such as Baker's yeast,[9] $[Et_3NH][HSO_4]$,[5] HBTU,[10] DCC,[11] Montmorillonite K-10,[12] DBSA[13] MCM supported Schiff base[14], $La(NO_3)_3.6\ H_2O$[15], nano-$Fe_3O_4$@$SiO_2$ supported ionic liquid[16] and supported protic acid[17] have been employed. However, most of these procedures suffer from one or more drawbacks such as the use of hazardous organic solvents, low yield, long reaction time, use of expensive catalysts, or tedious workup procedures.

In order to overcome these disadvantages, some new types of greener solvents are strongly needed. In the sense, quaternary salts like choline chloride-based deep eutectic solvents have been explored as environmentally benign solvents for several chemical processes, metal deposition, material chemistry, and purification of biodiesel.[18-24] These deep eutectic solvent DESs or low melting mixtures currently receive more attention due to their tunable physiochemical properties and ability to carry out

many types of reactions like addition, condensation, cyclisation, oxidation, and multicomponent reactions .[18] Recently, we published our work related to the synthesis of various 1,3-thaizolidin-4-one derivatives using ChCl:Gly deep eutectic solvent. (Scheme 1).[20] Present work discussed in this article is the extension of our previous efforts to develop biological active functionalized thiazolidinone derivatives by a greener method.

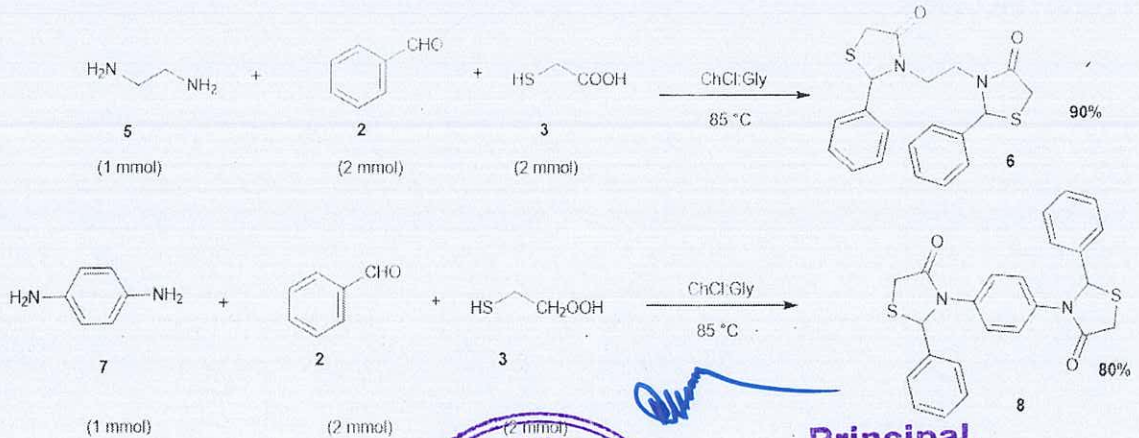



Scheme 2 Multicomponent reaction of 1, 2 and 3 to form 1, 3-thiazolidin-4-one. [20]

In this protocol, the scope and applicability of this methodology have been extended. Initially, Choline chloride-based deep eutectic solvent (DES) was again explored for the synthesis of structurally diverse functionalized thiazolidinone derivatives by cyclisation of aryl aldehydes, diamines, or and thioglycolic acid under mild reaction conditions. (Scheme 2 and 3).

