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Research Article

[NMP][HSO_4]-MEDIATED ENVIRONMENTALLY BENIGN SYNTHESIS OF 4-THIAZOLIDINONE DERIVATIVES

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ABSTRACT

A *N*-methyl-2-pyrrolidonium hydrogensulfate [NMP][HSO_4] bronsted acidic ionic liquid-promoted cyclocondensation-cyclization pathway has been established using one pot reaction of anilines, aldehydes and mercaptoacetic acid to give 4-thiazolidinone derivatives in good to promising yields using microwave irradiation. Applications for this protocol are easy workup, high yields, short reaction times, variability of functional groups, recyclability and solvent-free conditions.

Keywords:[NMP][HSO_4], Environmentally benign, Multicomponent reactions, 4-Thiazolidinones.

1. INTRODUCTION

Ionic liquids (ILs) are environment-friendly solvents because of their interesting properties and alternative to the harmful organic solvent. Furthermore, these are useful in the catalytic reaction [1], organic synthesis [2], because of their unique properties make them superior media for increasing selectivity, reactivity and recyclability. Various ionic liquids have been significantly used in heterocyclic synthesis as a solvent or catalyst [3].

Multicomponent reactions (MCRs) have been widely used for the synthesis of a wide range of heterocycles. As heterocyclic fragments are present in the majority of natural products and drug-like compounds, efficient synthesis of diverse heterocyclic compounds remains a key issue until now. MCR is one of the most effective synthetic techniques for the development of various heterocyclic compounds, applying subsequent trans-formation, cyclization and functionalization, leading to a diversity-oriented synthesis [4]. Thus, various medical chemists have been attracted by the development of multi-component reaction protocols for the synthesis of heterocyclic compounds.

4-Thiazolidinones and its derivatives have drawn considerable attention of the researchers in the recent years due to their medicinal properties. 4-Thiazoli-dinones is a medicinally significant pharmacophore with wide range of remarkable pharmacological activity such as anti-convulsant [5], anti-inflammatory [6], anti-microbial [7], anti-viral [8], antitumor [9], antidiabetic [10], antituberculosis [11], antiparasitic [12], analgesic [13], antidiarrhoeal [14], antiarthritic [15], cardio-vascular activity [16], anti-HIV [17] and FSH agonist [18]. Furthermore, 4-thiazolidinones conjugates also exhibit prominent cancer activity against several cancer cell lines such as antiproliferative activity against Reh and Nalm6 cells [19], antiapoptotic biocomplex (Bcl-XL-BH3) [20], breast cancer JSP-1 inhibitor [21], Cyclin B/CDK1 inhibitor [22], MCF-7 [23], integrin avb3 receptor [24], HT29 colon cancer cell line [25] and tumor necrosis factor (TNFα) [26]. Structures of representative bio-active 4-thiazolidinone motifs are disclosed in Fig. 1.

Due to the wide range of biological activities of 4-thiazolidinones, several synthetic pathways have been established for the synthesis of 4-thiazolidinone conjugates. Because of wide range of pharmacological activities, numerous catalytic routes have been established including, acetic acid [27], ammonium persulfate [28], acid catalysed [29], Bi[SCH_2COOH]$_3$ [30], Cd-Zr_4- [PO_4]$_6$ [31], DIPEA [32], DBSA [33], montmorillonite K-10 [34], *N*-methyl pyridinium tosylate [35], nano-Fe_3O_4@SiO_2 [36], silica gel [37], silica supported $CoFe_2O_4$@SiO_2/$PrNH_2$ [38], $SnCl_2$

[39], ionic liquid [bmim]OH [40] and Y[OTf]$_3$ [41]. However, the above well developed protocols have some drawbacks such as hazardous solvent, extended heating, separation, recycling of catalyst and tedious work-up procedure.

