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Passerini three-component cascade reactions in deep eutectic solvent: an environmentally benign and rapid system for the synthesis of α-acyloxyamides

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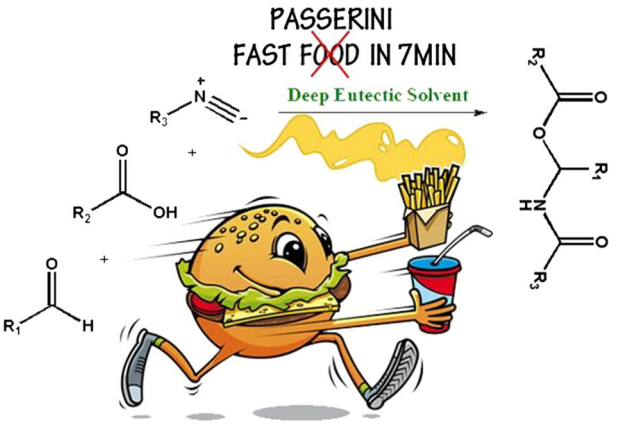
Abstract A greener synthesis of α-acyloxyamides was achieved by one-pot three component cascade Passerini reactions in a deep eutectic solvent (DES). Reactions proceeded rapidly and afforded the corresponding α-acyloxyamides by a variety of aldehydes, acids and isocyanides in excellent yields. The DES has the advantages of easy workup, high yield, and an environmentally benign and, moreover, shortened time as the greatest exclusivity of this system compared with previously reported methods.

Graphical Abstract Passerini three-component cascade reaction in a deep eutectic solvent: an environmentally benign and rapid system for the synthesis of α-acyloxyamides.



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Introduction

What makes a solvent green? The prevalent opinion is that the ideal green solvent is safe for both the human beings and the environment and its use and manufacture are sustainable [1]. Environmental and health issues are quickly emerging as a major challenge for the twenty-first century with the basic aspects of efficiency and economy being relevant to all aspects of science. From a synthetic chemist's viewpoint, this emphasis on environmental and health issues is leading to an increased focus on developing green efficient strategies for the synthesis of materials without using any hazardous solvents or reagents. For this inspiration, scientists have tried to replace toxic solvents with eco-friendly materials.

A closely related class of solvents with physical properties and phase behaviors very similar to those of room temperature ionic liquids (RTILs) are deep eutectic solvents (DESs), which were developed by Abbott et al. [2, 3]. The most common DESs are based on choline chloride (ChCl) and hydrogen-bond donors such as urea, citric acid, sugar, and glycerol. DESs are attractive alternatives to RTILs, as they are less expensive, more synthetically accessible, nontoxic and biodegradable [4, 5]. For instance, the preparation of DES in a pure state is considerably easier and economically viable as it shows 100 % atom economy with satiety to post-synthesis purification that was used for the ILs.

Heretofore so much chemical processing such as extraction [6], polymerizations [7], biomass processing [8], materials synthesis [9], and organic reactions [10–18] have been applied in DES medium. Quite recently a review has reported information about Deep Eutectic Solvents (DESs) and their applications in metal electrodeposition, metal extraction, synthesis of nanoparticles, gas adsorption, and synthesis applications [19].

Multicomponent reactions (MCRs) are useful and powerful tools in which three or more different starting materials react to form novel and complex molecules in a one-pot strategy with great efficiency and atom economy. The development of such processes in which several bonds are formed without isolation of intermediates receives considerable attention for the preparation of structurally diverse libraries of drug-like poly-functional compounds [20–24]. Isocyanides have emerged as a magic compound in organic and medicinal chemistry, allowing the formation of different molecular architectures from readily accessible precursors in a convergent manner and also have recently gained attention in macromolecular synthesis [25–27]. The ability of isocyanides to undergo facile addition with both nucleophiles and electrophiles makes it a popular reactant class for the development of novel MCRs. Because of the unique reactivity of the isocyanide functional groups, isocyanide-based multi-component reactions (IMCRs) are among the most versatile, in terms of number and variety of compounds that can be generated [28–30]. One of particular importance in this area is the Passerini three-component reaction (P-3CR) involving the condensation of an isocyanide, an aldehyde, and a carboxylic acid followed by

