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Comparative studies for the synthesis of aliphatic thiazolidinone derivatives from novel 1, 4 diaminobutane based dicationic ionic liquids

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Abstract

Most of the heterocyclic reactions are toxic and hazardous in nature in previous dates. The synthesis strategies are continue to more attraction for the construction and development and find in wide range of applications in recent years with green chemistry. In this thiazolidinone are one the most active biological theme compounds and varying this study we perform a one pot three component synthesis route of greener method with aliphatic aldehyde and aromatic amines with Thioglycolic acid in presence of catalyst like Novel 1, 4 diaminobutane based dicationic ionic liquids. Which reduce reaction time. Its high range and easy recyclability abilities give high yield without any loss of its catalytic activity. Synthesis compound were confirm by TLC and its characterization like $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, $^{19}\text{F-NMR}$, $^{31}\text{P-NMR}$, FT-IR. Allowing the product obtained with better identifications.

Keywords: Green approach, DCILs, Aromatic amines, Aliphatic aldehydes, Thiazolidinone. Corresponding Author: Prashant N. Muskawar, Amolakchand Mahavidyalaya, Yavatmal, Maharashtra, India

1. Introduction

Thiazolidinone derivatives represent an significant class of sulfur- and nitrogen-containing five-membered heterocycles that have attracted considerable attention due to their wide spectrum of biological activities, including anti-cancer, anti-microbial, anti-inflammatory, anti-diabetic, and anti-viral properties [1-4, 34, 35]. Thiazolidinones carbonyl group at the 4 presence structural flexibility, reactive position and are allows extensive functionalization, making valuable scaffolds in drug discovery in medicinal chemistry [2, 5, 36]. Traditionally, aliphatic thiazolidinone derivatives improved pharmacokinetic and gained interest enhanced lipophilicity compared to aromatic analogues [3, 6].

Thiazolidinone derivatives are synthesized from one-pot multicomponent reactions involving aliphatic aldehydes, amines, and thioglycolic acid (mercaptoacetic acid) under conventional or hazardous process with organic solvents such as, ethanol, toluene, or acetonitrile [5-7, 32]. However, the use of toxic and volatile organic solvents are suffer from some several drawbacks in all methods including with prolonged reaction times, less product yield, low yields, which pose environmental and safety concerns [6, 8, 26]. In accordance with green chemistry principle the development of these limitations have prompted sustainable and environmentally benign synthetic strategies [9, 30]. In this paragraph, Beyond acting as a solvent ionic liquids (ILs) have emerged as promising alternatives to carcinogenic solvents method due to their physicochemical properties, such as, high thermal stability, non-flammability, negligible vapor pressure and tunable polarity [9-11, 27, 28]. In various organic transformations ionic liquids to act solvents not only as green catalyst but also as efficient catalysts In recent years increases Dicaionic liquid-mediated reactions exhibit enhanced reaction rates, higher selectivity, improved yields and minimize environmental impacts as compared to traditional methodologies [10-12, 40]. Furthermore, Dicationic ionic liquids can be easily recovered and reused more times than Monocationic ILs, thereby reducing waste products and improving process sustainability [12-13, 43].

Among various classes of Monocationic ionic liquids (MILs) and dicationic ionic liquids

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(DCILs) have attention for increasing and attraction to their superior physicochemical and catalytic properties compared to mono-cationic ionic liquids [12, 14]. The attention shifted on Dicationic Ionic for the development of new subclass having two cationic centers linked by a flexible spacer, resulting they demonstrated lower toxicity and stronger electrostatic interactions, enhanced hydrogen bonding capability, and also improved thermal stability. These features highlight contribution to their enhanced catalytic efficiency and recyclability [13-15, 31]. Particularly, heterocyclic synthesis with suitable for multicomponent reactions process making them catalytic active DILs [11, 12, 44]. In comparison, monocationic ionic liquids often exhibit lower catalytic activity than Dicationic ionic liquids and limited recyclability due to weaker ionic interactions [9].

The application of dicationic ionic liquids in the synthesis of aliphatic thiazolidinone derivatives has been extensively explored in recent decades. Several reports have demonstrated the efficient synthesis of thiazolidinone derivatives using Chloride and acetate based ionic liquids under mild and solvent-free conditions [12-14]. For instance, Dicationic liquid-mediated multicomponent reactions have been shown to produce aliphatic thiazolidinone in high yields with improved selectivity and shorter reaction times [13, 15, 32].