## 2. Result and Discussions:

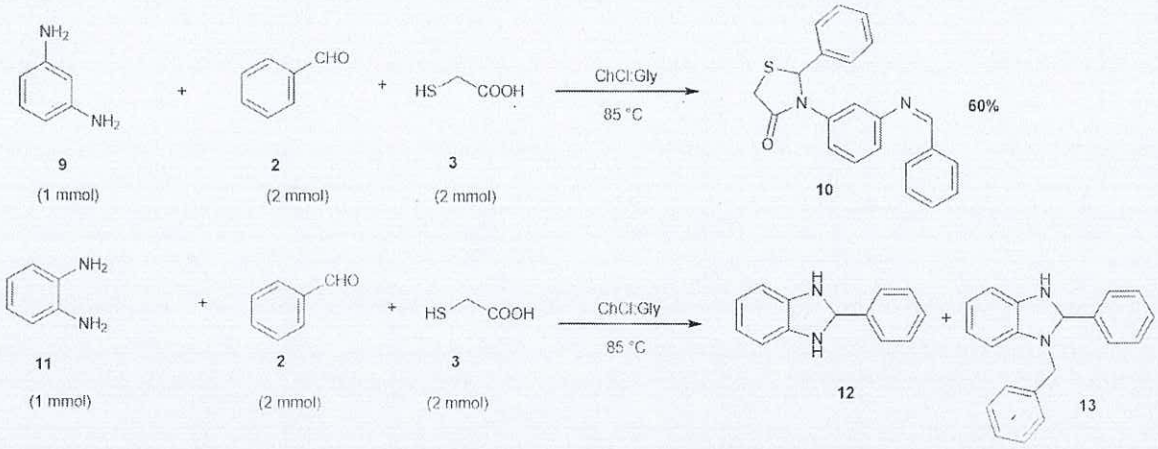
Herein, we describe a simple, and convenient procedure for the synthesis of thiazolidinones with a one-pot reaction of arylaldehyde, diamines, and thioglycolic acid without using any metal catalyst. Ethylene diamine, and o/m/p-phenylenediamines are used instead of primary amines to extend our research work. Good yields of bisthiazolidinones were observed with diamines like ethylene diamine and p-phenylene diamine in which the amino groups are away from each other, and cyclisation took place easily.




Scheme 2 Synthesis of bis-thiazolidinones by using *p*-phenylene diamine and ethylene diamine.

Bisthiazolidinones were not produced when the reaction was carried out with *m*-phenylenediamine and *o*-phenylene diamine. In the case of ortho and meta-phenylenediamines, the amino groups are closer

to each other due to this cyclisation was not observed and resulted in a mixture of undesired products. (Scheme 3).




Scheme 3 The reaction of *o/m*-phenylenediamine with benzaldehyde and thioglycolic acid.

In the model reaction, the synthesis of Bis-thaizolidinone was investigated with the reaction of ethylene diamine (1 mmol), benzaldehyde (2 mmol), and thioglycolic acid (2 mmol). In our study, we initially carried out the reaction without any catalyst or solvent at room temperature for 12 hours, but no product was obtained. We carried out the same reaction at 65 °C but the reaction didn't take place (Table 1 Entry 1, 2). Furthermore, we used some greener solvents including deep eutectic solvents at 65 °C to evaluate the model reaction and the results are summarized in Table 1. It is observed that a considerably good yield was obtained with ChCl: Gly (1:2). (Table 1, Entry 13). The results confirm the effective use of DES ChCl: Gly (1:2) as a catalyst cum reaction media for the preparation of bis-thiazolidinones.

Considering the promoting catalytic activity of choline chloride-based deep eutectic solvent ChCl:Gly (1:2) we carried out a blank reaction of ethylenediamine (1 mmol), benzaldehyde (2 mmol) and thioglycolic acid (2.0 mmol) in ChCl: Gly (1:2) at ambient temperature but a low yield of the product was observed. Thus, we decided to optimize the temperature and evaluated the same reaction at different temperatures ranging from 65 to 95 °C. It was observed that an increase in temperature gradually increased the yield of the product and at 85 °C reaction produced 90 % of the yield within 55 minutes (Table 1, Entry 15).

Moreover, we studied the concentration of DES ChCl: Gly (1:2) for the model reaction. The expected product was not observed, in the absence of the eutectic solvent. After varying the concentration of DES (ChCl: Gly) at 0.5, 1.0, 1.5, 2.0, 2.5 g at 85 °C, the desired product was observed at 45%, 50%, 65%, 80%, 85%, 85% respectively. It suggests that 2.0 g of eutectic solvent (ChCl: Gly) is sufficient for a better yield of the desired product.

