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SOME PHYSICAL AND THERMAL PROPERTIES OF IONIC LIQUIDS

Abstract: *The diverse application areas of ionic liquids based on their unique characteristics-and the expansion of these fields necessitate a detailed analysis of ionic liquids. In turn, this will facilitate the expansion of research into formulations based on various combinations of anions and cations enabling the synthesis of new ionic liquids with improved properties and lead to increased production volumes.*

Precise information regarding the thermal and physical properties of ionic liquids is essential for evaluating their suitability for various applications. Currently, significant attention is focused on issues such as why certain ionic liquids exhibit higher electrical conductivity than others and how this relates to ion diffusion rates—as well as the structural characteristics that determine the wide range of viscosities observed in ionic liquids.

Key words: *ionic liquid, viscosity, vapor pressure, nanostructures, thermal properties.*

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Introduction

Ionic liquids (ILs) have emerged as a prominent class of materials that have received substantial scientific attention over the past several decades, owing to their distinctive physicochemical characteristics and broad application potential. Continuous advances in ionic-liquid chemistry have resulted in the development of diverse structural families and successive generations of ILs with specifically tailored properties. This rapid expansion has stimulated extensive research into the fundamental physicochemical behavior of ILs, the rational design and synthesis of novel ionic-liquid systems, their processing and chemical-engineering aspects, as well as their implementation in laboratory, pilot-scale, and industrial applications.

Early research on ILs was predominantly associated with the principles of green chemistry, where these materials were investigated as alternative solvents to conventional volatile organic compounds. Their negligible or low volatility, high thermal stability, and tunable chemical structure have subsequently facilitated their integration into a much broader range of technological disciplines. At present, ILs are being extensively investigated in areas including catalysis, organic and polymer synthesis, electrochemistry, analytical chemistry, energy conversion and storage, nanotechnology, and biotechnology.

Within the field of polymer science, ionic liquids have attracted particular interest because of their multifunctional roles in polymer-related processes. They have been employed as alternative reaction media for polymerization, specialized solvents for the dissolution and processing of biopolymers such as cellulose, and as co-catalysts or initiating species in polymerization reactions. In addition, their incorporation into polymer formulations has enabled the development of functional additives and advanced polymeric materials with tunable properties.

A particularly significant development has involved the covalent incorporation of ionic-liquid-derived functionalities into polymeric architectures. This approach has enabled the emergence of a distinct class of charged polymeric materials generally referred to as polymeric ionic liquids (PILs), or poly(ionic liquid)s. These materials combine the structural characteristics of polymers with the physicochemical features associated with ionic liquids, thereby providing considerable opportunities for the design of functional polyelectrolytes with controllable properties.

The introduction of ionic-liquid-derived cationic and anionic moieties, including imidazolium, pyrrolidinium, and pyridinium cations, as well as tetrafluoroborate, hexafluorophosphate, and triflate anions, has significantly expanded the structural diversity of functional polymeric systems. Taking these factors into account, the present article reviews the viscosity, vapor pressure, transport properties, thermal decomposition, and other characteristics of ionic liquids to shed light on these questions.

Viscosity of ionic liquids

Viscosity is considered a key characteristic of ionic liquid systems, as the ionic liquid medium strongly influences the dissolution process and the diffusion of dispersed particles. Factors such as the nature, size, and symmetry of the cations and anions, as well as hydrogen bonding, typically play a decisive role in determining the viscosity of ionic liquids. Achieving low viscosity is one of the most significant objectives in research within the field of ionic liquids. Specifically, low viscosity facilitates use in the liquid

