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EXPERIMENTAL PAPER



Greener Synthesis of 3,4-Disubstituted Isoxazole-5(4H)-ones in a Deep Eutectic Solvent

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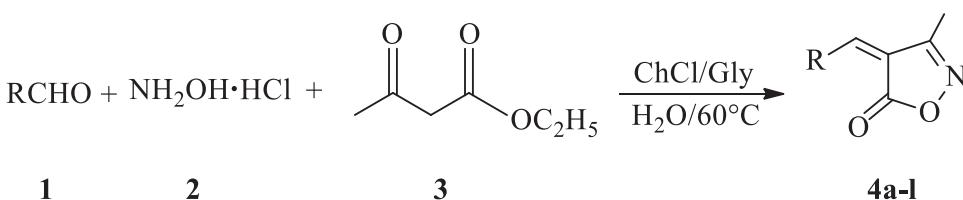
Developing a cost effective and environmentally benign solvent system is of the utmost importance in the chemical industry. One current proposal is the replacement of conventional hazardous volatile organic solvents by nonvolatile alternatives so that they do not emit toxic or flammable vapors over a wide range of temperatures.¹ During the past few years, some green solvents have been used. These include water,² supercritical fluids,³ perfluorinated solvents,^{4–5} glycerol and its derivatives,⁶ bio-based solvents,⁷ and ionic liquids (ILs).^{8–9} Notwithstanding their value, the use of these solvents is still in its early stages and is limited by a number of concerns.

Deep eutectic solvents (DESs), also known as deep eutectic ionic liquids (DEILs), or low-melting mixtures (LMMs), or low transition temperature mixtures (LTTMs) in the literature, have become more attractive due to their interesting properties and benefits. Among these are the low cost of components, ease of preparation, tunable physico-chemical properties, negligible vapor pressure, nontoxicity, bio-renewability and biodegradability. In fact, DESs have several features in common with ionic liquids. They have a large number of molecular components which are typically hydrogen bond donors. They are more industrially promising than ionic liquids due to their low cost and tolerance to humidity, thus avoiding potential problems of hydrolysis or hygroscopicity.^{10–11} DESs are defined as combinations of two or three safe and cheap components which are capable of self-association, to form a eutectic mixture, which is a liquid at temperatures lower than 100 °C, with a melting point lower than that of each of the individual components.¹²

Multicomponent reactions (MCRs) have emerged as a powerful strategy for the synthesis of numerous desirable chemical compounds, notably including natural products and biologically active compounds. Moreover, MCRs have such advantages as operational simplicity, high atom-economy, reduced production of waste, and the avoidance of intricate purification processes.^{13–16}

Carrying out MCRs in water as the reaction medium would be one of the most suitable methods, going a long way toward the fulfilment of the goals of green chemistry. Use of water not only diminishes the risk of organic solvents but also improves the rate of many chemical reactions.^{17–18}

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Scheme 1. Synthesis of 3,4-disubstituted isoxazole-5(4H)-one derivatives.

Isoxazole derivatives are important heterocyclic compounds that possessing numerous pharmacological and biological activities such as anti-HIV,¹⁹ antifungal,²⁰ analgesic,²¹ antitumor,²² COX-2 inhibitory,²³ antiviral,²⁴ and antioxidant²⁵ properties.

Several procedures have been reported in the literature for the synthesis of 3,4-disubstituted isoxazol-5(4H)-ones. Among these we may note Fe₂O₃ nanoparticles,²⁶ citric acid,²⁷ sodium silicate,²⁸ sodium benzoate,²⁹ sodium sulfide,³⁰ boric acid,³¹ potassium phthalimide (PPI),³² phthalimide N-oxyl salts,³³ Ag/SiO₂,³⁴ DABCO,³⁵ pyridine,³⁶ 2-hydroxy-5-sulfobenzoic acid (2-HSBA),³⁷ Dowex1-x8OH resin,³⁸ montmorillonite K10 supported Sn^{II} composite (Sn^{II}-MontK10),³⁹ imidazole,⁴⁰ hydroxyapatite (HAp) nanoparticles,⁴¹ K₂CO₃,⁴² and LiBr.⁴³

It is thus of considerable interest that DESs based on choline chloride have been used in an increasing number of organic reactions.^{44–47} As part of our ongoing research program in green chemistry,^{48–59} we now report on the use of a DES based on choline chloride and glycerol (ChCl/Gly) for the synthesis of 3,4-disubstituted isoxazole-5(4H)-ones in water (Scheme 1). To the best of our knowledge, there are no citations in the literature on the synthesis of compounds 4 using DESs. We sought an organic reaction enhancement methodology, which would be faster and greener than other methods.