Mumm's rearrangement, leading to α-acyloxyamide scaffolds [31]. The Passerini reaction broadly has been used in synthetic and medicinal chemistry [36–40]. The α-acyloxycarboxamide scaffold is a frequent motif in many natural products as well as in pharmacologically active depspeptides thus making the Passerini reaction a particularly valuable method by which highly diverse libraries of these compounds can be constructed fast and screened for biological activity. For example, compound **A** exhibits inhibitory activity for the RNase H function of HIV-1 reverse transcriptase [32] and compound **B** is reported to have herbicidal activity (Fig. 1) [33].

Various strategies, reagents, and catalysts have been used over the past decades due to the profound biological activity, synthetic utility, and diverse structural variety of new compounds [34, 35]. The Passerini reaction is generally performed in an organic solvent such as CH_2Cl_2, toluene or MeOH and long reaction times are often required. However, several optimizations have been achieved to improve the yield, reduce the cost, the ecological impact, and the reaction times of this reaction. Thus, processes have been described in aqueous solution [36], ionic liquid [37, 38], without using solvents either at room temperature [39], or under microwave irradiation activation [40]. Recently, this reaction has been performed under high temperature (180 °C) under solvent free conditions [41]. Therefore, developing a more efficient and environmentally friendly methodology for the Passerini reaction is highly desirable.

Results and discussion

As part of our studies on MCRs [42–46], herein we wish to report an efficient, fast, and environmentally compatible procedures for the synthesis of α-acyloxyamide derivatives with a Passerini three-component cascade reaction in various choline chloride-based deep eutectic solvents. It was found that simple mixing of an aldehyde, a carboxylic acid, and an isocyanide in DES without using any catalysts afforded products in excellent yield. In order to obtain the best medium, the catalytic activities of various DESs were evaluated. The model reaction of

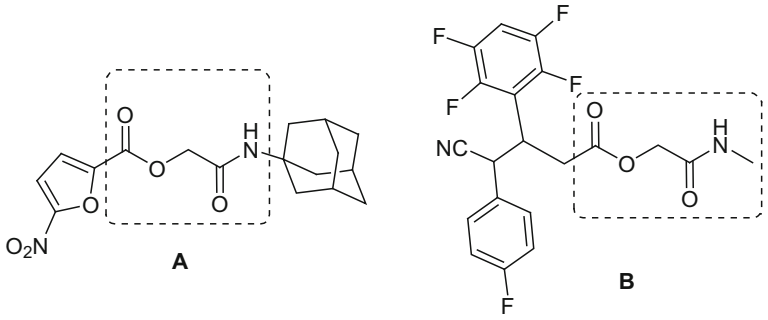


Fig. 1 Examples of α-acyloxyamide-based drugs

benzaldehyde (0.20 mmol), benzoic acid (0.20 mmol), and cyclohexyl isocyanide (0.20 mmol) was investigated in various choline chloride-based DESs systems at different temperatures. Furthermore, the utilization of some DESs based on ChCl such as ethylene glycol and resorcinol/urea provided the desired product in trace yields at ambient temperature, but, at 60 °C, ChCl-based ethylene glycol was observed at about 50 %. Likewise, utilization of organic acids on DESs such as malonic acid, PTSA ,and citric acid indicated the moderate yield, while also for this optimization $ZnCl_2$ as a Lewis acid has been used.

As indicated in Table 1, the best conditions for this reaction were observed while the starting materials were heated at 60 °C in choline chloride and urea.