Most research scholar nowadays focusing on aromatic synthesis strategies systems despite the advancement of synthesis of Aliphatic based thiazolidinone using DILs remains underexplored. Although, aliphatic compounds have more efficient catalytic system required for achieving the high yield and selectivity [3, 5, 44]. Therefore, the synthesis of aliphatic thiazolidinone using Dicationic ionic liquids is for the development of green and scalable employing methodologies and in significant and industrial interests.

Herein, our goal is study green and efficient method for the synthesis of aliphatic based thiazolidinone derivatives using as catalyst i.e. 1, 4 diaminobutane based dicationic ionic liquids (DCILs) these both catalyst and solvent have been employed. The best catalyst developed protocol offers. For the advancements, such as less reaction time, highest yield, and recyclability of Dicationic ionic liquid in environmentally benign conditions. The synthesized compounds were further characterization by using spectroscopic techniques including ¹H NMR, ¹³C NMR, FT-IR, and mass spectrometry for confirming their structural identity.

2. Experimental

2.1. Materials

All reagents and chemicals were analytical grade and used without further purification and commercially available, 1,4 Diaminobutane, 1- Methyl Imidazole, Chloroacetyl Chloride, Potassium Carbonate, Lithium Perchlorate and Potassium hexafluorophosphate were obtained from sigma Aldrich (India), sodium tetrafluoroborate, diethyl ether, Ethyl acetate, Sodium sulphate, Aldehydes, Primary aliphatic amines and Thioglycolic acid all chemical were purchased from S.D fine Chemicals and Co. Ltd. and Avra synthesis Pvt. Ltd. All the chemical were used as received unless it is specified all the glassware are special washed and dried under suitable temperature in Oven at 120 °C to 150 °C. TLC of the synthesized compound was taken on TLC Silica gel 60 F254 aluminum coated sheet. NMR Spectra of synthesized ionic liquids and products were recorded in

DMSO-d₆ on a Bruker Spectrometer operating at 400 MHz and chemical shifts are given in ppm downfield from TMS ($\delta=0.00$). FT-IR Spectra of the synthesized compounds were obtained by using Fourier Transform Infrared Spectrophotometer (Shimadzu, IR Affinity-1 Japan) in the wave range 400-4000 cm⁻¹ in the transmittance mode using KBr pellets.

2.2. Characterization

The Dicationic Ionic liquid catalyst and All synthesized Aliphatic based Thiazolidinone derivatives were characterized using analytical techniques as ¹H NMR and ¹³C NMR, ¹⁹F NMR, ³¹P NMR, IR, Mass, spectroscopy. Also the melting points were measured for all synthesized derivatives.

2.3. Method

2.3.1 Scheme 1: 3, 3'-((butane-1,4-diylbis(azanediyl))bis(2-oxoethne-2,1-diyl))bis(1-methyl-1H-imidazol-3-ium) dicationic Ionic Liquid.

Step - I: Alkylation of 1, 4 Diaminobutane

According to lit. [45] N, N'-(butane-1,4-diyl)bis(2-chloroacetamide) (2) known procedure with some minor modification and analytical data encounters with literature [15, 16].

Step-II: Synthesis of 3,3'-((butane-1,4-diylbis(azanediyl))bis(2-oxoethne-2,1-diyl))bis(1-methyl-1H-imidazol-3-ium) chloride dicationic Ionic Liquid. (2a)

According to lit. [46] N, N'-(butane-1,4-diyl)bis(2-chloroacetamide) (2) (2.41 g, 10 mmol) was loaded in R. B. flask with 1-methylimidazole (1.64 ml, 20 mmol) in 1:2 ration in 50 mL acetonitrile. Then taken on magnetic stirrer for next 24 hours under reflux condition. White sticky solid was obtained which separated by pouring of acetonitrile directly and rinsed ethyl acetate (3 x 20 ml) and finally followed by diethyl ether. After drying under high vacuum pure white solid of [EDAMIM] Cl₂ product was obtained. Yield: 72%, White solid; M.p 94°C; ¹H NMR (400MHz, D₂O) ($\delta=0.00$), 8.765 (s, 2H), 7.437 - 7.452 (d, 4H) 5.012 (m, 2H) 3.88 (s, 6H), 3.37 (dd, 4H), FT-IR (KBr, ν/cm^{-1}): 3177, 3055, 2828, 1668, 1564, 1354, 1276, 1180, 1075, 962, 874, 779, 700, 614, 578.