Table 3 Optimization of reaction conditions.[a]

| Entry | Solvent | Temp. (°C) | Time (min) | Yield (%)[b] |
|---|---|---|---|---|
| 1. | - | RT | 720 | - |
| 2. | - | 65 | 720 | - |
| 3. | Water | 65 | 90 | 10 |
| 4. | MeOH | 65 | 90 | 15 |
| 5. | Toluene | 65 | 90 | 10 |
| 6. | Acetonitrile | 65 | 90 | 20 |
| 7. | ChCl: Urea (1:2) | 65 | 90 | 60 |
| 8. | ChCl: Thiourea (1:2) | 65 | 90 | 58 |
| 9. | ChCl: Oxalic Acid (1:1) | 65 | 90 | 50 |
| 10. | ChCl: Adipic Acid (1:1) | 65 | 90 | 45 |
| 11. | ChCl: Succinic Acid (1:1) | 65 | 90 | 45 |
| 12. | ChCl: Malonic Acid (1:1) | 65 | 90 | 40 |
| 13. | ChCl: Glycerol (1:2) | 65 | 55 | 70 |
| 14. | ChCl: Glycerol (1:2) | 75 | 55 | 80 |
| 15. | ChCl: Glycerol (1:2) | 85 | 55 | 90 |
| 16. | ChCl: Glycerol (1:2) | 95 | 55 | 90 |
| 17. | Glycerol | 85 | 55 | 62 |
| 18. | ChCl | 85 | 55 | trace |

[a] Reaction conditions: ethylenediamine (1 mmol), benzaldehyde (2 mmol) and thioglycolic acid (2 mmol) in ChCl:Gly (2.0 g); [b] Isolated yield; ChCl: Choline chloride.

Therefore, the optimized reaction conditions for this reaction are as follows: 2-aminobenzimidazole (1 mmol), aryl aldehyde (2 mmol), and thioglycolic acid (2 mmol), DES 2.0 g at 85 °C for 55 minutes (Table 1, Entry 15).

When the model reaction was performed in glycerol, only 62 % yield was observed, whereas ChCl produced no significant yield at optimal reaction conditions. It suggests that the reaction produces a better yield due to DES (ChCl: Gly) and not because of its individual components. (Table 1, Entry 16, 17).

The deep eutectic solvent ChCl: Gly (1:2) was prepared by simple mixing of glycerol (200 mmol) and choline chloride (100 mmol) and heated to 70 °C with continuous stirring till a homogeneous clear solution was formed, which was used further without any purification.

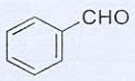
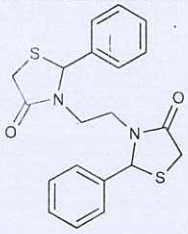
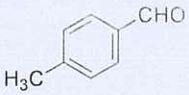
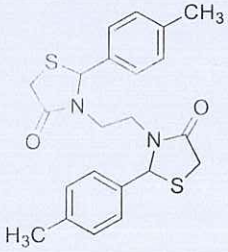
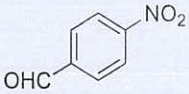
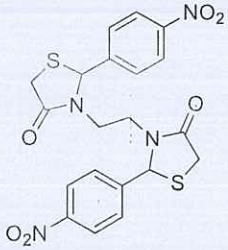
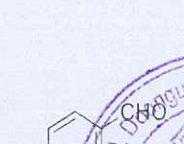
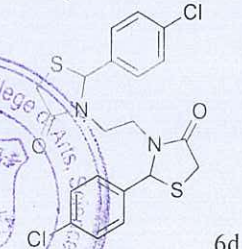
In this methodology, we used diamines like o/m/p-phenylenediamines and ethylene diamines instead of aniline derivatives. Initially, ChCl: Gly eutectic solvent was used in cyclisation reaction of ethylene diamine with aryl aldehyde and thioglycolic acid which gave an 80 % yield of desired product 6. Furthermore, when using p-phenylenediamine with 2 and 3 we observed a 90% yield of the 8. However, the same reaction with m-phenylenediamine didn't result in bis-thiazolidinone instead of that it gives 10. Whereas o-phenylenediamine produced a mixture of products 12 and 13.

