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Recent developments of task-specific ionic liquids in organic synthesis

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Task-specific ionic liquids (TSILs) have received increased attention over the past few years as it is possible to form any specific ionic liquid (IL) composition depending upon user’s need of the desired physical, chemical, and biological properties. These fascinating materials have shown promising results in various areas such as organic synthesis, catalysis, and specially recent emerging trend of use as functionalized ILs for chiral and nanoparticle synthesis. Present review gives an update of recent developments in the field of TSILs with emphasis on their applications in organic synthesis.

Keywords: TSILs; solid catalyst; acidic ionic liquids; basic ionic liquids; chiral ionic liquids

Introduction

From simple detergents to life saving drugs to plastics, many of life’s essential commodities would be impossible without the chemical industry. Few people choose to live without the benefits of modern chemistry, but often the benefits are associated with other fields, such as medicine, materials, or engineering. Over recent decades, the chemical industry has been increasingly regulated by stringent and compelling demands for greener processes, and the development of cost-effective and environmentally benign catalytic systems in order to reduce harmful emissions and effluents and ensure worker safety. However, many chemical industry processes have been or still are highly polluting. The past 15 years, ionic liquids (ILs) have provided solution to problems accompanying increased production with sustainable green approaches. It is mainly attributed to their unique physical properties that are mainly in accordance to a green chemistry point of view and a chemical point of view. Such properties include polar, non-coordinating nature, generally low flammability due primarily to low volatility, and their ability to dissolve a range of organic and inorganic compounds. Exploitation of these properties has resulted in an increased use of ILs in both separations and in catalysis.

Even though the term IL was introduced recently, the history of ILs dates back to 1914 when Walden (1) reported the physical properties of IL [EtNH₃][NO₃]. Earlier developments in the field of ILs were critically reviewed by Gorden (2) in 1969. It was the pioneering study of Wilkes and co-workers (3,4), that resulted in increasing popularity of ILs as reaction and extraction media in research and development, and then widely been promoted as “green solvents.” The first successful use of an IL, dialkyldimethylammonium chloroaluminate, as a catalyst in Friedel–Crafts acylations was reported in 1986 (5). However, moisture sensitivity and decomposition of this chloroaluminate salt under normal atmospheric conditions were two major drawbacks that precluded their practical use. After this report, a resurgence of the rich chemistry of ILs began. The pioneering work of Earle and Seddon then engraved the ILs to its well-known versatility (6).

Davis and co-workers (7) first demonstrated the concept of designing IL to interact with a solute in a specific fashion, showing that a thiazolium IL could function as solvent-catalyst for the benzoin condensation. He then outlined the concept in a brief review (8) by introducing the term “task-specific ionic liquids” (TSILs) to describe IL which incorporate functional groups designed to impart to them particular properties or reactivities (9). The TSILs may be defined as ILs in which functional group is covalently tethered to the cation or anion (or both) of the IL. These can be considered as liquid version of solid-supported catalyst with added advantage of kinetic mobility and large operational surface area. Today TSILs form an important class of ILs that has received increasing attention over the past few years because it is possible to form any specific IL.

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composition depending upon user’s needs of desired physical, chemical, and biological properties. Apart from this, there are several rationales for the incorporation of a functional group into an IL. First, the inclusion of the functional group will doubtless alter the solvent parameters of an IL. The parameters like polarity, H-bond acidity and basicity, polarizability, etc. are the attributes that make any chemical a good or poor solvent for a given solute. A second rationale for the inclusion of a functional group in an IL is to imbue the salt with a capacity to covalently bind to or catalytically activate a dissolved substrate. For example, a primary amine functionalized imidazolium salt can separate CO₂ from gas streams (ample, a primary amine functionalized imidazolium catalytically activate a dissolved substrate. For example, a primary amine functionalized imidazolium salt can separate CO₂ from gas streams (10), while ILs bearing appended sulfonic acid groups were used as solvent-catalyst for esterification (11). During past 5 years various types of TSILs have been designed to accomplish the specific tasks like catalysis, organic synthesis, chiral induction, synthesis and stabilization of nanomaterials, electrochemical applications, catalyst surface modifiers, etc., and they have been comprehensively reviewed by various authors. The first in-depth review in the field of TSIL appeared in the year 2004 by Davis (8) which dealt with the synthesis of TSIL and their applications in organic synthesis, catalysis, separation, and electrochemistry. Later on developments in the field of functionalized imidazolium TSILs has been reviewed by Lee (12). Winkel et al. (13) gave an account of applications of task-specific chiral ionic liquids (CILs) in asymmetric synthesis. In a recent review of TSILs, Giernoth (14) has taken account of TSILs as catalyst and support for synthesis, as new magnetic materials, as gas reservoir, in chromatography and in industrial applications. Present review attempts to give an update of recent developments in the field of TSILs with particular emphasis on their applications in organic synthesis, catalysis, and nanoparticle synthesis.

