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Neoteric Trends in Green Chemistry: Harnessing the Power of Ionic Crystals

Kumari Suman[1]

[1]Assistant Professor, Department of Chemistry,
Pt. Deen Dayal Upadhyaya Government Girls P.G. College,
Rajajipuram, Lucknow

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Abstract: Neoteric solvents, particularly ionic liquids (ILs) and ionic liquid crystals, have emerged as transformative alternatives to conventional organic solvents, addressing critical environmental and sustainability challenges in chemical processes. This review elucidates the necessity of green solvents amid escalating solvent waste—exceeding 26 million tons annually—and highlights ILs' tunable physicochemical properties, including low vapor pressure, high thermal stability (up to 400°C), wide electrochemical windows, and designer versatility through cation-anion combinations. We detail IL synthesis via quaternization and metathesis, classification (e.g., 1-alkyl-3-methylimidazolium systems), and properties enabling applications in electrochemical devices (supercapacitors, lithium batteries, DSSCs), organic synthesis (Diels-Alder, Heck reactions), extraction technologies, pharmaceuticals, gas handling, renewable energy, waste recycling, and advanced fluids. While ILs enhance process green metrics through recyclability and selectivity, challenges like toxicity and cost persist, underscoring the need for biocompatible variants and scalable production. Ionic liquid crystals, with stimulus-responsive anisotropy, show particular promise for energy storage electrolytes. This overview advocates broader adoption of ILs to advance sustainable chemistry, alongside emerging alternatives like switchable solvents.

Keywords: *Ionic Liquids, Green Chemistry, Neoteric Solvents, Ionic Liquid Crystals, Sustainable Electrolytes, Electrochemical Applications, Solvent Extraction, Renewable Energy.*

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I. INTRODUCTION

Solvents play pivotal roles in both industrial and domestic contexts, but their potential environmental impact, including losses and emissions, necessitates concerted efforts for mitigation or complete avoidance. This heightened awareness, particularly since the 1990s, has propelled green chemistry into the forefront, introducing the concept of environmentally friendly "green" solvents. While this concept has spurred significant research and the development of novel "neoteric" solvents, their widespread adoption remains limited. An analysis of existing literature underscores the inefficiency of searching for less impactful solvents without considering the specific industrial conditions in which they will be utilized. Furthermore, broader sustainability concerns, such as sourcing organic carbon from non-fossil origins for solvent production, outweigh mere "greenness" in importance. Although solvency is a universal necessity, the quest for a universal solvent, akin to the mythical alkahest, remains an unattainable ideal.

In adherence to the principles of Green Chemistry, there has been a notable surge in interest in non-conventional extraction techniques over the past few decades. Various innovative approaches have emerged, including the utilization of biobased and neoteric solvents such as ionic liquids.