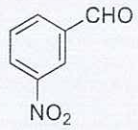
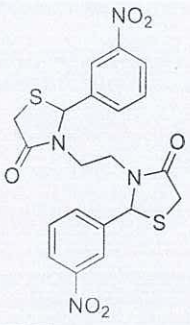
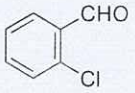
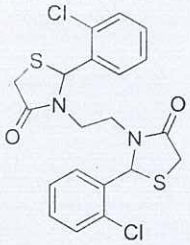
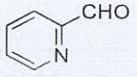
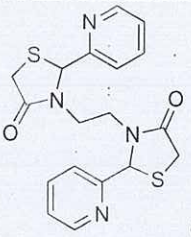
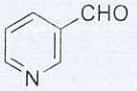
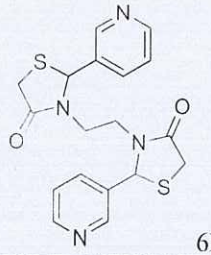
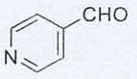
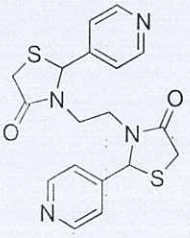
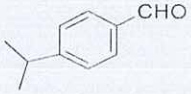
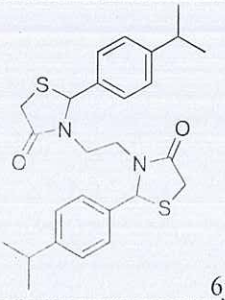
Under optimized reaction conditions, to elaborate the generality and scope of this protocol various aromatic and heterocyclic aldehydes were studied and the results are summarized in table 2. Aromatic aldehydes, such as nitro benzaldehyde, benzaldehyde, ortho, and para chlorobenzaldehyde and nicotinaldehyde, have shown to be effective substrates and produced Bis-thiazolidinone in excellent yields (85-92%). However aliphatic aldehydes didn't participate in the reaction.

To study the industrial applicability of the reaction, the scale-up experiment was done, ethylene diamine (100 mmol) with benzaldehyde (200 mmol) and thioglycolic acid (200) mmol in DES was carried out under the optimized conditions and a 90% isolated yield of the bis-thiazolidinone was observed.

In order to develop structurally diverse and biologically active heterocyclic functionalized thiazolidinone compounds, we employed this choline chloride-based deep eutectic solvent for the synthesis of 3-enzimidazolyl-2-aryl thiazolidine-4-one by using heterocyclic amine 2-aminobenzimidazole with aryl aldehyde and aniline derivatives.

Table 4 DES mediated synthesis of Bis-thiazolidinone derivatives (6a-j).

| Entry | Aldehyde | Product (6a-j) | Time (min) | Yield (%)[b] | Melting Point (°C) | Reference |
|---|---|---|---|---|---|---|
| 1. |  |  6a | 55 | 90 | 155-157 | 155-157 [21] |
| 2. |  |  6b | 55 | 90 | 156-58 | 158-60 [21] |
| 3. |  |  6c | 50 | 92 | 165-67 | 164-166 [21] |
| 4. |  |  6d | 50 | 92 | 151-153 | 150-152 [21] |