After optimization, the scope of this reaction was examined using different carboxylic acids, aldehydes, and isocyanides in 1 mL of DES (ChCl:Urea). The results of these reactions are summarized in Table 2. Various carboxylic acids such as benzoic acid, 4-methyl benzoic acid, 4-bromo benzoic acid, 2-naphthoic acid, and *trans*-styryl acetic acid were used in this process. Our literature survey at this stage revealed that there was no report yet available on the use of 2-naphthoic acid in the Passerini reactions (**4p**). Aliphatic acids such as propionic acid (**4k**) were applied successfully in this reaction and afforded the corresponding α-acyloxyamides in excellent yields. Also, aromatic aldehydes containing both electron-donating and withdrawing groups on the phenyl ring reacted equally well in this protocol. Heteroaromatic aldehydes such as pyridine-4-carbaldehyde and furfural are suitable substrates in this reaction (**4j, 4p**). In addition, aliphatic aldehyde such as

Table 1 Passerini reaction in various choline chloride-based DESs[a]

Entry	DES (molar ratio)	T (°C)	Yield (%)
1	Choline chloride:malonic acid (1:1)	25	35
2	Choline chloride:malonic acid (1:1)	60	60
3	Choline chloride:citric acid (2:1)	25	46
4	Choline chloride:citric acid (2:1)	60	75
5	Choline chloride:PTSA (1:1)	25	20
6	Choline chloride:PTSA (1:1)	60	66
7	Choline chloride:$ZnCl_2$ (1:2)	25	35
8	Choline chloride:$ZnCl_2$ (1:2)	60	63
9	Choline chloride:ethylene glycol (1:2)	25	Trace
10	Choline chloride:ethylene glycol (1:2)	60	50
11	Choline chloride:urea:resorcinol (1:2:3)	25	Trace
12	Choline chloride:urea:resorcinol (1:2:3)	60	10
13	Choline chloride:urea (1:2)	25	65
14	Choline chloride:urea (1:2)	60	90
15	Choline chloride:urea (1:2)	80	92

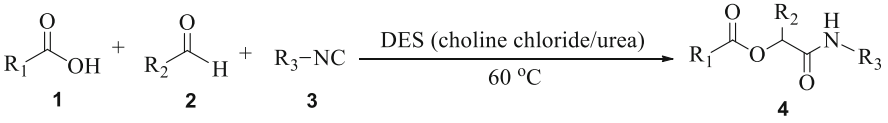
[a] Reaction conditions: benzaldehyde (0.20 mmol), benzoic acid (0.20 mmol) and cyclohexyl isocyanide (0.20 mmol)

Table 2 Passerini reaction in DES conditions

Entry	R_1	R_2	R_3	Time (min)	Product	Yield (%)[a]
1	C_6H_5	C_6H_5	cyclohexyl	7	(**4a**)	90
2	C_6H_5	*o*-$NO_2C_6H_4$	cyclohexyl	20	(**4b**)	85
3	C_6H_5	*m*-$NO_2C_6H_4$	cyclohexyl	15	(**4c**)	82
4	C_6H_5	*p*-$NO_2C_6H_4$	cyclohexyl	10	(**4d**)	94
5	C_6H_5	*o*-$CH_3C_6H_4$	cyclohexyl	20	(**4e**)	85
6	C_6H_5	*p*- $CH_3C_6H_4$	cyclohexyl	12	(**4f**)	88
7	C_6H_5	*p*-BrC_6H_4	cyclohexyl	10	(**4g**)	90
8	C_6H_5	*p*-ClC_6H_4	cyclohexyl	7	(**4h**)	90
9	*p*-BrC_6H_4	*p*-$CH_3C_6H_4$	cyclohexyl	15	(**4h**)	85
10	*p*-$CH_3C_6H_4$	4-Pyridine	cyclohexyl	12	(**4j**)	88
11	Propionic	*p*-BrC_6H_4	cyclohexyl	18	(**4k**)	85
12	C_6H_5	Isopropyl	cyclohexyl	25	(**4l**)	65
13	C_6H_5	Piperonal	*tert*-butyl	20	(**4m**)	84
14	C_6H_5	*p*-ClC_6H_4	*tert*-butyl	15	(**4n**)	90
15	*p*-$CH_3C_6H_4$	2-Naphthyl	*tert*-butyl	10	(**4o**)	92
16	2-Naphthyl	2-Furan	*tert*-butyl	12	(**4p**)	89