Step -III: Metathesis of Anion

1) Synthesis of 3,3'-((butane-1,4-diylbis(azanediyl))bis(2-oxoethne-2,1-diyl))bis(1-methyl-1H-imidazol-3-ium) hexafluorophosphate dicationic Ionic Liquid. (2b)

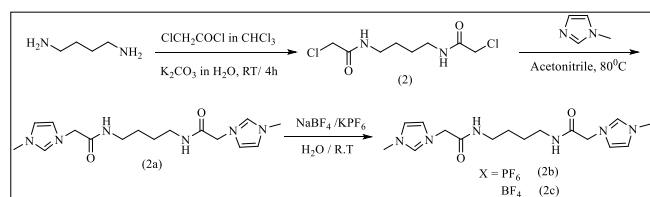
The crude product was dissolved in Deionised water in minimum quantity and then add Potassium Hexafluoride (Dissolve in small quantity of deionised water) 1:2 ratio drop wise at room temperature and take it for continuous stirring for next 4 hours. After the completion of the reaction Ionic Liquid get Filter and then dry under high Vacuum. The White Solid soft Crystal are obtained.

Yield: 92%, White solid; m.p 94 °C; ¹H NMR (400MHz, D₂O) ($\delta=0.00$), (m, 2H), 8.709 (m, 2H), 7.422 - 7.451 (d, 2H) 7.92 (m, 2H) 7.75 (m, 2H), 4.99 (j, 4H) 3.89 (s, 6 H), 3.37 (dd, 4H), ³¹P NMR (D₂O, 162 MHz) $\delta_P = -136.33$ to -158.20 (1P, sept, PF₆). ¹⁹F NMR (D₂O, 377MHz), $\delta_F = -71.90$ to -71.95 (6F, d, PF₆). FT-IR (KBr, ν/cm^{-1}): 3177, 3055, 2828, 1677, 1564, 1276, 1180, 1075, 962, 874, 779, 614, 578.

2) Synthesis of 3,3'-((butane-1,4-diylbis(azanediyl))bis(2-oxoethne-2,1-diyl))bis(1-methyl-1H-imidazol-3-ium) tetrafluoroborate dicationic Ionic Liquid. (2c)

The crude product was dissolved in Deionised water in minimum quantity and then add Sodium tetrafluoroborate (Dissolve in small quantity of deionised water) 1:2 ratio drop wise at room temperature and take it for continuous stirring for next 4 hours. After the completion of the reaction Ionic Liquid get Filter and then dry under high Vacuum. The White Solid soft Crystal are obtained.

Yield: 92%, White solid; m.p 95 °C; ¹H-NMR (400MHz, DMSO-*d*₆/ TMS): δ = 8.730 (s, 1H) 7.417 - 7.443 (s, 2H), -4.987 (m, 2H), 3.886- 4.022 (s, 3H), 3.223 (s, 2H), 1.513 (s, 3H) ¹⁹F-NMR (D₂O, 377MHz), δF = -130.65 - 130.81 (6F, d, PF₆). FT-IR (KBr, ν/cm⁻¹): 3177, 3055, 2828, 1677, 1564, 1276, 1180, 1075, 962, 874, 779, 614, 578.

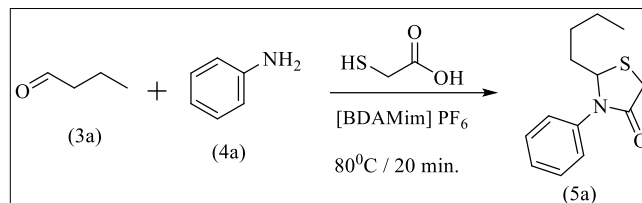


Scheme 1: Synthesis of 3,3'-((butane-1,4-diylbis(azanediyl))bis(2-oxoethne-2,1-diyl))bis(1-methyl-1H-imidazol-3-ium) [BDAMim] X₂ dicationic Ionic Liquid. (2a-c)

2.3.2. General Procedure for the Synthesis of Aliphatic Thiazolidinone derivatives (5a - b)

In a clean and dry 2 Neck RB flask take Propionaldehyde 10 mmol. (0.72) ml with p-toluidine 10 mmol (0.988) ml in 1:1:1.2 ratio dissolve with Thioglycolic acid of 0.89 ml (10 mmol). And then 10% of [BDAMim] (PF₆⁻)₂ Dicationic Ionic Ionic Liquid. Then the RB flask take on Stirring at 80 °C, Completion of reaction checked by TLC chromatography. After the completion of reaction cool it then the crude product was extracted in ethyl acetate (15

mL) and separated catalyst i.e. [BDAMim] (PF₆⁻)₂ was washed with ethyl acetate (3 x 5 mL), decanted and dried in oven at 60 °C. Finally, Thiazolidinone derivative was isolated by removing ethyl acetate using rotary evaporator and solid product was purified by recrystallization in Methanol / Ethanol or some compounds were purified by column chromatography. The melting points and spectroscopic data of these thiazolidinone derivatives are in good concurrence with the literature.