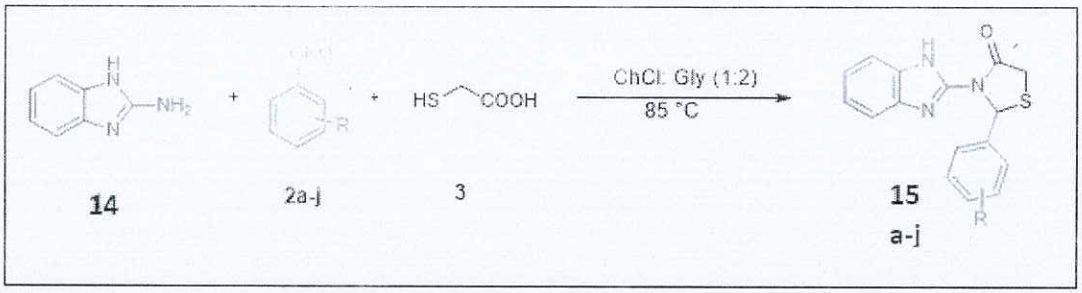
| Entry | Aldehyde | Product (6a-j) | Time (min) | Yield (%)[b] | Melting Point (°C) | Reference |
|---|---|---|---|---|---|---|
| 5. |  |  6e | 50 | 90 | 222-224 | 222-224 [21] |
| 6. |  |  6f | 50 | 92 | 143-145 | 143-145 [21] |
| 7. |  |  6g | 55 | 90 | 168-170 | 170-172 [21] |
| 8. |  |  6h | 55 | 90 | 193-195 | 191-193 [21] |
| 9. |  |  6i | 55 | 95 | 222-224 | 221-223 [21] |
| 10. |  |  6j | 60 | 85 | 163-65 | 163-165 [21] |

[a] Reaction conditions: ethylenediamine (1 mmol), benzaldehyde (2 mmol), and thioglycolic acid (2 mmol) in ChCl:Gly (2.0 g); [b] Isolated yield; ChCl: Choline Chloride.



The reaction of 2-aminobenzimidazole (1.1 mmol), aromatic aldehyde (1 mmol), and thioglycolic acid (1 mmol) was selected as a model reaction. (Scheme 3). The optimized reaction conditions for this reaction are as follows: 2-aminobenzimidazole (1.1 mmol), aryl aldehyde (1 mmol), and thioglycolic

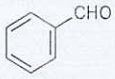
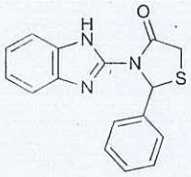
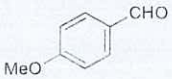
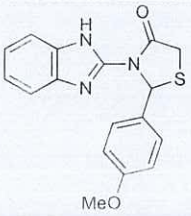
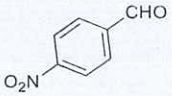
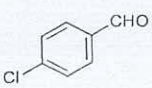
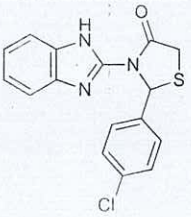
acid (1 mmol), DES 2.0 g at 85 °C for 55 minutes (Table 1, Entry 15). The reaction produced a better results under these optimized reaction conditions.

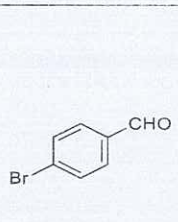
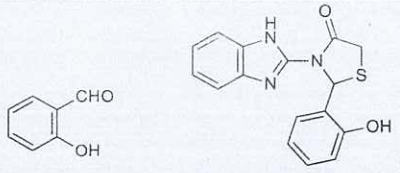
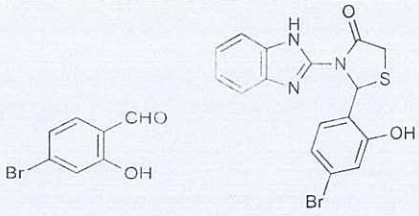
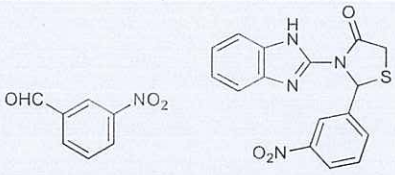
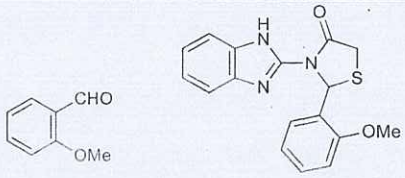
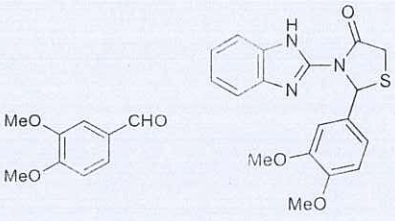



Scheme 3 Synthesis of 3-benzimidazolyl-2-aryl thiazolidinones using DES.