II. WHY GREEN SOLVENTS ARE NECESSARY

The imperative to enhance environmental sustainability underscores the adoption of strategies such as repurposing waste into valuable resources, epitomizing a proactive approach in achieving "greenness." A fundamental tenet of green chemistry, as elucidated by Wilkinson in 1997, is the aspiration towards source prevention, advocating for the avoidance of toxic substance generation altogether. Solvent utilization pervades not only the chemical industry but also various allied sectors, constituting an indispensable element of contemporary life. In 1991, global manufacturing of the 25 most commonly used solvents surpassed a staggering 26 million tons annually. Notably, five of the top 10 disposed chemicals were solvents, including methanol, toluene, xylene, methyl ethyl ketone, and dichloromethane. The collective release or disposal of these chemicals exceeded 687 million pounds, representing a significant portion of the total quantity of TRI-listed chemicals for that year, as detailed by Sullivan in 1997. Given the ubiquitous nature of solvent usage and the associated hazards and safety concerns, the development and selection of solvents have been perennially fraught with challenges. Initially, solvents with overtly hazardous properties, such as high flammability or explosivity, were shunned. However, with an enhanced understanding of the health and environmental ramifications of solvent usage, there has been a concerted effort to scrutinize alternative solvents for all hazardous properties. Consequently, new solvent systems are being developed to align with environmentally benign principles. Numerous solvents, ingrained in industrial practices for decades, have been found to harbour serious toxicological properties. Notably, halogenated solvents such as carbon tetrachloride, perchloroethylene, and chloroform have been implicated as probable and/or suspect carcinogens, while other solvent classes exhibit neurotoxicological effects. The pursuit of unconventional solvents represents a pivotal stride towards greener and more environmentally friendly analysis and separation processes. Alternative solvents like ionic liquids (ILs) and supercritical fluids offer an additional allure to researchers owing to their tunability. The ability to fine-tune solvent properties enables their substitution for specific solvents across diverse processes or the development of novel sample processing methodologies, thereby advancing environmental sustainability efforts. Ionic liquid crystals represent a unique class of organic salts, blending the distinctive properties of both ionic liquids and liquid crystalline materials. These compounds feature non-covalently bound delocalized ion pairs comprising large organic cations and anions. Remarkably, they exhibit stimulus-responsive anisotropic phase transitions, leading to heightened ionic diffusion and conductivity. Consequently, they emerge as promising candidates for electrolyte applications in energy storage systems. In this chapter, our objective is to provide an overview of the significant advancements in the realm of ionic liquid crystalline electrolytes. By doing so, we aim to spark interest within the broader research community, encouraging collaborative efforts to address challenges in electrolyte materials for sustainable energy-related devices. These devices encompass a range of technologies, including supercapacitors, lithium batteries, fuel cells, and dye-sensitized solar cells (DSSCs). A comparison of the key properties of organic solvents versus ILs, originally outlined by Plechkova and Seddon in 2008, has been updated to highlight the differences between these two classes of materials. This comparison underscores the tuneable nature of ILs, allowing for the synthesis of solvents with tailored properties for specific applications, a capability less common with organic solvents.

III. WHAT ARE IONIC LIQUIDS?

Ionic liquids, often abbreviated as ILs, represent a unique class of substances that exist in the liquid phase under typical conditions. Various terms such as room-temperature ionic liquid (RTIL), nonaqueous ionic liquid, molten salt, liquid organic salt, and fused salt have been employed to describe salts in their liquid state (Welton, 1999). Unlike conventional solvents, which consist of molecules with distinct positive and negative charges, ionic liquids exclusively comprise anions and cations. These molecular solvents can encompass a wide range of neutral species such as benzene, methanol, chloroform, and water. Traditional molten salts typically exhibit high melting points, such as 801°C for sodium chloride and 614°C for lithium chloride, limiting their applicability as solvents in many contexts. In contrast, RTILs remain in the liquid state at or below room temperature. The upper-temperature threshold for classifying a substance as an "ionic liquid" is generally set at 100°C, although some may solidify into glasses upon cooling. Ionic systems with higher melting points are typically referred to as molten salts (Visser et al., 2002)

- *Features*

Ionic liquids are chemically intriguing for several reasons. Firstly, they possess a wide temperature range and low vapor pressure, making them highly stable under a variety of conditions. For instance, 1-butyl-3-methylimidazolium hexafluorophosphate demonstrates stability in air up to 300°C. Additionally, ionic liquids exhibit exceptional solvent properties and can significantly influence chemical reactions without undergoing alteration themselves (Welton, 1999). These liquids are essentially salts where the ions are weakly coordinated, allowing them to remain liquid below 100°C or even at room temperature, hence termed as room temperature ionic liquids (RTILs). Typically, at least one ion in an ionic liquid has a delocalized charge, and one component is organic, preventing the formation of a stable crystal lattice (Wasserscheid, 2000). Moreover, the vast array of possible combinations of cations and anions in ionic liquids leads to numerous permutations, enabling the fine-tuning of their physical and chemical properties almost at will (Anon, 2001). This flexibility enhances their applicability across various domains of chemistry and materials science

IV. CLASSIFICATION OF IONIC LIQUIDS

In a basic ionic liquid configuration, a single cation pairs with a single anion. In general, anions tend to be small, whereas cations are often distinguished by their large alkyl chains. In simple ionic liquid systems, commonly used anions include Cl, Br, BF_4, CF_3SO_3 (OTf), and $N(SO_2CF_3)_2$ (NTf_2). Among the various cations found in ionic liquids, the 1-alkyl-3-methyl-imidazolium cations are widely researched and frequently observed.