In addition, many of these processes employ organic solvents act as the reaction media. Hence, this protocol which overcomes these limitations requests a lot of interest for the scientist. Hence, the improvement toward fashionable reaction with reusability of catalyst, easy isolation of product, no waste is highly attractive. Recently, [NMP][HSO_4] was widely used as catalysts in different research areas. Reaction of HSO_4 with cation gives novel NMP based ionic liquids [42]. The large number of functionalized ILs has been considered for diverse purposes. Due to this wide range of applications, they are used as a suitable solvent for wide array of synthetic protocols [38]. The synthesis of NMP is based on ionic liquid *via* assembling the zwitterionic precursors to these functionalized acidic-SO_3H ionic liquid [43]. As per our study, the existential of this work is to begin efficient and rapid synthetic protocol for the synthesis of 4-thiazolidinone derivatives under ecofriendly conditions. As an extension of emerging efficient and economic strategy to expand biologically considerable active compounds, herein, we disclose first time microwave assisted solvent free synthesis of 4-thiazolidinone derivatives using NMP-based acidic ionic liquid to give good to prominent yields.

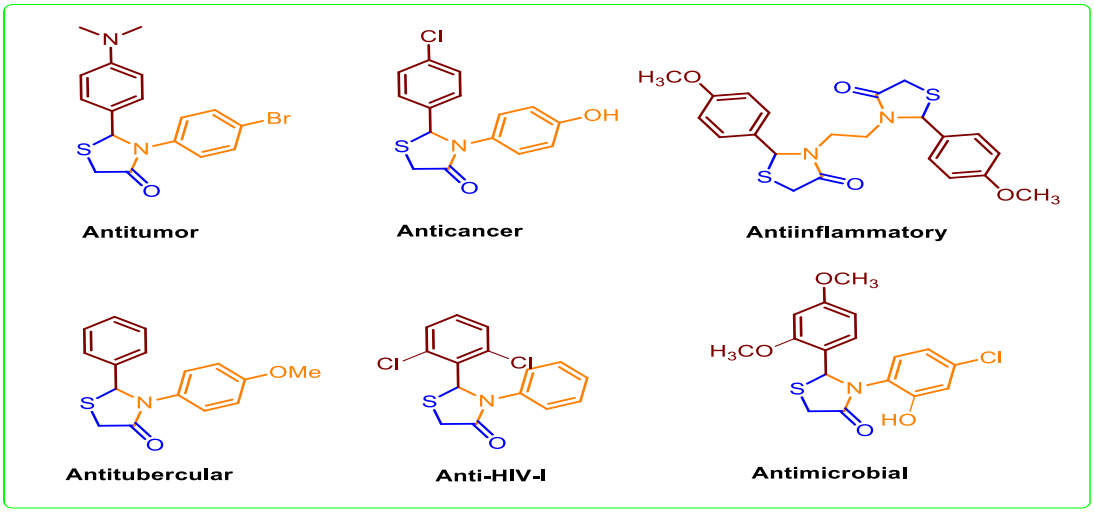


Fig. 1:Structure of bio-active 4-thiazolidinones conjugates

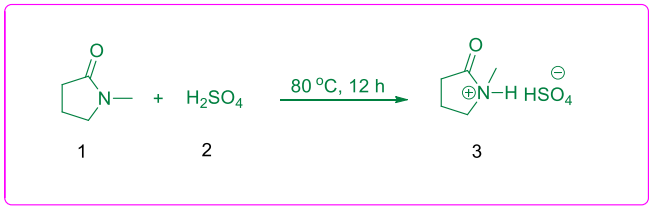
2. EXPERIMENTAL

2.1. Preparation of [NMP][HSO_4]

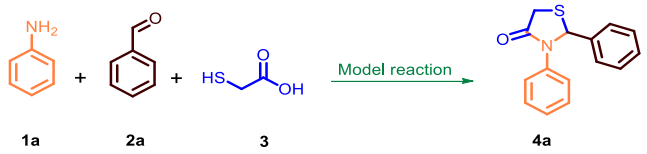
1-Methyl-2-pyrolidone (0.2mol) was charged into a 250-mL three-necked flask containing a magnetic stirrer. Then an equimolar amount of concentrated sulfuric acid (98 wt %) was added dropwise slowly into the flask at 80°C for 12 h. The mixture was washed by ether three times to remove non-ionic residues and dried under vacuum by a rotary evaporator to obtain the viscous clear [NMP][HSO_4] compound. The pH of the resulted ionic liquid (10% w/v) was determined and the obtained pH was equal to 1.2. Reaction details are shown in Scheme 1.