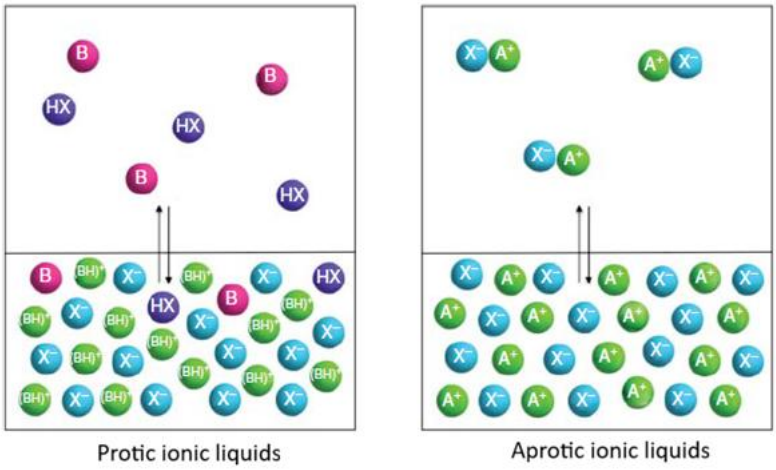
state whether for various scientific research purposes or for commercial processes while also enhancing the performance of electrochemical devices by enabling high ion transport.

The viscosity of ionic liquids is significantly higher at least tenfold than that of other solvents, such as water (approaching the consistency of oil or even honey). This is due to strong intermolecular interactions within the ionic liquid, such as van der Waals forces, hydrogen bonding, and Coulombic forces. Although mixing ionic liquids (ILs) with molecular solvents is considered the most effective method for reducing viscosity, this leads to increased vapor pressure, a higher tendency for flammability, and reduced electrochemical stability. As with other properties of ionic liquids, the experimental measurement of viscosity requires pure, dry ionic liquid and an inert atmosphere. The choice of viscometer for this purpose is made by taking into account the Newtonian or non-Newtonian behavior of the ILs [1,2]. When ionic liquids exhibit Newtonian behavior, their viscosity can be accurately measured using falling-sphere, capillary, or other types of viscometers. A rotational viscometer is used to evaluate the non-Newtonian viscosity of the ionic liquid.

The currently employed magnetic resonance method is considered a research technique that enables the acquisition of extensive information regarding the actual behavior of ionic liquids, both with and without the presence of suspended nanoparticles [3]. This method can be linked to the actual behavior of ionic liquids as well as to the ordering effects within their liquid structure [4,5].

Vapor pressure of ionic liquids

Although vapor pressure is considered a physically significant property regarding the application of ionic liquids (ILs), it has not been extensively studied. For instance, the dependence of this parameter on the nature of the ionic liquid and the precise meaning of "negligible" vapor pressure remain unclear. Contrary to earlier understanding in this field, Earle and his colleagues demonstrated that certain aprotic ionic liquids actually possess significant vapor pressure, as they can be distilled [6]. A combination of a Knudsen effusion apparatus and a quartz crystal microbalance can be cited as a method for measuring vapor pressure as a function of temperature [7,8]. It should be noted that the vapor pressure of aprotic ionic liquids differs significantly from that of protic ionic liquids. Specifically, in protic ionic liquids, this process occurs through the evaporation of the constituent acid and base as neutral molecules into the gas phase [9].



Pic.1. Description of the difference between protic ionic liquids and aprotic ionic liquids, which exist in the gas phase as tightly bound ion pairs.

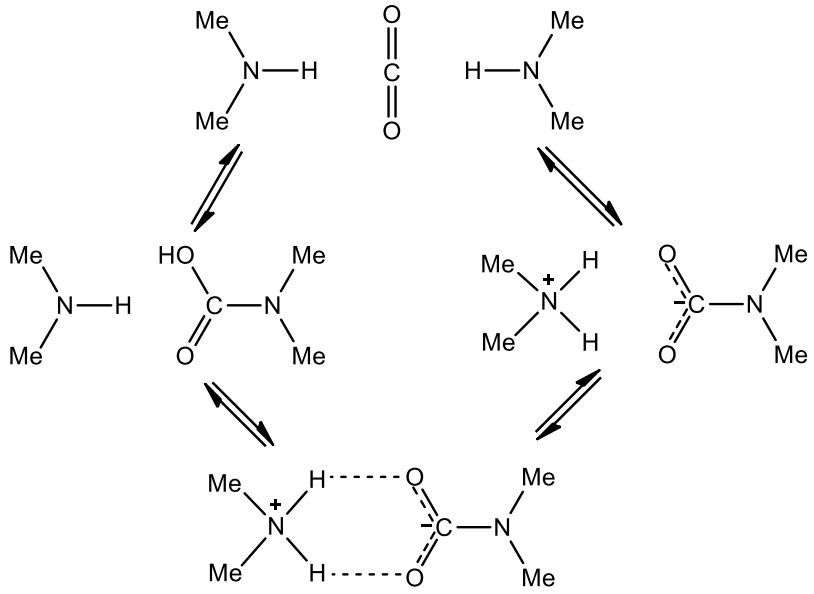
It is currently understood that aprotic ionic liquids exist in the gas phase as strongly associated ion pairs. This has been confirmed by both experimental and theoretical studies [10, 11]. Data regarding the enthalpy of vaporization of ionic liquids also provide insight into the strength of the interactions between ions in the liquid phase.