In order to obtain the best conditions for our reaction, we optimized the parameters as listed in Table 1. It is observed that water/60 °C gave the best result for this transformation in the presence of ChCl/Gly (entry 1). This reaction was carried out in the lone presence of either choline chloride or glycerol and gave low yields after 20 min (entries 9, 10). In the absence of ChCl/Gly, no significant product formation was observed even after 40 min (entry 11).

The optimized conditions were then used for different aldehydes to test the generality of the work. The results are summarized in Table 2. Substituted aromatic aldehydes containing electron-withdrawing groups or electron-donating groups underwent this multicomponent synthesis with hydroxylamine hydrochloride and ethyl acetoacetate to afford 3,4-disubstituted isoxazole-5(4H)-ones in excellent yields (mean yield 92%).

In order to show the characteristics of this method, we have compared our results with results from other sources in the literature. The data listed in Table 3 show that the DES ChCl/Gly was a very suitable catalyst for this transformation, with dramatically shorter reaction times than some methods. It is also noteworthy that ChCl/Gly, is inexpensive compared with other catalysts.

In conclusion, a green and simple synthesis of 3,4-disubstituted isoxazole-5(4H)-ones using the DES ChCl/Gly is reported. The advantages of the present procedure are experimental simplicity, easy work-up, and high yields of products. Furthermore, this procedure is sustainable because of the efficiency of the readily available and biodegradable deep eutectic solvent.

Table 1. Optimization of reaction conditions for **4a**.^a

The reaction scheme shows the synthesis of compound **4a** from benzaldehyde (**1**), hydroxylamine hydrochloride (**2**), and ethyl acetoacetate (**3**). Compound **4a** is a 3,4-disubstituted isoxazole-5(4H)-one derivative.

Entry	Catalyst (ml or g)	Conditions	Time (min)	Yield (%)
1	ChCl/Gly (0.2 ml)	Water/60 °C	20	95
2	ChCl/Gly (0.2 ml)	Ethanol/60 °C	20	78
3	ChCl/Gly (0.2 ml)	Water/Ethanol (2:1, v/v)/60 °C	20	89
4	ChCl/Gly (0.2 ml)	Solvent-free/60 °C	20	64
5	ChCl/Gly (0.2 ml)	Water/50 °C	20	90
6	ChCl/Gly (0.2 ml)	Water/rt	20	35
7	ChCl/Gly (0.3 ml)	Water/60 °C	20	92
8	ChCl/Gly (0.1 ml)	Water/60 °C	20	84
9	ChCl (0.04 g)	Water/60 °C	20	21
10	Gly (0.1 ml)	Water/60 °C	20	48
11	— ^b	Water/60 °C	40	— ^c

^aReaction conditions: benzaldehyde (1 mmol), hydroxylamine hydrochloride (1 mmol) and ethyl acetoacetate (1 mmol) in 0.1 ml water.

^bThis reaction was carried out in the absence of ChCl/Gly.

^cNo product **4a** observed.

Experimental section

All starting materials and DES components were commercially available and purchased from Merck. Melting points were determined on Electrothermal 9200 apparatus and are uncorrected. IR spectra were recorded as KBr pellets on a Shimadzu 435-U-04 spectrophotometer. ¹H and ¹³C NMR spectra were determined on a Bruker DRX-300 Avance spectrometer using CDCl₃ or DMSO-d₆, and shifts are given in ppm downfield from tetramethylsilane (TMS) as an internal standard. All the reactions were monitored with thin layer chromatography (silica gel, eluting solvents: n-hexane/ethyl acetate) and UV light as detecting agent.

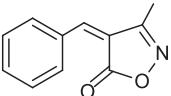
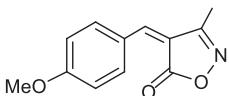
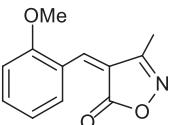
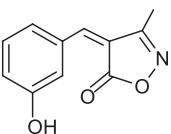
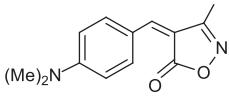
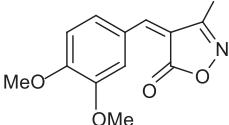
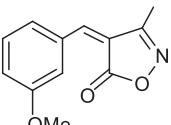
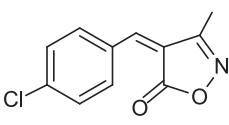
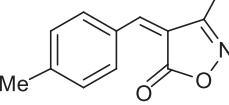
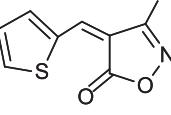
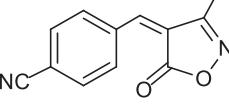
Preparation of ChCl/Gly

The preparation of the DES involved the reaction of choline chloride (1 mol) with glycerol (2 mol) at 100 °C in a flask with stirring for 2 h to produce a clear solution. Thus was suitable for the reactions without purification.¹¹