2.2. General Procedure for Synthesis of 4-thiazolidinone derivatives (4a-l)

A mixture of anilines (**1a**) (1 mmol), aryl aldehyde (**2a**) (1 mmol), mercaptoacetic acid (**3**) (1 mmol) and [NMP][HSO_4] 20 mol% was kept under microwave irradiation at 240 W for 8 min. The progress of the reaction was monitored by thin layer chromatography (*n*-Hexane/EtOAc 8:2). Further, ice cold water (10 mL) was added, stirred for 10 min. The obtained solid was filtered, washed with cold water to remove the ionic liquid. The obtained crude compounds were recrystallized using ethanol. The synthesized compound is confirmed by MP, ^{1}H NMR and ^{13}C NMR spectra.



Scheme 1:Synthesis of [NMP][HSO_4] ionic liquid



Scheme 2:Model reaction

3. RESULTS AND DISCUSSION

3.1. Characterization of compounds

3.1.1. 2,3-Diphenylthiazolidin-4-one (4a)

The **4a** was observed *via* cyclocondensation of **1a**, **2a** and **3** as white solid; Mp:130-132°C (lit.[46] 128-130°C); Yield:91%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):3.83 (d, 1H, *J* = 16 Hz), 3.97 (d, 1H, *J* = 16 Hz), 6.09 (s, 1H, S-CH-N), 7.20 (d, *J* = 7.9 Hz, 3H) and 7.22 (dd, *J* = 9.6, 4.9 Hz, 7H).

3.1.2. 2-phenyl-3-(p-tolyl)thiazolidin-4-one (4b)

The **4b** was observed *via* cyclocondensation of **1b**, **2a** and **3** as white solid; Mp:110-112°C (lit.[47] 110-112°C); Yield:89%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):2.27 (s, 3H, CH_3), 3.89 (d, 1H, *J* = 16 Hz), 3.99 (d, 1H, CH_2, *J* = 16 Hz), 6.05 (s, 1H, S-CH-N), 7.16 (m, 2H), 7.23 (d, *J* = 8.3 Hz, 2H) and 7.38-7.26 (m, 5H).

3.1.3. 3-(4-fluorophenyl)-2-phenyl thiazolidin-4-one (4c)

The **4c** was observed *via* oxidative cyclocondensation of **1c**, **2a** and **3** as white solid; Mp:115-116°C (lit.[48] 115-116°C); Yield:84%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):3.72 (d, 1H, *J* = 16 Hz), 3.82 (d, 1H, CH_2, *J* = 16 Hz), 6.05 (s, 1H, S-CH-N), 6.96 (dd, *J* = 24.1, 6.9 Hz, 2H) and 7.21-7.04 (m, 7H).

3.1.4. 3-(4-Chlorophenyl)-2-phenylthiazolidin-4-one (4d)

The **4d** was observed *via* cyclocondensation of **1d**, **2a** and **3** as white solid; Mp:110-112°C (lit.[48] 110-112°C); Yield:86%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):3.82 (d, 1H, *J* = 16 Hz), 3.89 (d, 1H, CH_2, *J* = 16 Hz), 5.98 (s, 1H, S-CH-N), 7.17-7.02 (m, 5H), 7.31-7.27 (m, 2H) and 7.41-7.26 (m, 2H).

3.1.5. 3-(4-bromophenyl)-2-phenylthiazolidin-4-one (4e)

The **4e** was observed *via* cyclocondensation of **1e**, **2a** and **3** as white solid; Mp:114-115°C (lit.[48] 115-116°C); Yield:85%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):3.86 (d, 1H, *J* = 16 Hz), 3.97 (d, 1H, CH_2, *J* = 16 Hz), 6.07 (s, 1H, S-CH-N), 7.12 (dd, *J* = 6.6, 4.7 Hz, 2H) and 7.38-7.18 (m, 7H).

3.1.6. 2-(4-chlorophenyl)-3-(p-tolyl)thiazolidin-4-one (4f)

The **4f** was observed *via* cyclocondensation of **1f**, **2b** and **3** as yellow solid; Mp:164-166°C; Yield:88%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):2.28 (s, 3H, CH_3), 3.84 (d, 1H, *J* = 16 Hz), 3.98 (d, 1H, CH_2, *J* = 16 Hz), 6.04 (s, 1H, S-CH-N), 7.01 (dd, *J* = 8.2, 2.8 Hz, 2H), 7.09 (d, *J* = 6.2 Hz, 2H) and 7.26 (dd, *J* = 8.0, 4.4 Hz, 4H); ^{13}C NMR (101 MHz, cdcl$_3$) δ 20.31, 32.70, 64.30, 124.95, 128.37, 129.19, 133.88, 134.00, 136.11, 137.44 and 170.16.