Acidic and basic task-specific ionic liquids (TSILs)

The chemical property that imparts a variety of physical characteristics to the ILs which has been investigated is the relative acidity or basicity of the component ions. The commonplace starting point that many of the ions are inert is not always correct, as one looks further a field for task-specific ions. Here, we describe recent work with a range of anions and cations having distinct Lewis acidity or basicity and functionalization of anion or cation depending upon the task. The ILs containing Lewis base anions can exhibit a base catalysis phenomenon, which can be utilized, for example, in a variety of acetylation reactions. The majority of ions used in the formulation of ILs can be considered in terms of the Lewis definition of acids and bases, i.e. whether they are capable of accepting or donating an electron pair. However, there are also a number of ions that must be considered according to the Brønsted definition viz. those that are able to donate or accept a proton. For example, a number of ILs containing protonated cations and anions like dihydrogen phosphate and hydrogen sulfate fall into this category.

We investigated the Knoevenagel condensation of Meldrum’s acid with aldehyde in various ILs. The enhancement in rate of reaction was observed in Brønsted acidic IL [Hmim]Tfa. Knoevenagel condensation of Meldrum’s acid with aromatic and heteroaromatic aldehydes resulted in corresponding ylidene derivatives (15) (Scheme 1) whereas that with ortho-hydroxyaldehydes gave corresponding coumarin-3-carboxylic acid derivatives (16) (Scheme 2).

Dabiri et al. (17) reported a novel one-pot tandem synthesis of 2-styryl-4(3H)-quinazolinones in same acidic IL [Hmim]Tfa. Isatoic anhydride, a primary aniline or ammonium acetate, and triethylorthoacetate were reacted in the presence of imidazolium trifluoroacetate [Hmim]Tfa. Subsequently an aromatic aldehyde was added to the mixture to afford corresponding quinazolinone product in high to excellent yields (Scheme 3).

Wang et al. (18) developed some Brønsted acidic benzimidazolium-based ILs and explored their use as environmentally benign catalysts for acetalization of aldehydes. They found the IL [PSebim]HSO₄ (Figure 1) more acidic among the studied ILs and was efficient for acetalization. Advantages of this IL over previous reports are good solubility of aromatic aldehydes, high activity and selectivity, no need of excess diols (quantitative use).

Both the homogeneous and heterogeneous catalysts have counter (interlocking) advantages and disadvantages. So the catalyst having properties of both can be a “dream catalyst.” Leng and co-workers...
(19) developed a non-conventional propane sulfonate functionalized heteropolyacid-based IL for esterification (Scheme 4). These ILs comprised of three organic cations and an inorganic heteropolyanion. Their melting points above 100°C make them non-conventional ILs. These ILs being soluble in starting materials, i.e. carboxylic acids and alcohols works as homogeneous catalyst and being insoluble in product, i.e. esters at the end of reaction IL comes out of reaction mixture as if it is a heterogeneous catalyst.

In a very recent report of TSIL, Das and co-workers (20) have reported a sulfonic acid functionalized IL for efficient synthesis of indole derivatives (Scheme 5). The lucrative advantage of this IL is that it could be reused up to 10 cycles without any substantial loss of catalytic activity. The catalyst is versatile as it is also applicable to both aliphatic and aromatic amines and in synthesis of bis(indolyl)methane.