Additionally, there are other cations composed of phosphorus or nitrogen-containing organic ions, often featuring attached alkyl chains of varying lengths.

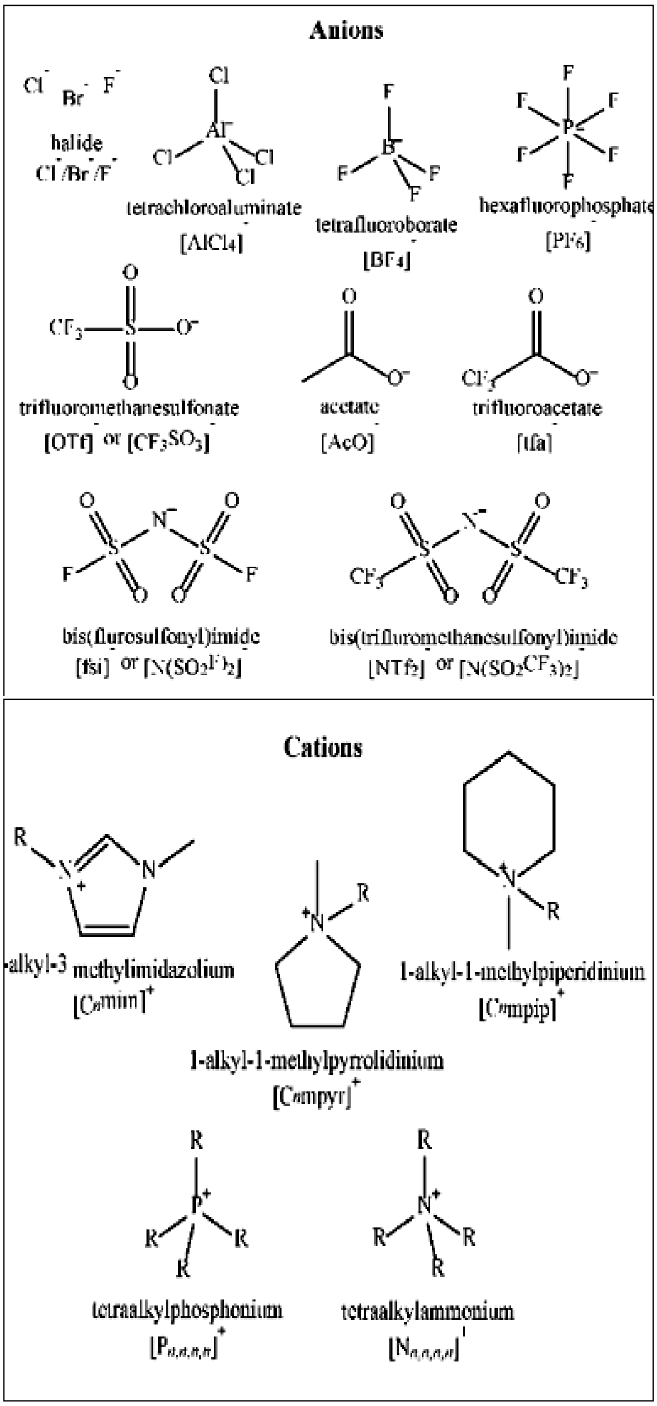

Fig 1 Anionic and Cationic Ionic Crystal

The nomenclature of an ionic liquid begins with the cation, followed by the anion. In the case of the most studied systems with imidazolium-based cations, cations are named based on the lengths of their alkyl chains, followed by "imidazolium." For example, a 1-butyl-3-methyl-imidazolium cation can be abbreviated as C_4C1Im, BMIm, BMIM, bmim, or ByMeIm. Anions are typically named according to general principles, such as using abbreviations like "Ac" for acetate. It's worth noting that more than 600 different ionic liquid systems can be generated from approximately 10 simple anions like $(BF_4)^-$ and $(PF_6)^-$ combined with variations of the 1-alkyl-3-methylimidazolium cation substituted with various alkyl groups in the 2-, 4-, or 5-position, or N-alkylpyridinium substituted in the 3- or 4-position. Additionally, when heteropolyanions and tetralkylammonium and tetraalkylphosphonium cations are considered, the total number of potential ionic liquid systems exceeds a quarter of a million. This wide variety allows for tailoring solvents to specific chemical reactions, as ionic liquids exhibit a range of physicochemical properties that can be finely tuned with remarkable precision, providing versatility and adaptability in various applications.