[a] Isolated yield

isopropyl carbaldehyde reacted with moderate yield. Likewise we found that the reactions proceeded very efficiently with alkyl isocyanides such as cyclohexyl isocyanide and *tert*-butyl isocyanide.



In addition, for completing library compounds, our research conducted the Passerini reaction with activated carbonyl group compounds such as alloxsans [47] and isatin [48] instead of aldehydes. The scope of the reaction was investigated and the results summarized in Table 3.

DES may play a dual role in this reaction as a solvent and a catalyst, which, in the latter case, was activated by the carbonyl group via hydrogen bonding. Also, one of the advantages of DES is their ability to perform as a recyclable reaction media. The DES was dissolved in water, and the crude product was obtained by extraction with EtOAc. The DES was recovered by evaporating water at 80 °C under vacuum and recycled for the next batch. Afterward, it was reused for subsequent reactions (Fig. 2).

The results of our reaction conditions were compared with previous reports for the synthesis of α-acyloxyamides with respect to their yields, temperatures, and the times required for the reaction. As shown in Table 4, in the case of DES, the

Table 3 Synthesis of α-acyloxyamides by alloxsans and isatin

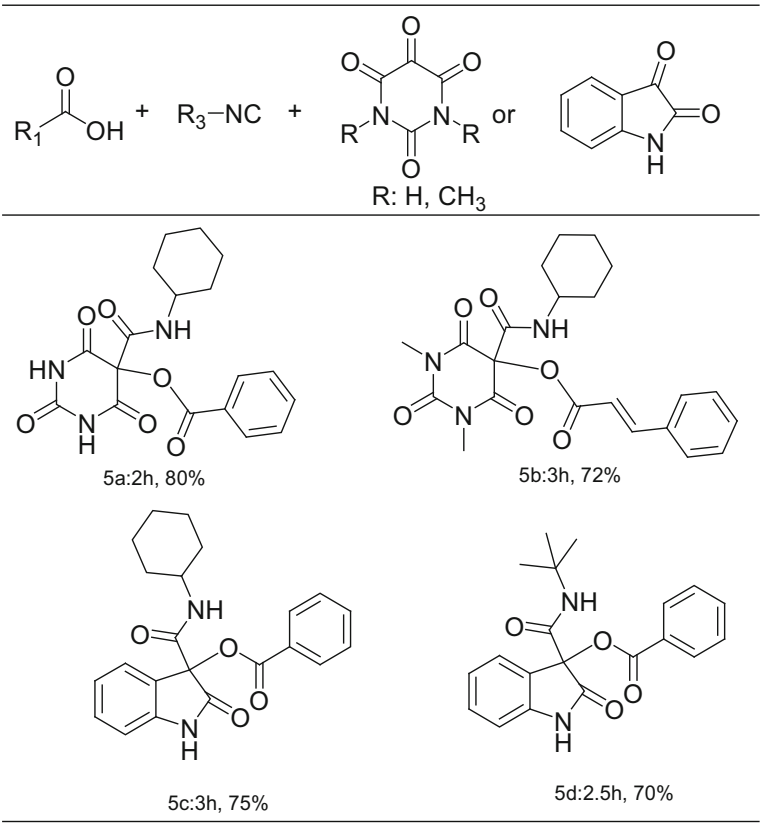
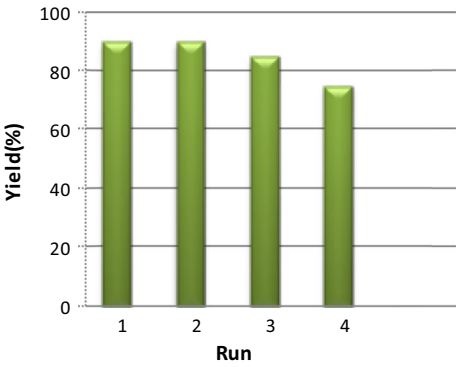