Scheme 2: Synthesis of 2-butyl-3-phenylthiazolidin-4-one

3. Result and Discussion

The developed method successfully yielded aliphatic thiazolidinone derivatives with excellent efficiency.

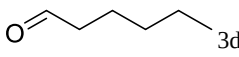
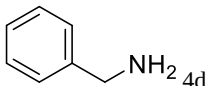
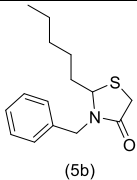
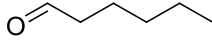
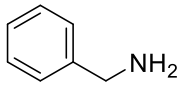
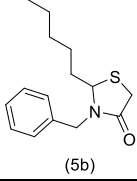
3.1 Catalytic Studies

The present work exhibits a novel Dicationic based ionic liquid catalyst and solvent. The safe and simple method efficiently yielded thiazolidinone derivatives by using aniline, aliphatic aldehydes and Thioglycolic acid in 1:1:1.2 equimolar amount.

The catalytic activity of [BDAMim] (PF₆⁻)₂ catalyst as well as solvent examined towards the synthesis of thiazolidinone derivatives. In order to optimize reaction condition, the model reaction of Butyraldehyde aniline and Thioglycolic acid was carried out [BDAMim] (PF₆⁻)₂ Dicationic ionic liquid. The activities of the catalyst were evaluated by varying the amount of catalyst, reaction temperature and time. (Table 1).

Table 1: Optimization of synthesis of Thiazolidinone Derivatives synthesis.

Entry	Aldehyde	Amine	Product	DILs	Time (Min.)	Yield%	Melting Point (°C)
1	3a	4a	(5a)	[EDAMim] (Cl ₂) ₂	100	50	118 -120
2	3a	4a	(5a)	[EDAMim] (PF ₆) ₂	20	92	122-126
3	3a	4a	(5b)	[EDAMim] (BF ₄) ₂	60	70	120 -122
4	3c	4c	(5b)	[EDAMim] (Cl ₂) ₂	125	70	110-112

5				[EDAMim] (PF ₆) ₂	30	98	115-118
6				[EDAMim] (BF ₄) ₂	70	85	112-115

Reaction conditions: *Aniline* (1.0 mmol), *Butyraldehyde* (1.0 mmol), and thioglycolic acid (1.2 mmol), DILs (10%)^b Isolated yields

In the present study, a sustainable and efficient methodology has been developed for the synthesis of aliphatic thiazolidinone derivatives using ionic liquids as green catalytic systems. A systematic comparison was carried out between monocationic ionic liquid (MIL) and two structurally distinct dicationic ionic liquids (DCILs), all based on environmentally benign anions such as chloride (Cl⁻) and acetate (CH₃COO⁻). The monocationic ionic liquid, represented by an 1,4 diaminobutane -based BF₄ system exhibited moderate catalytic performance, affording product yields in the range of 80-90% with relatively longer reaction times (60-180 min). MCILs developed as an alternative to conventional solvents. But due to their single

cationic center limiting to stabilize reactive intermediates. The dicationic ionic liquids [BDAMim] (PF₆)₂, 1,4 diaminobutane based with PF₆ anion) stabilizes reactive intermediate more effectively with enhancing catalytic activities. [BDAMim] (PF₆)₂ gives excellent yields (92-98%) in less time (20-40 Min.). 1,4 diaminobutane centers facilitate stronger hydrogen bonding with charge delocalization and effective stabilization of intermediate while synthesizing Schiff bases.

Nano catalyst and conventional while still outperforming the monocationic system, exhibited slightly lower efficiency (4 - 12 hrs and 3-6hrs) with (70-80% yield; 50-70% yield) compared to. To the MILs and DCILs may be due to the absence of an aromatic cationic system, leading to weaker intermolecular interactions and less effective catalytic activation.

Table 2: Advanced Comparison Table Catalyst performance

Catalyst Type	Yield%	Temp (°C)	Time (min.-hour)	Reusability	Environmental
Conventional (Acid / Base)	50-70	70-120	4-12 hrs	No	Toxic Nature Solvents
Catalyst (Nano)	70-80	60-100	3-6 hrs.	Moderate	Semi-green
Mono Ionic Liquid(MILs)	80-93	40-70	1-3 hrs	Yes (3-5 Cycles)	Green
Dicationic ILs (DILs)	90-98	R.T to 80	20 - 40 min	Yes (5-10 Cycles)	Highly green

Table No.2: is comparisons of Different ILs and DILs according to literature and novel [BDAMim] (PF₆)₂ and BF₄ based Dicationic Ionic Liquids.