3.1.7. 3-Phenyl-2-(p-tolyl)thiazolidin-4-one (4g)

The **4g** was observed *via* cyclocondensation of **1a**, **2c** and **3** as brown solid; Mp:118-120°C (lit.[46] 116-118°C); Yield:91%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):2.29 (s, 3H, CH_3), 3.89 (d, 1H, *J*= 16 Hz), 4.02 (d, 1H, CH_2, *J* = 16 Hz), 6.05 (s, 1H, S-CH-N), 7.05 (dt, *J* = 6.4, 5.1 Hz, 4H) and 7.40-7.22 (m, 5H).

3.1.8. 3-(4-Chlorophenyl)-2-phenylthiazolidin-4-one (4h)

The **4h** was observed *via* cyclocondensation of **1d**, **2c** and **3** as white solid; Mp:110-112°C (lit.[46] 110-112°C); Yield:88%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):3.98 (d, 1H, CH_2, *J* = 16 Hz), 2.25 (s, 3H, CH_3), 3.89 (d, 1H, *J* = 16 Hz), 6.13 (s, 1H, S-CH-N), 7.16-6.93 (m, 4H), 7.45 (d, *J* = 8.2 Hz, 2H), 8.13 (d, *J* = 8.5 Hz, 2H).

3.1.9. 3-(4-chlorophenyl)-2-(4-nitrophenyl)thiazolidin-4-one (4i)

The **4h** was observed *via* cyclocondensation of **1d**, **2d** and **3** as white solid; Mp:150-152°C; Yield:79%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):3.94 (d, 1H, *J* = 16 Hz), 4.04 (d, 1H, CH_2, *J* = 16 Hz), 6.17 (s, 1H, S-CH-N), 7.14 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.55 (d, *J* = 8.6 Hz, 2H) and 8.16 (d, *J* = 8.2 Hz, 2H).

3.1.10. 3-(4-methoxyphenyl)-2-phenylthiazolidin-4-one (4j)

The **4j** was observed *via* cyclocondensation of **1a**, **2g** and **3** as yellow solid; Mp:62-64°C (lit.[48] 61-62°C); Yield:95%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):3.82 (d, 1H, *J* = 16 Hz), 3.86 (s, 3H, , CH_3), 3.96 (d, 1H, CH_2, *J* = 16 Hz), 6.10 (s, 1H, S-CH-N), 7.24-7.12 (m, 5H), 7.47 (d, *J* = 8.4 Hz, 2H) and 7.82 (d, *J* = 8.2 Hz, 2H).

3.1.11. 2,3-di-p-tolylthiazolidin-4-one (4k)

The **4k** was observed *via* cyclocondensation of **1b**, **2c** and **3** as white solid; Mp:122-124°C (lit.[48] 121-123°C); Yield:90%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):2.30 (s, 6H, CH_3), 3.84 (d, 1H, *J*=16 Hz), 3.95 (d, 1H, CH_2, *J* = 16 Hz), 6.14 (s, 1H, S-CH-N), 7.54 (d, *J* = 8.1 Hz, 4H) and 7.84 (d, *J* = 8.2 Hz, 4H).

3.1.12. 2-(4-methoxyphenyl)-3-phenylthiazolidin-4-one (4l)

The **4l** was observed condensation of **1b**, **2c** and **3** as white solid; Mp:98-100°C (lit.[48] 97-99°C); Yield:92%; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):3.83 (s, 3H, -OCH_3), 3.97 (d, 1H, *J* = 16 Hz), 4.12 (d, 1H, CH_2, *J* = 16 Hz), 6.10 (s, 1H, S-CH-N), 7.40-7.26 (m, 5H), 7.64 (d, *J* = 8.3 Hz, 2H) and 7.88 (d, *J* = 8.1 Hz, 2H).

3.2. Chemistry

To attain optimized reaction conditions procedure based on the reaction of aniline (1a), benzaldehyde (2a) (1 mmol) and mercaptoacetic acid (3) (1 mmol) as representative reaction (Scheme 1), we screened solvents, catalyst loading, temperatures and the details of this study are illustrated in Table 1.