Ranu and Banerjee (21) described path-breaking results in which the TSIL [bmim]OH provided chemo selectivity in Michael addition of active methylene compounds on conjugated ketones, esters, and nitriles. The vinyl ketones and chalcones gave mono addition products while unsaturated esters and nitriles gave bis-addition products (Scheme 6). The method is operationally simple and giving high yields in shorter reaction times for a wide ranging substrates. In addition to this the IL can be reused up to six cycles.

A basic functionalized IL, 1-butyl-3-methylimidazolium hydroxide ([bmim]OH), catalyzed the three-component condensation reaction (22) of acid chlorides, amino acids, and dialkyl acetylenedicarboxylates in water to afford functionalized pyrroles in high yields. Yavari and co-workers (22) studied the reaction in 10 different ILs out of which [bmim]OH was found to be an effective at 80°C. The reaction
was found to be faster for benzoyl chlorides containing electron-withdrawing substituents (Scheme 7).

Zhang et al. (23) synthesized and used the basic TSIL [mammim]TfO (Scheme 8) for promotion of hydrogenation of CO₂ to formic acid with Ruthenium supported on silica as heterogeneous catalyst. The advantage of this method is that it has satisfactory activity and high selectivity. The unique features of recovery of formic acid and reuse of catalyst makes this approach compatible to industrial application.

Pavlinac and co-workers (24) in their recently published review have taken an account of halogenation of organic compounds in ILs. The review involves brief discussions on IL-mediated fluorofunctionalization, chlorofunctionalization, bromofunctionalization and ioddofunctionalization of organic compounds. Wang et al. (25) described application of a meticulously designed and synthesized ethanalamine functionalized TSIL for the palladium-catalyzed Heck reaction (Scheme 9). Here this IL performs a multifunctional role of base, ligand, and reaction media with added advantage of recyclability of the system. The catalyst system is very effective for a wide spectrum of substrates giving excellent yields.

**Task-specific ionic liquids (TSILs) – solid catalysts**

Green and cost-effective catalysts with high efficiency and selectivity are the cornerstones of contemporary synthetic chemistry. Even though easily separable heterogeneous catalysts are preferred in industry, these have some shortcomings like heat or mass transfer and lower chemo- and stereoselectivities. This can be circumvented by surface modification of solid catalysts. Recently, the concepts of solid catalysts with ionic liquids (SCILs) and supported ionic liquid phase catalysts (SILPCs) have drawn much attention because their combination is looked upon as a future option to integrate heterogeneous and homogeneous catalysts. In their first of its kind report Menhert et al. (26-27) and Risger et al. (28-29) demonstrated the use of ILs in supported liquid phase catalysts (SLPCs). Gu and co-workers (30) used hydrophobic IL for coating the solid surface of silica-supported sulfonic acid catalyst to enhance the selectivity. They successfully demonstrated (Scheme 10) the significant impact on activity of silica-supported sodium catalyst by coating it with the IL [DBIm]SbF₆ (1-butyl-3-decylimidazolium) leading to enhanced activity toward the Michael
reaction of indoles and α-β-unsaturated ketones in water. The enhanced activity provides scope to perform the reaction under neutral conditions making it possible to carry out the reaction with acid labile substrates. Interestingly, this reaction did not proceed without IL highlighting the significance of IL.

Apart from enhanced activity, catalytic selectivity is another significance of coating the supported catalysts with ILs. The IL showed catalytic selectivities when a set of organic reactions in water were catalyzed by catalyst coated with IL. Etherification of 1-tolyl-1-ethanol catalyzed by SiO$_2$-SO$_3$H led to 30%
of 4-methylstyrene as side product reducing the yield of the desired ether to 61%. However, coating of SiO$_2$–SO$_3$H with 25 wt% of [C$_8$MIm]NTf$_2$ (1-methyl-3-octylimidazolium) led to a significant selectivity improvement (31) and the ether was obtained in 96% (Scheme 11).