V. SYNTHESIS OF IONIC LIQUIDS

Ionic liquids can be synthesized using three main methods: quaternization, metathesis of halides, and the direct combination of a halide salt with a metal halide.

A. *Quaternization Reaction:*

Most quaternization reactions involving an annular nitrogen atom and an alkylating agent proceed via an SN2 mechanism, resulting in the inversion of the configuration of an achiral reagent. These reactions are typically kinetically controlled and form products irreversibly. Alkyl halides are commonly used as alkylating agents in quaternization reactions. In this process, the amine and the selected alkyl halide are mixed, and the reaction mixture is heated under an inert nitrogen or argon atmosphere (Bonhote et al., 1995; Chauvin, 1995). Additionally, quaternization can be performed using microwave irradiation (Namboodiri et al., 2002).

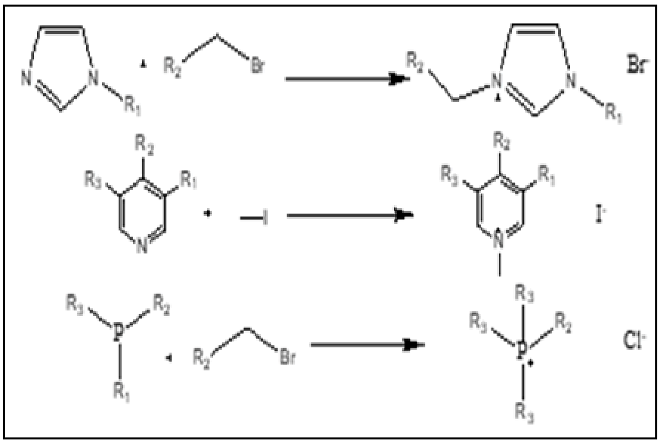

Fig 2 Quaternization Reaction

B. *Metathesis of Halide*

Many ionic liquids are synthesized through a metathesis reaction involving a halide or a similar salt of the desired cation. This metathesis reaction can be categorized into two types based on the water solubility of the target ionic liquid: metathesis via metal salts and metathesis via silver salts (Wilkes et al., 1990).