Fig. 2 Reusability of DES



reaction yield is increased at the shorter reaction times under mild conditions and avoids problems associated with such as handling, safety, pollution, corrosiveness, harsh reaction conditions, toxicity of metal-containing waste, tedious work-up, and catalyst recovery.

Experimental section

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were recorded on a Shimadzu IR-470 spectrometer. The elemental analyses were performed with an Elementar Analysensysteme GmbH VarioEL. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz. NMR spectra were obtained in DMSO-d_6. The chemicals used were purchased from Merck and Fluka Chemical Companies.

Preparation of choline chloride based deep eutectic solvents (DESs)

Choline chloride-based deep eutectic solvent was prepared according to the literature [2, 3]; choline chloride and the second component were mixed on the basis of reported relationships as in Table 1 and heated until a liquid appeared. The obtained DES was used without any further purification.

General procedure for the synthesis of α-acyloxyamides

A mixture of an aldehyde (0.25 mmol), a carboxylic acid (0.25 mmol), and an isocyanide (0.25 mmol) were placed in choline chloride–urea-based deep eutectic solvent (1 mL) and vigorously stirred at 60 °C for a certain period of time as indicated in Table 1. After completion of the reaction, as indicated by TLC (5:5 EtOAc:Hexane), water (10 mL) was added. The solid was separated by filtration and washed twice with a saturated aqueous solution of $NaHCO_3$. In most of the cases, the pure products were obtained without further purification. If necessary, the purification was performed by recrystallization from ethyl acetate or ethanol.

Table 4 Comparison of the results of the Passerini reaction using different systems with those obtained by reported conditions

Conditions	T (°C)	Time	Yield (%)	Ref.
Water	25	3.5 h	72–95	[42]
Solvent free	180	4 min	64–92	[47]
Solvent free	25	24 h	20–89	[45]
Ionic liquid [bmim][PF_6]	r.t	2–14 h	35–91	[44]
PEG 400	r.t	1–6 h	60–92	[43]
Ionic liquid [bmim][BF_4]	60	5–9 h	40–92	[43]
CH_2Cl_2	Reflux	15 h	78	[44]
Toluene	60	10 h	77	[44]
MeOH	Reflux	18 h	32	[44]
Microwave	60	3–5 min	61–82	[46]
DES (choline chloride:urea)	60	7–25 min	65–94	This work

Selected data of new compounds

*(Cyclohexylcarbamoyl)(4-bromophenyl)methylbenzoate (**4g**)*

White powder; m.p. 210 °C (dec). IR (KBr) cm^{-1}: 3368, 3330, 2913, 2850, 1647, 1615, 1511, 1440. ^1H NMR (300 MHz, DMSO-d_6): δ (ppm) 1.08–1.24 (m, 5H), 1.56–1.77(m, 5H), 3.37 (m, 1H), 6.07 (s, 1H), 7.57–7.85 (m, 7H), 8.03 (d, 2H, $J = 7.8$ Hz), 8.32 (brs, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 24.8, 24.9, 25.5, 32.4, 32.6, 48.1, 75.2, 122.3, 129.3, 129.5, 129.7, 129.9, 131.9, 134.1, 135.9, 165.3, 166.9 Anal. Calcd for $C_{21}H_{22}BrNO_3$: C, 60.59; H, 5.33; N, 3.36; found C, 60.22; H, 5.64; N, 3.45.