**Metals and nanomaterials in ionic liquids (ILs)**

The search for more efficient catalytic systems that might combine the advantages of both homogeneous (catalyst modulation) and heterogeneous (catalyst recycling) catalysis is an essential requirement of modern chemistry. With the advances of nanotechnology, it has been possible to prepare “soluble” analogs of heterogeneous catalysts. However, one of the difficulties in using nanoparticles as catalyst is providing stability of small nanoparticles while retaining the activity. ILs have been shown to provide “electrostatic” stabilization for metal nanoparticles and more surface area for the reaction to take place (32). So ILs internally control the stability and activity of nanoparticles. Multiphase systems of IL-nanoparticles facilitate easy recovery of nanoparticles. There have been several reviews (32,33) about the preparation and application of nanoparticles in ILs, with the most recent having been published in the 2009 (34). The present review is intended to update the reader about the recent work in the field of combination of two cutting-edge topics – nanoparticles and ILs.

Recently, Redel and co-workers (35) have synthesized stable cobalt, rhodium, and iridium nanoparticles by thermal decomposition under argon from Co$_2$(CO)$_8$, Rh$_6$(CO)$_{16}$ and Ir$_4$(CO)$_{12}$ dissolved in the ILs [bmim]BF$_4$, [bmim]OTf and [btmA]NTf$_2$ [bmim = n-butyl-methyl-imidazolium, btmA = n-butyl-tri-methyl-ammonium, OTf = O$_3$SCF$_3$, NTf$_2$ = N[O$_2$SCF$_3$]$_2$]. They achieved very small and uniform nanoparticle size of about 1–3 nm in [bmim]BF$_4$. Increase in size was observed with increase in molecular volume of the IL anion from [bmim]OTf to [btmA]NTf$_2$. Importantly, among these nanoparticles the rhodium or iridium nanoparticle/IL systems function as highly effective and recyclable catalysts in the biphasic liquid–liquid hydrogenation of cyclohexene to cyclohexane (Scheme 12) with activities of up to 1900 mol$_{product}$/mol$_{metal}$ h and quantitative conversion.

Cal et al. (36) reported the use of palladium nanoparticle for Ullmann reactions in tetrabutyl ammonium salt ILs as a reservoir of catalyst with aldehydes as the reductant (Scheme 13). This type of “ligand-free” catalysis is gaining considerable importance because it avoids the use of toxic or expensive phosphane ligands and allows catalyst recycling. The role of TBA in this process, as this IL behaves simultaneously as a base, ligand, and reaction medium and has effect on the chemoselectivity of the catalysis which cannot be obtained by replacing this IL with a generic source of acetate anions.

Taher and co-workers (37) reported Pd-NHC–IL matrix immobilized into IL layers coated on the surface of Fe$_3$O$_4$ (Scheme 14) by a simple process. These immobilized Pd-NHC exhibited both high-catalytic activity and stability for the coupling between aryl bromide and arylboronic acid in water. Importantly, this catalyst was simply recovered by an external permanent magnet and recycled without a significant loss in the catalytic activity.

In one of our recently published article (38) we have demonstrated the dramatic effect of ILs on shape of copper nanoparticles (Figure 2) and their
application in 1,3-dipolar cycloaddition reactions of azides and aryl- and sugar-based terminal alkynes. Change in anion has led to change in shape of the nanoparticle. It was observed that both the ILs used could not stabilize the nanoparticles and we had to use polyvinyl pyridine (PVP) as capping agent for these nanoparticles. Spherical nanoparticles were obtained in [bmim]BF₄ while cubical nanoparticles were obtained in [bmim]PF₆. Both the types of nanoparticles have shown profound effect on the cycloaddition reaction between azides and terminal alkynes (Scheme 15).

Very recently, Wang and co-workers (39) demonstrated an effective depolymerization of poly (ethylene terephthalate), PET (Scheme 16), using iron-containing imidazolium-based IL 1-butyl-3-methylimidazolium tetrachloroferrate [bmim]FeCl₄. The advantages of use of this IL are relatively low-reaction temperature, good conversion with enhanced selectivity in comparison with FeCl₃ and [bmim]Cl.