General procedure for the synthesis of 3,4-disubstituted isoxazole-5(4H)-ones (**4a-l**)

ChCl/Gly (0.1 ml) was added to a mixture of the aldehyde (1 mmol), ethyl acetoacetate (1 mmol) and hydroxylamine hydrochloride in a test tube. Water (0.1 mL) was added and the reaction mixture was stirred and heated at 60 °C using an oil bath for heating (for solid aldehydes, 10 drops of ethanol was added). The progress of reaction was controlled by TLC (silica gel, n-hexane/ethyl acetate: 4/1). After completing the reaction, 5 mL water was added to the reaction mixture and the solid was filtered. The products were recrystallized from ethanol (96%) to give pure compounds **4a-l**. All of the products

Table 2. Green synthesis of 3,4-disubstituted isoxazole-5(4H)-ones (**4a-I**) by ChCl/Gly in water.

Entry	Products	Yield (%)	Time (min)	mp (°C)	
				Found	Lit.
4a		95	20	140-141	141-142 ⁴¹
4b		94	20	174-175	173-174 ⁴¹
4c		90	25	139-140	137-139 ³⁷
4d		93	20	201-203	200-201 ²⁹
4e		95	20	218-220	220-221 ⁴¹
4f		92	20	135-137	134-135 ⁴⁰
4g		90	25	134-135	130-132 ⁴²
4h		95	25	212-214	210-211 ²⁹
4i		95	20	133-135	134-136 ²⁸
4j		90	20	142-144	143-144 ⁴¹
4k		92	20	195-197	193-194 ⁴³

(continued)

Table 2. Continued.

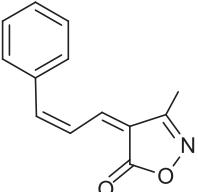
Entry	Products	Yield (%)	Time (min)	mp (°C)	
				Found	Lit.
4l		85	20	178-180	180-182 ³⁸

Table 3. Comparison of methods for the synthesis of 3,4-disubstituted isoxazole-5(4H)-ones.

Compounds	Conditions	Time (min)	Yield (%)
4a	ChCl/Gly/Water/30 °C (present work)	20	95
	Citric acid/water/rt ²⁷	480	90
	Sodium silicate/water/rt ²⁸	150	89
	Sodium sulfide/ethanol/rt ³⁰	150	80
	Ag/SiO ₂ /water/rt ³⁴	60	93
	2-HSBA/water/rt ³⁷	110	87
	Nano-MMT-Sn/water/ultrasonication/30 °C ³⁹	40	92
	K ₂ CO ₃ /water/reflux ⁴²	180	70
4b	ChCl/Gly/Water/30 °C (present work)	20	94
	Citric acid/water/rt ²⁷	300	90
	Sodium benzoate/water/rt ²⁹	90	87
	Ag/SiO ₂ /water/rt ³⁴	60	90
	2-HSBA/water/rt ³⁷	80	92
	Dowex1-x8OH/water/rt ³⁸	60	95
	Imidazole/water/ultrasonication/30 °C ⁴⁰	25	95
	K ₂ CO ₃ /water/reflux ⁴²	60	83

were known compounds and were identified by matching their melting points with those reported in the literature cited in **Table 2**. Characterization data for some representative compounds are provided below.

3-Methyl-4-(3-methoxyphenyl)methylene-isoxazole-5(4H)-one (4g)

¹H NMR (300 MHz, DMSO-d₆): 2.38 (s, 3H, CH₃), 3.83 (s, 3H, OMe), 7.22-7.25 (m, 1H, ArH), 7.48-7.52 (m, 1H, ArH), 7.90-7.91 (m, 1H, ArH), 7.93 (s, 1H, =CH), 8.10 (s, 1H, ArH); ¹³C NMR (75 MHz, DMSO-d₆): 11.70, 56.71, 117.97, 119.47, 120.70, 127.12, 130.36, 134.14, 152.06, 159.50, 162.58, 166.30; IR (KBr, cm⁻¹): 3482, 3098, 2981, 1741, 1622, 1477, 1571, 1440, 1278, 1170, 1051, 877, 775, 680.

3-Methyl-4-(4-cyanophenyl)methylene-isoxazole-5(4H)-one (4k)

¹H NMR (300 MHz, DMSO-d₆): 2.26 (s, 3H, CH₃), 7.15 (d, 2H, ArH), 7.56 (s, 1H, =CH), 8.52 (d, 2H, ArH); ¹³C NMR (75 MHz, DMSO-d₆): 11.74, 115.13, 115.25, 115.69, 126.20, 137.37, 151.71, 161.72, 164.72, 169.08; IR (KBr, cm⁻¹): 3462, 3090, 2978, 2223, 1731, 1590, 1434, 1279, 1170, 1021, 873, 560.

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