$$R \quad X \quad M^{+}A^{-} \longrightarrow R + A^{-} + M + X^{\cdot +}$$

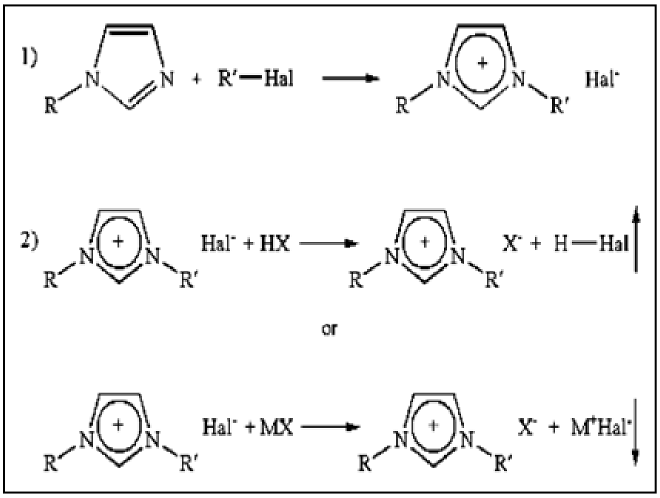

Fig 3 Metathesis of Halide

VI. PHYSICOCHEMICAL PROPERTIES OF IONIC LIQUIDS

The study and synthesis of ionic liquids are still relatively recent, yet they exhibit a wide array of physicochemical properties. Ionic liquids, also known as room-temperature ionic liquids (RTILs), remain in a liquid state over a broader temperature range compared to other liquids. Most 1-alkyl-3-methyl-imidazolium ionic liquids display a glass transition temperature ranging from -10°C to -60°C and exhibit chemical stability within the temperature range of 250°C to 400°C, with variations depending on the alkyl chain length and the choice of anion. Below their melting point, ionic liquids typically form glasses and exhibit a pronounced tendency for supercooling. The high thermal stability and minimal vapor pressure of ionic liquids make them well-suited for high-temperature reactions. The melting point of ionic liquids decreases with increasing alkyl chain length and increases with a higher degree of symmetry. The heat capacity of commonly used imidazolium-based ionic liquids was reported in 2003. The negligible vapor pressure of most ionic liquids can be attributed to the strong Coulombic forces between the anions and cations in the liquid. Ionic liquids are often more viscous than organic solvents, sometimes by several orders of magnitude. This wide variation in physicochemical properties, coupled with the ease of modifying their design, earned ionic liquids the moniker "designer solvents" in the latter part of the 1990s. Ionic liquids are typically polar solvents due to the presence of both cations and anions. However, they possess the unique ability to dissolve both polar and nonpolar solutes owing to their polarity, which contributes to their solvation capability. In ionic liquids, solvation occurs through various mechanisms, including ion-ion interactions, van der Waals forces, dipole interactions, and pi-pi interactions. The solubility of ionic liquids in water is an important consideration.

It primarily depends on the degree of coordination achievable with the ions present. Basic ions such as $(NO_3)^{-}$ can strongly coordinate with water, whereas acidic ions are non-coordinating. Neutral ions like $(BF_4)^-$ and $(NTf_2)^-$ exhibit weak coordination with water. Furthermore, the length of alkyl chains on the cation also influences the miscibility of water in an ionic liquid. Longer alkyl chains result in a more hydrophobic ionic liquid, affecting its solubility in water. This interplay between ion coordination and alkyl chain length contributes to the diverse solvation behaviour of ionic liquids and their applicability in various solvation processes.

VII. APPLICATION

The narrative around ionic liquids (ILs) has certainly evolved since they were hailed as "solvents of the future" in 2003. Initially, their potential to replace traditional solvents in industrial processes due to their low toxicity, flammability, and volatility seemed promising. However, as their usage has increased, concerns have emerged regarding their environmental impact and toxicity, particularly towards aquatic organisms. The manufacturing, use, and disposal of ILs now require careful consideration due to their potential environmental consequences. Studies have highlighted the impact of IL structure on toxicity, with factors such as the cation/anion choice, alkyl chain length, and functionalized alkyl groups playing significant roles. Certain ILs containing fluorine anions can pose additional challenges due to their hydrolytic instability, leading to the release of hazardous compounds like hydrofluoric acid (HF). This has led to a shift towards more stable alternatives, albeit at a higher cost. Despite these concerns, ILs still offer advantages over traditional solvents in terms of thermal stability, low flammability, and high conductivity. These properties make them suitable for applications in high-temperature processes, such as batteries, and even space-related endeavours. Additionally, their ability to disperse heat effectively during reactions has led to their use as electrolytes and heat transfer fluids. However, it's essential to remember that while ILs may offer benefits in terms of safety and performance compared to traditional solvents, thorough toxicity testing remains crucial, especially considering their potential environmental impact.

The interest generated by the prospect of replacing conventional solvents with ionic liquids (ILs) has been substantial in both academic and industrial circles. This shift aligns with EU REACH regulations, which aim to enhance safety and environmental protection by phasing out hazardous chemicals and encouraging the adoption of safer alternatives like IL-based processes. However, transitioning to IL-based processes requires thorough compatibility assessments with all operational units involved. While ILs indeed serve as alternative solvents, their versatility extends beyond this role.

Fig 3 Applications of Ionic Crystal

A. *Electrochemical Applications*

ILs are also extensively applied in electrochemical devices due to their favourable properties such as high ionic conductivity, wide electrochemical windows, and thermal stability. Their applications include**:**

- *Supercapacitors:*

ILs act as electrolytes in supercapacitors, providing high capacitance and energy density.

- *Lithium-Ion Batteries*

ILs are used as electrolytes in lithium-ion batteries, enhancing safety by reducing flammability and enabling stable operation at higher voltages.

- *Polymer-Electrolyte Fuel Cells:*

ILs improve the ionic conductivity and stability of polymer electrolytes, leading to better performance of fuel cells.

- *Dye-Sensitized Solar Cells:*

ILs serve as electrolytes, improving the efficiency and stability of dye-sensitized solar cells by providing a stable ionic environment (Lu W. et al., 2002; Silvester D., 2006).