**Task-specific ionic liquid (TSIL) as a reaction media**

Nucleoside chemistry is an important area of research, but one in which solvent choice is severely compromised by poor solubility. Most organic solvents are not suitable; the exceptions are undesirable from an environmental point of view. In one of our earlier investigation (40), it was shown that the ILs were suitable, since the solubility of nucleosides was very good, especially in a series of methanesulfonate salts. The acylation of 2'-deoxyribonucleosides was studied in this IL. This investigation illustrated the benefits of solubility and recoverability, as well as efficient reaction conditions, which ILs can provide for nucleosides (Scheme 17).

Recently, TSILs based on 1,2,3-triazolium salts have been prepared by well-known Cu-catalyzed click reaction with the intention of simultaneous multifunctionalization of the cation (Scheme 18) (41). This has an added advantage of controlling both chemical
tion of IL and tertiary alcohol was acceleration of reaction with minimization of side reactions (Table 1). The weak H–F hydrogen bonding maintained the inherent nucleophilicity and reduced the basicity of fluoride anion which in turn reduced the side reactions.

Bellina et al. (43) synthesized a series of glycerylimidazolium (Figure 3) based ILs aiming at their applications to palladium-catalyzed coupling reactions. They extensively studied the physical and physico-chemical parameters such as thermal stability, hydrogen bonding, conductivity, and application of these series of TSIL for Heck reaction.

Park and co-workers (44) studied the cynosilylation of carbonyl compounds (Scheme 19) using Scandium triflate as catalyst and found a dramatic increase of catalytic activity in IL [bmim]SbF$_6$. This enhanced catalytic activity is attributed to enhancement of the Lewis acidity of the catalyst by anion exchange between IL and Scandium triflet.
In their first of its kind report of highly selective anodic fluorination of aromatic compounds, Sawamura et al. (45) used a TSIL of iodoarene (Figure 4) as mediator in a HF-based IL. Compared with conventional mediatory systems, TSIL as mediator has reduced mobility due to viscosity of IL further leading to easy fluorination.

In a recent report, Kundu et al. (46) put forth a new class of zwitterionic catalyst for stereoselective aza-Henry reaction (Scheme 20). The catalyst demonstrated high stereoselectivity with syn product forming predominantly. Authors further demonstrated commercial applicability of the catalyst based on effective scaled-up reaction and reusability of the catalyst.

In their attempt toward understanding the role of IL in catalysis at molecular level, Chakraborti and Roy (47) have studied role of IL in O-t-Boc formation and deprotection (Scheme 21), with the aid of NMR and MALDI-TOF-TOF-MS techniques. In their study they found that apart from imidazolium-based electrophilic activation of substrate through hydrogen bonding, the hydrogen bonding interaction of counter anion of IL plays a crucial role in nucleophilic activation of other reactants. So there is a synergetic effect of both the ions of an IL in catalyzing any reaction.

**Chiral ionic liquids (CILs)**

CILs are receiving increasing attention due to their ease of synthesis and properties. These new chiral solvents could play a central role in enantioselective organic chemistry and hopefully expand the scope of chiral solvents. A significant transfer of chirality in these solvents can be expected due to their high degree of organization. It has been reported that most of the ILs possess a polymeric behavior and are highly ordered H-bonded liquids (three-dimensional networks of anions and cations linked together by hydrogen bonds). These specific properties suggest that CILs could outperform the classical chiral solvents for asymmetric induction (48–50).

Many CILs have been synthesized which incorporate one or more functional groups, and thus they belong to the class of functionalized CILs. Since the functional group is intended to perform a desired task, these functionalized ionic liquids are also referred to as TSILs. CILs and functionalized CILs are discussed in this section (51).
There have been several reviews about the preparation and application of CILs, with the most recent two having been published in the middle of 2005 (52,53). The present review is intended to update the reader about the recent work in the field of CILs and functionalized CILs since then.

The more efficient, economic, and simple way to prepare enantiomerically pure ILs is to use precursors derived from the chiral pool either for the generation of the CILs anion or cation or for both. Therefore, CILs are mainly compounds having a central chirality. However, some new CILs having an axial or a planar chirality have also been developed. The first example dealing with the preparation of such a CIL was reported by Earley in 1999 (Scheme 22) (54). The chirality was brought by the lactate anion. The [bmlim] lactate IL was simply prepared by anion exchange between [bmlim]Cl and commercially available sodium (S)-2-hydroxypropionate in acetone.