Overall Comparisons of Dicationic ionic liquids exhibit significantly enhanced catalytic performance as compared to Monocationic ionic liquids, Nano catalyst and Conventional (Acid / Base) method in the synthesis of aliphatic thiazolidinone. Dicationic ionic liquids showed higher catalytic activity with their dual ionic nature provides improved interaction with reactants, leading to higher yields, faster reactions, and better sustainability as compared to conventional solvents and Monocationic ILs Recyclability up to 4-5 cycles, Therefore, DILs are more suitable for advanced green synthetic and Recyclability up to 5-10 cycles remain superior in catalytic applications.

3.2 Recyclability

The recyclability of the solvent/catalyst [BDAMim] (PF₆)₂ was investigated in the model reactions of Butyraldehyde, Aniline and thioglycolic acid. After every run, thiazolidinone was diluted with ethyl acetate, and the [BDAMim] (PF₆)₂ was obtained by solvent decantation. Any leftover traces of the non-ionic compounds were removed by washing with ethyl acetate and diethyl ether. After being vacuum-dried for three hours at 60 °C, the [BDAMim] (PF₆)₂ was used for the next run of the model reaction. Our catalyst is recyclable, as shown by Fig. 1,

where [BDAMim] (PF₆)₂ has shown good catalytic activity across seven runs without a noticeable drop in yield.

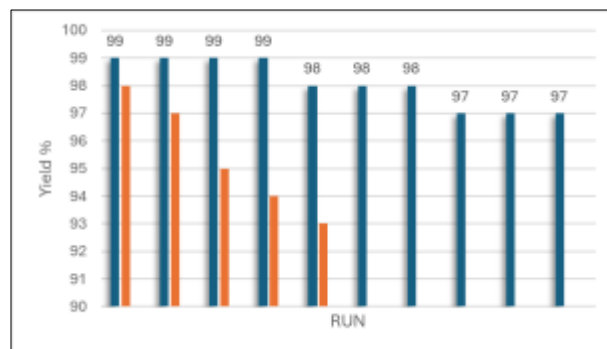
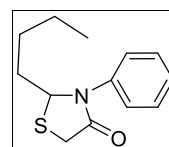


Fig 1: Comparative Recyclability of [BDAMim] (PF₆)₂ in Aliphatic Thiazolidinone synthesis

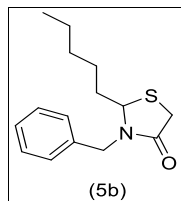
4. Spectral Data for synthesized products of Thiazolidinone.

2-butyl-3-phenylthiazolidin-4-one (5a)



Yield: 98%, Yellow solid; m.p 118-120°C; ¹H-NMR (400MHz, DMSO-*d*₆/TMS): δ = 7.1 - 7.4 (m, 4H), 5.10 - 5.24 (dd, 1H), 3.30-3.88 (s, 2H); ¹³C-NMR (100MHz, DMSO-*d*₆/TMS) δ = 170.07, 141.35, 127.11, 62., 60.15, 37.13, 32.00 FT-IR (KBr, ν/cm⁻¹): 2925, 1720, 1686, 1613, 1373, 1334, 1298, 1246, 1216, 1179, 1128, 1019, 899, 826, 780, 720, 569, 505.

3-benzyl-2-phenylthiazolidin-4-one (5b)



Yield: 98%, Yellow solid; m.p 110-112°C; ¹H-NMR (400MHz, DMSO-*d*₆/TMS): δ = 7.10 - 7.40 (m, 4H), 5.10 (dd, 1H), 3.88 (dd, 2H), 1.30 (t, 1H); ¹³C-NMR (100MHz, DMSO-*d*₆/TMS) δ = 170.50, 141.30, 62.67., 60.00, 58.90, 37.13, 32.00, 29.50; FT-IR (KBr, ν/cm⁻¹): 2923, 1721, 1686, 1614, 1374, 1298, 1246, 1216, 1128, 826, 780, 720, 569, 505.

5. Acknowledgement

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5. Conclusion

Recyclability studies further highlighted the advantages of dicationic ionic liquids. DCILs [BDAMim] (PF₆)₂ retained its catalytic activity over 5-10 cycles with negligible loss in yield, whereas [EDAMim] (BF₄)₂ showed moderate stability up to 3-5 cycles. In comparison, the monocationic ionic liquid displayed a significant decrease in the other acid / base or nano catalyst methods activity after 2-3 cycles. The strong foundation for development of next generation in heterocycles in green catalytic synthesis of aliphatic thiazolidinone. The synthesis strategies are continue to more attraction in future for the construction and developments are found in wide range of applications.

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