B. *Applications in Organic Reactions*

Ionic liquids (ILs) have proven to be highly effective solvents for a variety of organic reactions, offering unique advantages over traditional solvents. Notable reactions where ILs have been utilized include:

- *Diels-Alder Reaction:*

ILs enhance reaction rates and selectivity due to their unique solvation properties.

- *Baylis-Hillman Reaction:*

ILs improve reaction efficiency and product yields.

- *Heck Reaction:*

ILs provide a stable environment for palladium catalysts, leading to increased reaction rates and selectivity.

- *Esterification:*

ILs facilitate the esterification process by providing a conducive medium that enhances the reaction kinetics.

- *Isomerization:*

ILs offer stability and control over the reaction conditions, leading to improved isomer yields.

- *Coupling Reactions:*

ILs serve as efficient solvents for various coupling reactions, including Suzuki and Sonogashira couplings, by stabilizing intermediates and catalysts (Kumar A., 2005; Fischer T., 1999).

C. *Extraction Technology*

Solvent extraction plays a crucial role in various industries, including nuclear reprocessing, ore processing, the production of fine organic compounds, and the perfume industry. The use of ionic liquids (ILs) for separating toxic metal ions and organic molecules has garnered significant attention. Currently, ILs are utilized as extraction solvents, particularly for extracting metal ions using crown ethers. This is especially important for the separation of rare earth elements, which, despite their similar chemical structures, are essential for various catalytic, electrical, magnetic, and optical applications. Traditional industrial methods rely on solvent extraction due to its recyclability and scalability, often using organophosphorus derivatives. The unique properties of ILs make them highly effective for these processes, enhancing efficiency and selectivity in the extraction of rare earth elements (Ahou D. et al., 2003; Dai S., 1997).

D. *Pharmaceuticals*

In the pharmaceutical industry, where approximately 50% of commercial drugs are organic salts, ILs have shown significant potential. Research has focused on developing ionic liquid forms of pharmaceuticals by combining pharmaceutically active cations with pharmaceutically active anions, resulting in dual-active ionic liquids. These innovative compounds combine the therapeutic effects of two drugs, potentially enhancing efficacy and reducing side effects.

E. *Gas Handling*

ILs are also highly suitable for gas storage and handling applications due to their distinctive properties, such as low vapor pressure, stability at high temperatures, and excellent solvation capabilities for a wide range of compounds and gases. The weakly coordinating anions and cations in ILs can stabilize polar transition states, making them ideal for various gas-handling processes. Additionally, many ILs can be reused with minimal loss of activity, which enhances their economic viability and sustainability in industrial applications.

F. *Applications in Renewable Energy and Waste Recycling*

ILs are particularly promising in renewable energy systems, such as solar thermal energy systems, where they serve as efficient heat transfer and storage media. These systems, like parabolic troughs and solar power towers, require materials that can operate at high temperatures (up to 600 °C or 1,112 °F), and ILs are well-suited due to their high thermal stability. In waste recycling, ILs facilitate the separation of synthetic goods, plastics, and metals, offering the specificity needed to differentiate similar compounds. This allows for more efficient recycling processes at lower temperatures, potentially reducing the environmental impact by minimizing the need for incineration or landfill disposal.

G. *Solar Thermal Energy*

Ionic liquids (ILs) hold significant promise as heat transfer and storage media in solar thermal energy systems. These systems, including concentrating solar thermal facilities like parabolic troughs and solar power towers, focus the sun's energy onto a receiver, generating temperatures up to approximately 600 °C (1,112 °F). The captured heat can then be used to produce electricity via steam or other thermodynamic cycles. ILs are particularly suited for this role due to their high thermal stability and ability to operate efficiently at such elevated temperatures. In addition to their application in renewable energy systems, ILs also show great potential in waste recycling, particularly for synthetic goods, plastics, and metals. ILs provide the specificity needed to separate similar compounds, such as different polymers in plastic waste streams. This can be achieved through lower temperature extraction processes compared to current methods, potentially reducing the need for incineration or landfill disposal of plastic. By facilitating more efficient and selective separation processes, ILs contribute to more sustainable recycling practices and resource recovery.