The aforesaid results encouraged us to generalize this methodology. We extended the reaction of 2-aminonbenzimidazole with different aryl aldehydes and thioglycolic acid under similar reaction conditions providing the respective 3-benzimidazolyl-2-aryl thiazolidine-4-ones in good to excellent yields. The results are summarized in Table 3. Physical and spectral data of known compounds are in agreement with those described in the literature.[15]

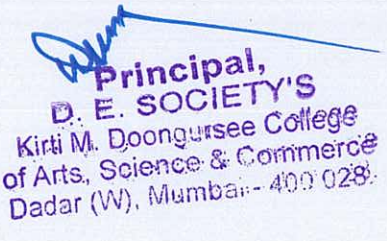
Table 5 DES mediated synthesis of 3-benzimidazolyl-2-aryl thiazolidine-4-one derivatives.

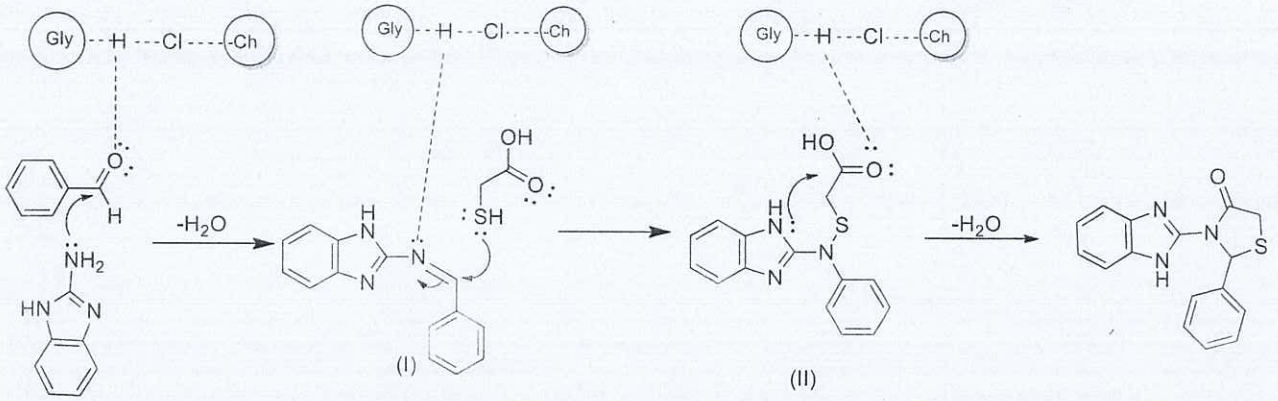
| Entry | Aldehyde | Product | Time (min) | Yield (%) | Melting Point (°C) Obs. | Lit. |
|---|---|---|---|---|---|---|
| 1. |  |  15a | 55 | 90 | 208-210 | 208-210[15] |
| 2. |  |  15b | 55 | 85 | 215-217 | 215-217[15] |
| 3. |  |  15c | 50 | 93 | 207-210 | 207-210[15] |
| 4. |  |  15d | 50 | 95 | 230-232 | 229-230[15] |

| | | | | | |
|---|---|---|---|---|---|
| 5. |  15e | 50 | 94 | 244-246 | 244-246[15] |
| 6. |  15f | 55 | 90 | 244-246 | 245-246[15] |
| 7. |  15g | 55 | 92 | 259-261 | 260-261[15] |
| 8. |  15h | 50 | 88 | 147-149 | 146-148[15] |
| 9. |  15i | 55 | 85 | 262-264 | 263-266[15] |
| 10. |  15j | 55 | 80 | 166-168 | 167-170[15] |

[a] Reaction conditions: 2-aminobenzimidazole (1.1 mmol), benzaldehyde (1 mmol) and thioglycolic acid (1 mmol) in ChCl:Gly (2.0 g); [b] Isolated yield; ChCl: Choline Chloride.

From above Table 3, we conclude that the experiential results were excellent in terms of reaction time, yields, and purity of products. Electron withdrawing substituent on aryl aldehydes favors the formation of product in the presence of DES.




Scheme 4 Plausible mechanism for the formation of 3-benzimidazolyl-2-aryl thiazolidine-4-one.