Although aprotic ionic liquids are often characterized as compounds with negligible vapor pressure, it has been noted that certain ionic liquids, such as [Cnmim][NTf2] (where n = 2, 4, 6, 8, 10, 12, and 16), can be distilled. This indicates that, at a certain temperature, there is sufficient energy to overcome the strong inter-ionic interactions within the liquid phase of the ionic liquid (IL), thereby enabling vaporization. Furthermore, upon cooling, these ions can condense to reform the ionic liquid phase without undergoing decomposition. Following this discovery, significant interest has arisen in identifying ILs capable of vaporizing without decomposition and in investigating the nature of ILs in the gas phase [9, 12]. The tendency of ionic liquids to vaporize under the

influence of temperature is of considerable practical importance. The ΔH_vap parameter is also significant for the thermodynamic modeling of ionic liquids, as it allows for the characterization of the strength of interactions between ions in the liquid phase.

Ionic liquid binary mixtures can also undergo vaporization; in this process, the more volatile neutral ion pair is distilled first. As previously discussed, the specific combination of cations and anions is not critical when two ionic liquids are mixed, since they form a mixture of the same composition in the liquid phase ([Cat1][A1] + [Cat2][A2] ≡ [Cat1][A2] + [Cat2][A1]). Furthermore, in the ideal state of an ionic liquid (IL) mixture, the mixture's vapor pressure depends solely on the two components and their molar ratios. Initial studies have demonstrated ideal behavior in mixtures of two aprotic ionic liquids [13].

Analogous theory applies to the composition of the gas phase as well; if [Cat1][A1] is the more volatile ion pair, it is the first component to form, regardless of which two ionic liquids were used to prepare the mixture. At the same time, for the distillation of the four possible ILs to be feasible, their vapor pressures must differ significantly. While the distillation of aprotic ionic liquids (ILs) requires high temperatures and high vacuum, other ILs can be distilled more easily. These include protic ionic liquids (PILs) and carbamate-based ILs. As previously noted, protic ionic liquids are obtained through the transfer of a proton from an acid to a base. The distillation of PILs is governed by the volatility of the neutral acid and base species; consequently, the vaporization processes differ significantly from those of aprotic ionic liquids, which involve the formation of ion pairs (Fig.2) [9].



Scheme 1. Dynamic equilibrium of N,N-dimethylammonium N′,N′-dimethylcarbamate [16].

Carbamate-based ionic liquids (ILs) are synthesized by combining carbon dioxide and a secondary amine in a 1:2 molar ratio. For instance, N,N-dimethylammonium N′,N′-dimethylcarbamate (DIMCARB, Scheme 3.1) and N,N-diethylammonium N′,N′-diethylcarbamate (DIETCARB) exist as liquids at room temperature [14]. However, at moderate temperatures (up to 60°C), these ILs can dissociate in the gas phase into their starting materials—namely, the amine and carbon dioxide. Conversely, recombination of the constituent amine and CO_2 leads to the reformation of the IL. Thus, when used as reaction media for dissolving and processing valuable materials such as biomass and lignite, these ILs can be easily removed from the product via distillation under very mild conditions [14, 15].

Nanostructures in ionic liquids

Another complex characteristic of ionic liquids is the formation of nanoscale structures within the liquid phase. Although this feature is not necessarily considered a primary advantage, it can significantly influence the properties of ionic liquids and the reactions in which they participate. This is an emerging field of research that encompasses X-ray and neutron diffraction techniques used to detect nanoscale structures [17]. The structure of the IM is more complex at the surface and at phase boundaries (liquid-liquid or liquid-gas interfaces), which in turn significantly influences its behavior in many areas of application.