H. *Batteries*

Researchers have identified ionic liquids (ILs) as promising substitutes for water in metal-air batteries. ILs exhibit significantly lower evaporation rates compared to water, which extends battery life by reducing the rate of drying. Additionally, ILs possess an electrochemical window of up to six volts, far exceeding water's 1.23 volts. This broader electrochemical window supports the use of more energy-dense metals, potentially enabling energy densities ranging from 900 to 1600 watt-hours per kilogram. In analytical applications, ILs have found commercial use in specialized equipment such as gas chromatography (GC) columns and scanning electron microscopy (SEM) supports. IL-based GC columns offer the advantage of easy modification of polarity, combined with superior thermal and chemical stability compared to traditional stationary phases like polysiloxane polymers or polyethylene glycols. Di/polycationic systems, including those based on imidazolium and phosphonium, are particularly valued for their higher thermal stability and improved separation efficiency at elevated temperatures. In SEM applications, hydrophilic ILs are employed to maintain the structure of hydrated specimens without dehydration or drying. The negligible vapor pressure of ILs makes them suitable for use in low-pressure environments. Furthermore, the intrinsic conductivity of ILs can prevent the accumulation of electron charge, which is a common issue in SEM analysis. This characteristic could potentially eliminate the need for sputtering insulating samples with a thin layer of conductive metal, a standard practice in SEM to improve image quality and accuracy.

I. *Operating Fluids*

Ionic liquids (ILs) are extensively researched as operating fluids rather than just solvents, particularly in their roles as heat transfer materials or lubricants. Mettop GmbH, a company that specializes in metallurgical process technology, collaborated with Proionic to develop ILTEC, a new cooling technology. ILTEC allows the direct replacement of water with an IL of similar viscosity but with superior properties, such as a higher operating temperature (up to 250 °C) and chemical inertness. This substitution not only increases the operating temperature range but also improves energy efficiency through better heat recovery. The development of ILs as lubricants is another significant area of advancement. Their high thermal stability, conductivity, low volatility, and low flammability make them particularly valuable in high-friction environments where excessive heat generation can be a concern. The highly tunable nature of ILs permits the customization of properties, including viscosity, to meet the specific requirements of different applications. For example, Klüber Lubrication has commercialized the use of ILs to enhance the electrical conductivity of lubricants, demonstrating the practical benefits of ILs in improving lubricant performance in various industrial contexts.

VIII. CONCLUSIONS

Ionic liquids (ILs) have not been used solely for their "green" properties; rather, their success is also attributed to other advantageous properties such as low volatility and low flammability. The composition of ILs, being entirely ionic, enables their use in applications demanding high electrochemical and thermal stability and conductivity qualities that are unmatched by other pure materials. Despite being typically more expensive than conventional solvents, the initial higher capital cost of ILs can be justified by improvements in various aspects of industrial processes. These include enhanced solvent recyclability, better catalyst recovery, faster reaction rates, increased selectivity, and more efficient product separation. Consequently, ILs are not restricted to bulk solvent applications and catalysis; they are also finding niche applications in performance additives and analytical materials. Their strong solvation properties and tunable nature present significant advantages over traditional solvents. However, replacing conventional solvents with ILs in a process is not straightforward and often requires careful consideration of process compatibility.

While ILs are not inherently green, they can contribute to the sustainability of a process by improving its green metrics, making it more environmentally and economically sustainable. This is achieved through the increased recyclability of the solvent and recovery of the catalyst. Additionally, tailoring the hydrophilicity of ILs can aid in product separation, though it is crucial to consider how structural changes may affect the toxicity of the IL. The cost of ILs has been steadily decreasing due to the development of new and scalable synthesis routes. Some protic ionic liquids (PILs) are now comparable in price to conventional organic solvents, paving the way for broader industrial application of these materials. It is anticipated that the commercial use of ILs will continue to grow over the next decade, with ILs becoming more accepted as generic solvents to be considered for various reactions. Moreover, attention should be directed towards alternative classes of solvents, such as switchable solvents and porous liquids, which have the potential to offer similar benefits to industrial processes. The exploration of these alternatives may further enhance the range of applications and advantages provided by advanced solvent technologies in the future.

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