Table 1:Screening study for the Synthesis of 4-Thiazolidinone (4a)[a]

Entry	Catalyst	Time (min)	Yield[b] (%)
1	-	30	42
2	*p*-TSA	30	71
3	Sulfamic acid	20	62
4	Sulfanilic acid	30	55
5	Boric acid	20	62
6	Citric acid	30	56
7	Phosphotungstic acid	30	51
8	Xanthan sulfuric acid	20	58
9	Silica sulfuric acid	20	69
10	CSA	30	71
11	[NMP][HSO_4]	8	95

*[a]Reaction conditions:aniline **1a** (1 mmol), benzaldehyde **2a** (1 mmol), mercaptoacetic acid **3** (1 mmol) and [NMP][HSO_4] (20 mol%) under microwave (MW = 240 W). [b]Isolated yields*

When the reaction was performed without using catalyst it gives lower yield of titled product after 30 min (Table 1, entry 1). We perform model reaction for the synthesis of compounds 4a using different catalysts such as Boric acid, Citric acid, CSA, Sulfanilic acid, Sulfamic acid, *p*-TSA, Phosphotungstic acid, Silica sulfuric acid, [NMP][HSO_4] and Xanthan sulfuric acid (Table 1, entries 2-11). Thus, [NMP][HSO_4] catalyst was screened for model reaction at room temperature to give 40% yield. When the reaction was performed using [NMP][HSO_4] under microwave it gives 95% yield of final product 4a (Table 1, entries 11). Therefore, above results suggest that [NMP][HSO_4] act as a green and excellent catalyst for the synthesis of 4-thiazolidinones and results are disclose in Table 1.

Further, we examine the efficiency of bronsted acidic [NMP][HSO_4] ionic liquid for the synthesis of 4-thiazolidinones derivatives. Using different loading of [NMP][HSO_4] catalyst on model reaction results change in the yield of 4-thiazolidinone **4a**. Catalyst concentration study suggest that 20 mol% of [NMP][HSO_4] catalyst is effective for synthesis of 4-thiazolidnone and results are disclose in (Table 2, entry 4).

Table 2:Effect of [NMP][HSO_4] Catalyst Loading[a]

Entry	Catalyst (mol %)	Time (min)	Yield[b] (%)
1	5	20	60
2	10	15	70
3	15	12	80
4	20	8	95
5	25	8	95

*[a]Reaction conditions:aniline **1a** (1 mmol), benzaldehyde **2a** (1 mmol), thioglycolic acid **3** (1 mmol) and [NMP][HSO_4] under microwave irradiation. [b]Isolated yield*

Next, we examine effect of microwave power on the model reactions. This examination results suggest that 240 W power levels effective for the synthesis of 4-thiazolidnone **4a** and results are disclosed (Table 3, entry 3).

Table 3:Effect of microwave power levels for the synthesis of 4a[a]

Entry	Power levels in Watt	Time[b] (min)	Yield[c]
1	140	12	68
2	210	10	85
3	240	8	95
4	280	8	95

*[a]Reaction conditions:aniline **1a** (1 mmol), benzaldehyde **2a** (1 mmol), thioglycolic acid **3** (1 mmol) in the presence of [NMP][HSO_4] 20 mol% under microwave irradiation. [b]Reaction progress monitored by TLC. [c]Isolated yield*

Enormously excellent protocol to greener and economic synthesis is recyclability and recovery of catalyst. Due to this, we screen the recyclability and

recovery of catalyst. This recyclability and recovery study results confirm that our NMP-based bronsted acidic ionic liquid is promising for the synthesis of 4-thiazolidnone without loosing its catalyst efficiency and results are disclosed in (Table 4, entry 2-5).