Later on many novel CILs have been synthesized. Recently, Bonanni et al. (55) reported a straightforward strategy for the synthesis of series of novel pyrrolidinium ILs based on tartaric acid (Figure 5).

Siyutkin et al. (56) designed and synthesized (S)-proline modified TSIL for the direct asymmetric aldol reaction between cycloalkanones and aromatic aldehydes in the presence of water (Figure 6).

Li and co-workers (57) synthesized novel sulfur-functionalized ILs. Epoxidation of aromatic aldehydes with benzyl bromides was investigated using these organocatalyst CIL. Trans-epoxides were obtained with excellent diastereoselectivity and enantioselectivity up to 72% ee. The organocatalyst could be easily reused for five times without remarkable decrease in yields and enantioselectivities (Scheme 23).

A library of novel CIL-modified silanes was synthesized by using combinatorial approach by Li and co-workers (58). The chiral discrimination abilities of these CILs were screened using 19F-NMR spectroscopy (Scheme 24).

The CILs prepared from TBAB and natural amino acids were used as cocatalysts in the asymmetric cycloaddition of carbon dioxide to propylene oxide (PO) at room temperature in presence of chiral silane catalyst. The synergistic effect between catalyst and IL was studied. The system was found to exhibit good activity for the asymmetric cycloaddition of carbon dioxide to epoxide under very mild conditions (Scheme 25) (59).

A polymeric IL-functionalized chiral salen ligand (PICL) was synthesized by covalent polymerization between amino (–NH2) group of 1,3-dipropylaminemidazolium bromide with chloromethyl (–CH2Cl) group at two sides of 5,5’ positions in the typical chiral salen ligand (Figure 7). Treatment of the synthesized PICL with Mn(OAc)2·4H2O and LiCl under aerobic
oxidation yielded the corresponding polymeric IL-functionalized chiral salen Mn(III) complex (PICC). Comparable catalytic activity and enantioselectivity relative to the monomeric chiral salen Mn(III) complex were observed. Furthermore, recovery of the polymeric catalyst was readily accomplished by simple precipitation in n-hexane, and subsequently reused (10 times) without significant loss of reactivity and enantioselectivity (60).

Highly enantioselective Michael addition of cyclohexanone to aryl nitroolefins in the presence of an IL anchored pyrrolidine (10 mol%) and TFA (5 mol%) generated the corresponding adducts in high yields (up to 95%) with excellent diastereoselectivities.

Figure 7.
(up to >99:1 dr) and enantioselectivities (up to > 99% ee) (Scheme 26). Furthermore, the catalyst could be recycled and reused at least eight times without loss of its catalytic activity (61).

A novel family of chiral imidazolium-based ILs containing a chiral moiety and a free hydroxyl function were designed and synthesized using isosorbide as a biorenewable substrate (Scheme 27). These CILs were found to catalyze the aza Diels–Alder reaction to give good yields and moderate diastereoselectivities. CILs were recycled without much loss in efficiency (62).

Various CILs based on simple chiral natural products such as carbohydrates (63) (Figure 8), terpenes (64) (Figure 9), abietane (65) (Figure 10) have been synthesized recently. These ILs could find applications as new chiral solvents or catalyst for a range of asymmetric reactions in future.

**Conclusion**

Even though, ILs have been explored for past 15 years, the domain of TSILs is still in infant stage. The ILs in conglomeration of their basic properties with desired properties for the reaction under study could become the next arsenals available in the armory of green chemistry. Present industrial applications of ILs involve acid scavenging agent, anti-static agent, in gas compressors, chromatography, batteries, and many more. Even ILs in their next generation can play an important role as tunable active pharmaceutical ingredients (APIs) (65)
pharmaceutical industry. However, there are some limitations with the use of ILs. The exact role of ILs in carrying out reactions is still under-explored. Apart from this, the other issues such as toxicity, purification, development of improved product isolations and high cost are needed to be addressed in future research for full commercialization of ILs.

However, taking into account the wide window of properties and applications of ILs, research in the field of TSILs have great potential to grow in near future.

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