Thermal properties of ionic liquids

Ionic liquids possess a number of thermal properties that are significant for their applications.

Among these properties, thermal stability is the most important and frequently cited. Precise information regarding the decomposition temperature of ionic liquids is crucial for evaluating their suitability for high-temperature applications. Such applications include lubrication, thermal energy storage, and use as heat-transfer fluids. Ionic liquids may also be subjected to temperature and other stresses when used as synthetic media to enhance the dissolution of biomaterials. Such a wide range of applications stems from the fact that the IM can be utilized under diverse conditions—such as in air or inert atmospheres, with or without gas flow, in contact with surfaces capable of catalyzing decomposition (e.g., aluminum), across volumes ranging from microliters to liters, and at high temperatures [18]. All these parameters can alter the thermal decomposition temperature or the rate of decomposition. Therefore, these factors must be taken into account when determining the most appropriate test conditions for evaluating thermal stability. The thermal stability of the IM, the quantitative measure of its decomposition rate, and the analysis of decomposition products are investigated using thermogravimetric analysis (TGA) coupled with mass spectrometry or via pyrolysis-mass spectrometry [19]. The most widely used method for evaluating the thermal stability of the IM is thermogravimetric analysis (TGA) conducted under conditions of linear temperature change (10°C per minute). This is a convenient and time-efficient method for the preliminary assessment of the thermal stability of various IMs. In some cases, this analysis also reveals a multi-stage decomposition process. In this analysis, the mass loss of the sample is recorded over time, making it possible to determine, for instance, the time required for a 1% mass loss. This enables the comparison of different insulating materials and allows one to track how the decomposition time changes with rising temperatures—specifically, the rate of the decomposition process at high temperatures. One significant outcome of such an analysis is the determination of the maximum operating temperature for the insulating material. In addition, it must be borne in mind that some ionic liquids (ILs) may vaporize at high temperatures, resulting in measurable mass loss (particularly when measured using TGA gas flow). Furthermore, when evaluating the impact of a specific structural change, the nature of the ions in the IL must also be considered a crucial factor. For instance, if the anion employed promotes decomposition at a relatively low temperature, then minor structural modifications to the cation may be disregarded. Based on the research conducted, certain general observations can be made:

– increasing the length of the alkyl chain in the cation slightly reduces the stability of the ionic liquid; this occurs because, as expected, compounds with longer alkyl chains form more stable carbocations or carbon radicals;

– the effect of branching in the alkyl chain depends on the anion used, as the decomposition mechanism may vary based on the anion (e.g., SN1 or SN2);

– the incorporation of oxygen-containing fragments into the cation typically results in reduced stability. – For instance, ionic liquids (ILs) bearing oxazolidinium or morpholinium cations are characterized by relatively lower stability compared to those with side chains containing alkoxy groups or with pyrrolidinium and piperidinium cations (which possess analogous ring structures but lack an oxygen heteroatom) [20-22];