Table 4:Recovery and Reusability of bronsted acidic [NMP][HSO_4] catalyst for model reaction

Entry	Run	Time[a] (min)	Yield[b]
1	fresh	8	95
2	2	8	95
3	3	8	84
4	4	8	82
5	5	8	80

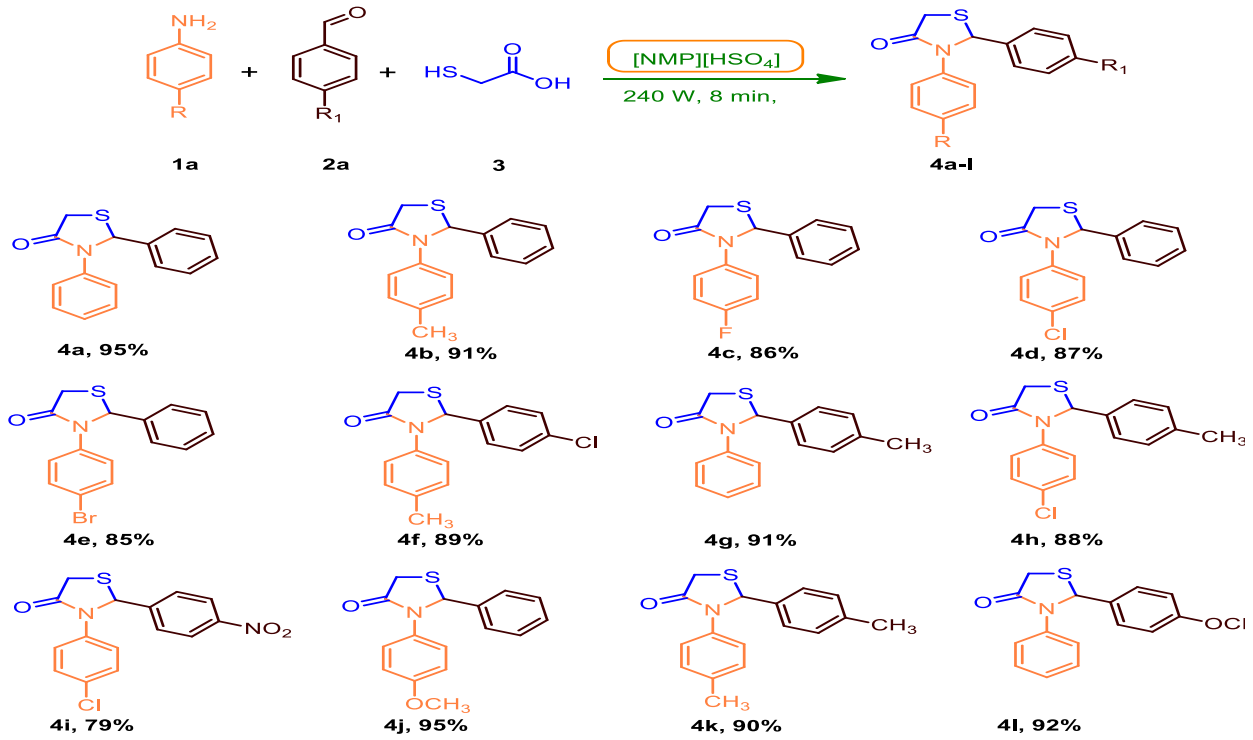
[a]*Reaction progress monitered by TLC.* [b]*Isolated yield*

We also examine the comparative study [NMP][HSO_4] catalyst with other reported protocol for the synthesis of 4-thiazolidinones. The study suggests that [NMP][HSO_4] is prominent catalyst for the efficient and facile synthesis of 4-thiazolidnones and results are disclosed (Table 5, entry 9).

The structural elucidation of synthesize **4f** compound was confirmed by ^{1}H and ^{13}C NMR analysis. In ^{1}H NMR spectra, the peak was observed at 2.28 δ ppm for the CH_3 group. The peak observed at doublet of a doublet 4.00-3.86 ppm due to the presence of -CH_2 protons in titled compound. The C-H proton of the 4-thiazolidinone ring was observed at singlet at δ 6.04 ppm. In ^{13}C NMR spectra, peak observed at 32.70, 64.30 and 170.1 ppm for the CH, CH_2 and C=O bond present in synthesized compounds.