*(Cyclohexylcarbamoyl)(4-bromophenyl)methyl propionate (**4k**)*

Cream powder; m.p. 102–104 °C. IR (KBr) cm^{-1}: 3335, 2927, 2840, 1651, 1531, 1435. ^1H NMR (300 MHz, DMSO-d_6): δ (ppm) 1.02–1.07(*t*, 3H, $J = 7.5$ Hz), 1.13–1.20(m, 5H), 1.54–1.69 (m, 5H), 2.41–2.44 (*q*, 2H, $J = 2.4$ Hz), 3.12 (m, 1H), 5.81 (s, 1H), 7.35–7.43 (m, 2H), 7.57–7.60 (m, 2H), 8.20 (brs, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 25.1, 25.5, 25.7, 27.1, 32.4, 32.6, 48.0, 53.6, 74.5, 122.1, 129.7, 131.7, 136.0, 167.0, 173.3; Anal. Calcd for $C_{17}H_{22}BrNO_3$: C, 55.44; H, 6.02; N, 3.80; found C, 55.22; H, 5.95; N, 3.93.

*(Tert-butylcarbamoyl)(naphthalen-2-yl)methyl 4-methylbenzoate (**4o**)*

White powder; m.p. 142–143 °C. IR (KBr) cm^{-1}: 3335, 2907, 2838, 1662, 1531, 1435. ^1H NMR (300 MHz, DMSO-d_6): δ (ppm) 1.23 (m, 9H), 2.40 (m, 3H), 7.39–7.44 (m, 2H), 7.58 (m, 3H), 7.73–7.76 (m, 1H), 7.89–7.98 (m, 3H), 7.99 (brs, 1H), 8.08–8.11 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 26.4, 33.5, 55.7, 80.7, 130.1, 131.0, 131.6, 131.8, 132.6, 132.8, 134.7, 137.4, 138.0, 139.0, 149.1, 162.8, 170.1; Anal. Calcd for $C_{24}H_{25}NO_3$: C, 76.77; H, 6.71; N, 3.73; found C, 76.97; H, 6.50; N, 3.45.

*(Tert-butylcarbamoyl)(furan-2-yl)methyl 2-naphthoate (**4p**)*

Cream powder; m.p. 168–171 °C. IR (KBr) cm^{-1}: 3348, 2910, 2843, 1657, 1625, 1511, 1480. ^1H NMR (300 MHz, DMSO-d_6): δ (ppm) 1.27 (m, 9H), 4.14 (s, 1H), 5.93 (m, 1H), 6.40 (m, 1H), 6.50 (m, 1H), 7.43–7.59 (m, 5H), 7.73 (brs, 1H). ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 33.5, 55.1, 75.9, 107.1, 108.3, 111.7, 131.2, 132.8, 134.7, 137.4, 138.0, 139.0, 149.1, 162.8, 170.1. Anal. Calcd for $C_{21}H_{21}NO_4$: C, 71.78; H, 6.02; N, 3.99; found C, 71.57; H, 5.64; N, 4.45.

*3-(Cyclohexylcarbamoyl)-2-oxoindolin-3-yl benzoate (**5e**)*

Yellow powder; m.p. 199–204 °C. IR (KBr) cm^{-1}: 3345, 2923, 2845, 1725, 1724, 1668, 1627, 1518, 1441. ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 0.92–1.20 (m, 10H),

3.70 (brs, 1H), 6.80–6.82 (m, 2H), 7.15–7.22 (m, 4H), 7.73–7.79 (m, 2H), 7.82 (brs, 1H). $C_{22}H_{22}N_2O_4$: C, 69.83; H, 5.86; N, 7.40; found C, 69.22; H, 5.64; N, 8.45.

Conclusion

In summary, we have described a green, effective, and rapid strategy for use of the Passerini reaction with an eco-friendly and biodegradable deep eutectic solvent based in a mixture of choline chloride and urea. Advantages of this system are simplicity, easy separation, reusability of DES, short reaction times, and high yields.

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