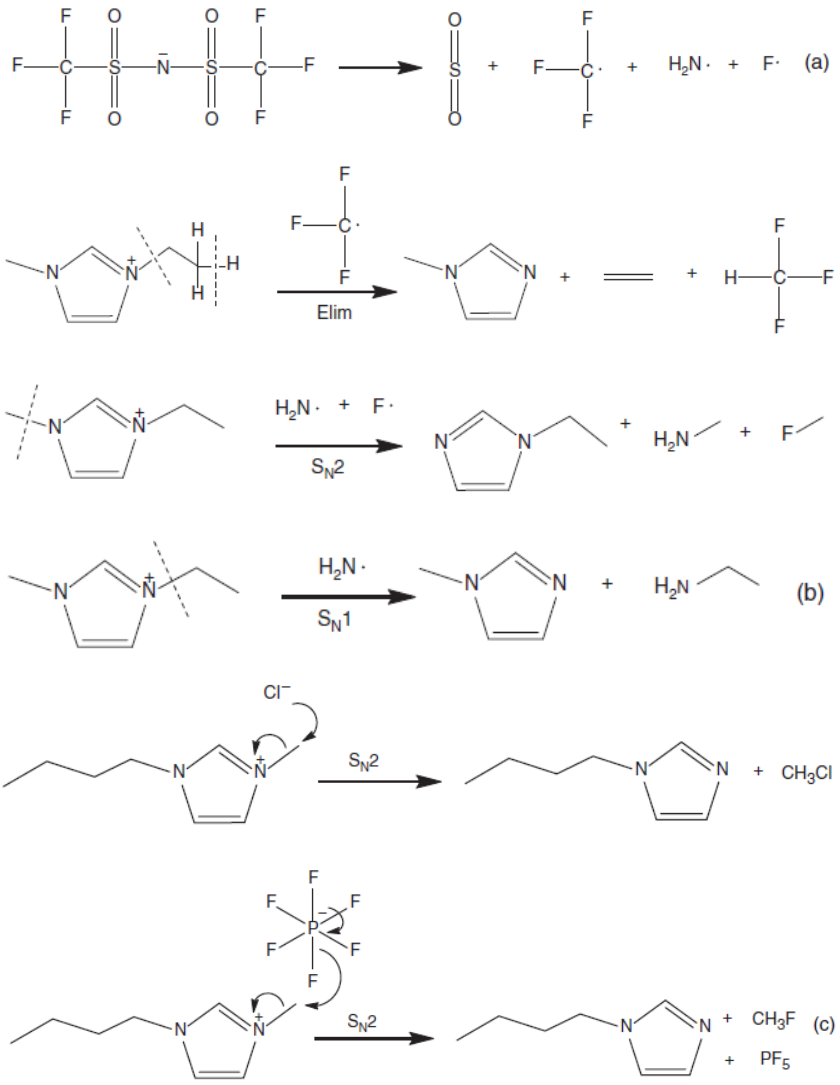
– Dicationic ionic liquids (ILs) possess higher thermal stability compared to monocationic ILs. This applies to both nitrogen-based and phosphonium-based dicationic ILs [23, 24]. The stability of the cations varies in the following order: phosphonium > pyrrolidinium > imidazolium > tetraalkylammonium > sulfonium.

Replacing the acidic proton at the 2-position of the imidazolium ring via methylation enhances thermal stability. In contrast, the use of long alkyl chains at this position has little to no effect [18]. Anions with low nucleophilicity positively influence stability; this is because they exhibit a reduced tendency to attack the cation, allowing the overall stability to be ranked in the order: $[NTf2]^-$ > $[BF4]^-$ ≈ $[PF6]^-$ > halides. For example, the stability of [C_2mim]-based ionic liquids varies according to the following anion series [25]:

$[CF3SO3]^-$ > $[CH3SO3]^-$ > $[CH3CO2]^-$ > $[CF3CO2]^-$

The $[FSI]^-$ anion is less thermally stable than the $[NTf2]^-$ anion, owing to the susceptibility of the FSO_2 group to decomposition [21].

The thermal decomposition mechanism of ionic liquids is quite complex. The nature of the cation and anion plays a particularly significant role here. It is presumed that factors such as the substrate, the medium, and impurities further complicate the decomposition mechanism. Just as mathematical modeling is employed to predict the decomposition pathways requiring the lowest energy—allowing for comparison with experimentally determined products—the use of hyphenated techniques such as TGA-MS (thermogravimetric analysis-mass spectrometry) or Py-GC (pyrolysis-gas chromatography) is crucial for identifying both decomposition temperatures and decomposition products [26-29]. Furthermore, it should be noted that energetic materials (EMs) can decompose via endothermic or exothermic processes; this provides additional insight into the decomposition mechanism [18,29]. As an example, the possible decomposition mechanisms for the widely used [C_2mim][NTf2] can be shown:



Scheme 2. Decomposition mechanism (a) in the presence of [C4mim][PF6], taking into account the possible mechanisms of subsequent attack by the NTf2 anion on the [C2mim] cation in [C2mim][NTf2] (b) and the possible decomposition pathways of [C4mim][Cl] (c).