Table 5: Comparative study of [NMP][HSO_4] with Reported Catalysts

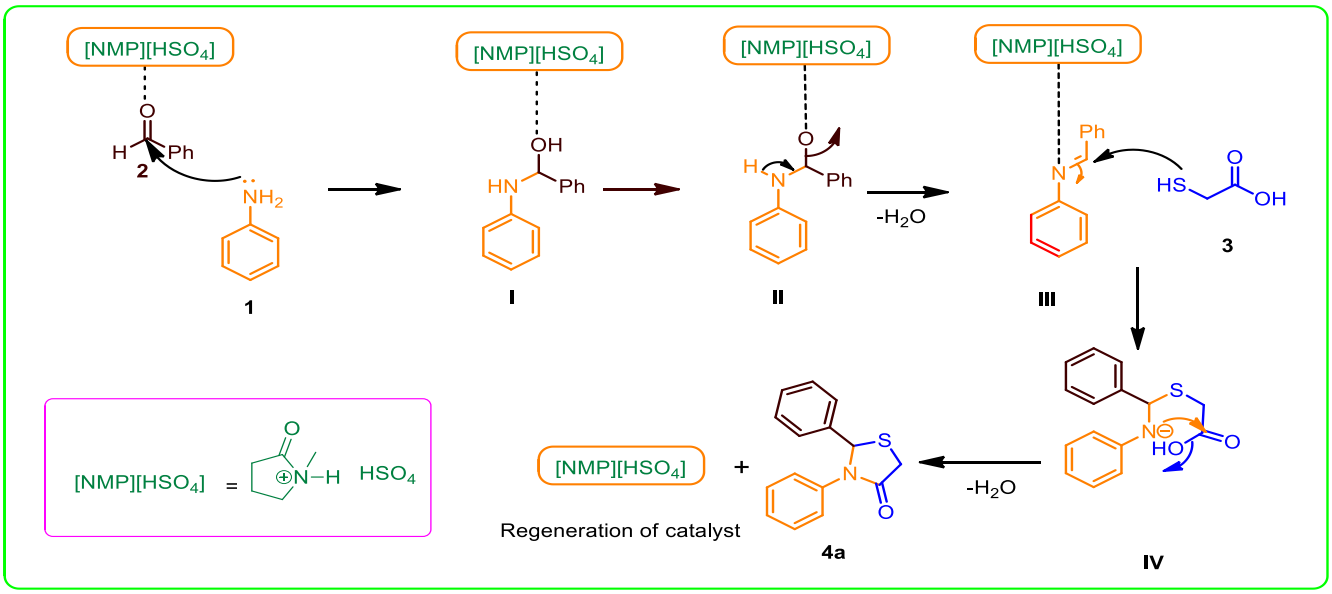
Entry	Catalyst	Time (min)	Yield (%)	condition	Ref.
1	[bmim][PF_6]	9 h	80	80°C	[44]
2	[bmim][BF_4]	1.7 h	82	80°C	[44]
3	[MOEMIM]TFA	9 h	90	80°C	[44]
4	$HClO_4$-SiO_2	5 h	85	PhMe/100	[45]
5	TfOH-SiO_2	5 h	72	PhMe/100	[45]
6	H_2SO_4-SiO_2	5 h	55	PhMe/100	[45]
7	Silica gel, DCM	6 h	96	DCM/RT	[37]
8	$Bi(SCH_2COOH)_3$	2 h	90	70°C	[40]
9	[NMP][HSO_4]	8 min	95	240W	Present work



Scheme 3: Substrate scope for the synthesis of 4-Thiazolidinones using [NMP][HSO_4] catalyst

3.3. Plausible Reaction Mechanism

Reaction mechanism for the synthesis of 4-thiazolidinones employing [NMP][HSO_4] is catalyst. In first step, benzaldehyde is activated followed by nucleophilic substitution of aniline which results formation of **I** intermediate. In next step, removal of water molecules from intermediates **I** with the help of [NMP][HSO_4] gives imine product **II**. In the third step, intermediate **III** reacts with **3** and afforded cycloaddition product **IV**. Further, intramolecular cyclization occurs to form final product **4a** *via* removal of H_2O molecule and regeneration of catalyst. Detailed reaction mechanism is disclosed in Scheme 4.



Scheme 4: Reaction mechanism for the synthesis of compound 4a

4. CONCLUSION

A highly efficient and environmentally benign protocol has been developed for the synthesis of 4-thiazolidinones using an recoverable and inexpensive [NMP][HSO_4] under microwave irradiation at 240 W for 8 min. This reaction protocol has many more advantages, such as uniqueness, high atom efficiency, clean reaction profiles, mild reaction condition, ecofriendliness, simple workup and without using any hazardous solvents.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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