Based on the literature,[20] a mechanism for the formation of 3-benzimidazolyl-2-aryl thiazolidine-4-one catalyzed by the eutectic solvent is outlined in Scheme 4. Initially, DES interacts with the oxygen atom of the carbonyl group of aryl aldehyde through H-bonding and increases the electrophilicity of the carbonyl carbon which enhances the attack of the $NH_2$ group and leads to the formation of imine intermediate (I). Sulfur atom of thioglycolic acid attacks intermediate (I) and forms the carbon-sulfur linkage. Further, DES activates the intermediate (II) which cyclizes to the product. Extensive hydrogen bonding within DES and successful activation of carbonyl groups during cyclisation is the key aspect of this mechanism, which enhances the rate of the reaction.

The synthesis of bis thiazolidinones also proceeds through the same mechanistic pathway. In consideration of the greener approach, the DES (ChCl: Gly) catalyst cum reaction media was further studied for recyclability. The model reaction of 2-aminobenzimidazole (1.1 mmol), benzaldehyde (1 mmol), and thioglycolic acid (1 mmol) carried out under optimized conditions. Upon completion of the reaction, the reaction mixture was neutralized by a saturated solution of sodium bicarbonate. The product was extracted using ethyl acetate. The DES which is water soluble recovered by evaporating the water under reduced pressure. The recovered eutectic solvent (ChCl: Gly) was recycled for the next run without any purification. The eutectic solvent was reused and recycled up to five times without any loss in the yield of the product. (Fig 1). Hence the protocol is beneficial over previously reported methods.

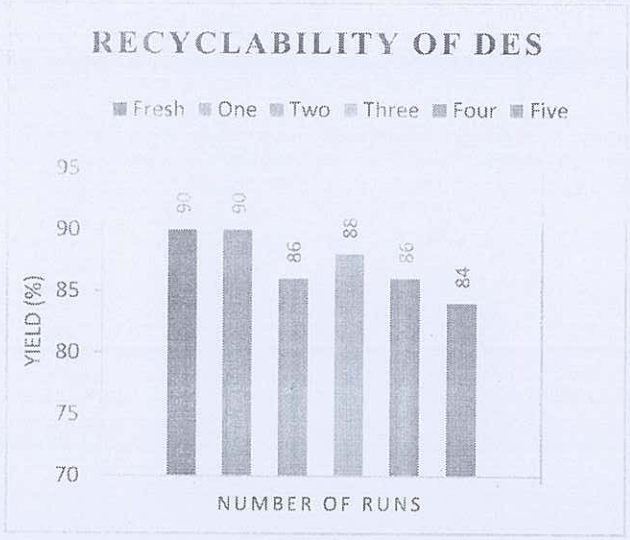



**Figure 2** Recyclability of DES (ChCl: Gly).

## 3. Experimental:

All commercially available reagents were used without purification and all solvents used were of reagent grade. The progress of all reactions was monitored by using TLC (Silica gel 60 F254). Melting points were determined in open capillaries and are corrected. The IR spectra of products were recorded on Perkin Elmer spectrometer. $^{1}H$ NMR and $^{13}C$ NMR of purified compounds were recorded on a Brucker-300 MHz instrument using $CDCl_3$ as a solvent and TMS as an internal standard.

3.1 Procedure for preparation of DES:

A mixture of choline chloride (100 mmol) and glycerol (200 mmol) was added to a 100 $cm^3$ round bottom flask and stirred magnetically at 70 °C until homogeneous liquid was obtained. [22] The resulted DES was used without any purification. This method gave 100 % atom economy since it forms a deep eutectic mixture with no side product formation.

3.2 Procedure for synthesis of bis-thiazolidinone (6a-j):

A mixture of thioglycolic acid (2 mmol), benzaldehydes (2 mmol), and o/m/p phenylenediamines or ethylene diamines (1 mmol) in 2.0 g ChCl: Gly (1:2) was stirred at 85 °C for 50-60 minutes (Scheme 2). The reaction progress was monitored by TLC. On completion of the reaction, the reaction mixture was cooled at room temperature and neutralized by using a saturated solution of sodium bicarbonate. The resulting product was extracted with ethyl acetate. The solvent (ethyl acetate) was evaporated under reduced pressure to obtain the desired product. The obtained products were recrystallized using EtOH.