Mass spectrometric analysis should demonstrate that, when the sampled material undergoes fragmentation or chemical transformation, distinct products are formed via various mechanisms. Elimination reactions are a commonly observed mode of fragmentation for ionic liquids, and this has been proposed as the dominant mechanism for [C2mim][NTf2] [29,30]. The nucleophilic attack of the anion on the cation can proceed via either a unimolecular (SN1) or a bimolecular (SN2) mechanism (Scheme 3.2, b). Of these two mechanisms, SN2 fragmentation is more favorable than SN1 fragmentation for [C_2mim][NTf2]. The theoretical differentiation of these mechanisms across various ionic liquids stems from the fact that the rate of the SN2 reaction depends not only on the strength of the nucleophile (e.g., variations observed with different halogens) but also on the nature of the substrate undergoing attack (e.g., steric hindrance associated with different cations). It should be noted that for [C_2mim][NTf2], since the anion does not act as the nucleophile in the substitution reaction, the anion must first dissociate—a phenomenon that has been experimentally observed [29]. Since the SN2 reaction is considered to have the lowest energy barrier, it has been accepted as the preferred mechanism for the decomposition of [C_4mim][Cl], [C4mim][PF_6], and [C4mim][BF4] compounds (Scheme 3.2, c) [29]. It should be noted that this process occurs in the absence of water, as the $[PF_6]^-$

and $[BF_4]^-$ anions readily undergo hydrolysis in the presence of water to form HF. Experimental and computational studies also indicate that the decomposition of [C2mim][OAc] proceeds primarily via an SN2 mechanism (involving the attack of the acetate group on the methyl substituent of the imidazolium ring) [28]. These ionic liquids are being extensively studied for biomass processing, and research into their thermal decomposition indicates that they should not be used at temperatures exceeding 120°C.

CONCLUSION

Ionic liquids have emerged as versatile functional materials whose physicochemical properties can be systematically tailored through the appropriate selection and combination of cations and anions. Their distinctive characteristics, including low volatility, tunable viscosity, high ionic conductivity, and comparatively high thermal stability, have contributed to their expanding use in diverse scientific and technological applications [31-43].

The analysis presented in this review demonstrates that the physical and thermal properties of ionic liquids are strongly dependent on their molecular structure, the nature of the constituent ions, intermolecular interactions, and experimental conditions. Viscosity is particularly important because it directly affects mass transport, ion mobility, diffusion, and the performance of ionic-liquid-based electrochemical and catalytic systems. The ability to control viscosity through structural modification or appropriate formulation is therefore essential for optimizing the practical performance of ILs.

Although ionic liquids are commonly regarded as materials with negligible vapor pressure, studies have demonstrated that their vaporization behavior is more complex. Depending on their chemical structure, ionic liquids may undergo vaporization through the formation of strongly associated ion pairs or through the release of neutral molecular species. This distinction is particularly relevant for aprotic, protic, and carbamate-based ionic liquids and provides important information for their purification, processing, and potential recovery by distillation.

The presence of nanoscale structural organization represents another important characteristic of ionic liquids. Such structural heterogeneity can influence their transport, interfacial, and solvation behavior and may therefore play a significant role in determining their performance in technologically relevant systems. In parallel, thermal stability remains a critical parameter for evaluating the suitability of ILs for high-temperature applications. Thermal decomposition is governed by both the cation and anion structures and may proceed through different reaction pathways, including elimination and nucleophilic substitution mechanisms.

Thermogravimetric and mass-spectrometric techniques, together with complementary experimental and computational approaches, provide valuable tools for identifying decomposition temperatures, reaction pathways, and degradation products. The available evidence indicates that thermal stability can be enhanced or diminished by specific structural modifications, including changes in alkyl-chain length, ion symmetry, heteroatom incorporation, charge density, and anion nucleophilicity. Therefore, thermal stability should not be considered an intrinsic property of an ionic-liquid family alone but rather as a structure-dependent characteristic that must be evaluated under defined experimental conditions.

Overall, a comprehensive understanding of the relationships between ionic structure, physical properties, transport behavior, and thermal decomposition is essential for the rational design of next-generation ionic liquids. Such knowledge can facilitate the development of ILs with optimized properties for applications in energy storage, electrochemistry, catalysis, separation technologies, polymer science, biomass processing, and thermal management. Future research should focus on establishing more reliable structure–property relationships, improving the long-term stability and environmental compatibility of ionic liquids, and developing task-specific ILs capable of meeting the increasingly demanding requirements of advanced technological applications.

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