3.3 General Experimental Procedure for preparation of 3-benzimidazolyl-2-aryl thiazolidine-4-one (15 a-j)

A mixture of 2-aminobenzimidazole (1.1 mmol), aromatic aldehyde (1 mmol), and thioglycolic acid (1 mmol) in 2.0 g ChCl: Gly (1: 2) was stirred magnetically at 85 °C for 50 to 55 minutes. The progress of the reaction was monitored by using TLC. (Using ethyl acetate and hexane as a solvent system). On

completion, of the reaction, the mixture was cooled and neutralized. The product was extracted using ethyl acetate, which was later evaporated under reduced pressure. The obtained crude product was recrystallized using ethanol and air dried.

## 4. Representative Spectral Data:

6a: *3,3'-(ethane-1,2-diyl)bis(2-phenylthiazolidin-4-one)*: The compound 6a was synthesized by condensation between benzaldehyde, ethylene diamine and thioglycolic acid in 55 minutes. Color: White solid; Yield: 90 %; Melting point: 155-157 °C (Lit[21] 155–157 °C), $^1$HNMR (600 MHz, DMSO-d6) δ (PPM): 2.43-2.78 (m, 2H), 3.61-3.64 (m, 2H), 3.69-3.75 (d, J = 16 Hz, 2H), 3.94-3.98 (dd, J = 1.8,1 6 Hz, 2H), 5.64 (d, J = 1.6 Hz, 2H),7.30-7.41 (m, 10 H); $^{13}$C NMR (400 MHz, $CDCl_3$) δ 32.76, 40.88, 63.70, 127.50, 129.30, 130.00, 138.25, 172.18; IR (FTIR) $cm^{-1}$ : 2948, 1655, 1405, 688; LCMS (m/z): 385.10 $[M+H]^+$ .

6d: *3,3'-(ethane-1,2-diyl)bis(2-(4-chlorophenyl)thiazolidin-4-one):* The compound 6d was synthesized by condensation between p-chlorobenzaldehyde, ethylene diamine, and thioglycolic acid in 50 minutes. Color: white solid; Yield: 92 %; Melting point: 151-153 °C (Lit[21]150-152 °C), IR (FTIR) $cm^{-1}$ : 2938, 1647, 1427, 1259, 694.

15a: *3-(1H-benzo[d]imidazol-2-yl)-2-phenylthiazolidin-4-one:* The compound 15a was synthesized by condensation between 2-aminobenzimidazole, benzaldehyde, and thioglycolic acid in 55 minutes. Color: light yellow solid; Yield: 90 %; Melting point: 208- 210 °C (Lit[15] 208–210 °C); $^1$H NMR (600 MHz, $CDCl_3$-d6) δ: 3.89 (1H, $SCH_2$, d, J = 16.54 Hz), 4.33 (1H, $SCH_2$, d, J = 16.56 Hz), 6.66 (1H, CH, s), 7.21 (2H, H-Ar, m), 7.23– 7.50 (6H, H-Ar, m), 7.69 (1H, H-Ar, d, J = 7.50 Hz), 13.01 (1H, NH, s)

## 5. Conclusions:

In conclusion, we reported a simple and green protocol for the formation of bis-thaizolidinone and 3-benzimidazolyl-2-aryl thiazolidine-4-one derivatives by using ChCl:Gly as an eco-friendly, biodegradable and cost-effective eutectic solvent. The recovered DES from the previous run could be recycled in up to five batches. The reaction procedure is rapid, catalyst-free, and proceeds under mild reaction conditions and gave the desired products in excellent yields.

6. Acknowledgment:

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Principal,
D. E. SOCIETY'S
Kirti M. Doongursee College
of Arts, Science & Commerce
Dadar (W), Mumbai - 400 028.

D. E. Society's Kirti M. Doongursee College of Arts, Science & Commerce Mumbai-400028