Measurement of vapour pressures of ionic liquids and other low vapour pressure solvents†

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The vapour pressures of several ionic liquids, liquid polymers and derivatives of glycerol were investigated by thermogravimetric analysis (TGA). The experimental method is described and discussed. Vapour pressure data for various solvents with low vapour pressures are reported at 100–120 °C. The thermogravimetric method for vapour pressure measurement is useful for the rapid screening of solvents. Ionic liquids have a low but detectable vapour pressure at 100–120 °C. The vapour pressures of liquid polymers such as poly(ethylenimine) and polyethylene glycols are in the same range, which might make these substances a less expensive alternative to ionic liquids.

Introduction

In recent years, ionic liquids have been proposed as alternative solvents for catalysis and separation processes. The main advantage of ionic liquids is their low vapour pressure. The use of solvents with low vapour pressure can overcome the existing problems of loss of solvent due to evaporation during reaction and separation processes. This could have benefits economically as well as environmentally.

The potential use of ionic liquids covers a wide variety of applications such as catalysis, extraction, absorption, distillation, and chromatography.[1–6] For these applications it is usually assumed that the vapour pressure of the ionic liquid is zero at room temperature. However, almost no vapour pressure data for ionic liquids have been reported so far.[1] Some studies have been carried out showing that ionic liquids can in fact be vaporised at low pressures and high temperatures.[4,7] For some ionic liquids, vapour pressure data could be obtained at temperatures below 300 °C.[3,8–9]

The knowledge of vapour pressure data is important for thermodynamic modelling as well as for comparison of different ionic liquids.[1,2] Therefore it is desirable to get a wide range of data on vapour pressures of ionic liquids. A rapid screening method for vapour pressure measurement is the use of a thermogravimetric analyser (TGA). This method has the advantage that only a small amount of substance is needed. The TGA method has been used to investigate the vapour pressure of solids as well as liquids.[10–14]

A disadvantage of ionic liquids for their use in industrial processes is their relatively high cost. Solvents such as liquid polymers or substances based on glycerol also have low vapour pressures and may be a cheap alternative to ionic liquids. However, few literature data are available on the vapour pressures of substances of this kind. It is therefore difficult to rate the vapour pressures of these substances in comparison with the vapour pressures of ionic liquids.

In this work, the TGA method was used to investigate the vapour pressures of several ionic liquids, liquid polymers and glycerol-based substances. The method is described and the results for the vapour pressures of the low vapour pressure solvents are presented and verified against existing literature data. The vapour pressures of the ionic liquids and the alternative solvents are also compared.

Materials and methods

Materials

The ionic liquids 1-ethyl-3-methylimidazolium ethylsulfate, 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide, and 1-hexyl-3-methylimidazol-ium trifluoromethanesulfonate as well as the poly(ionic liquid) poly[(p-vinylbenzyl) trimethylammonium hexafluorophosphate], were investigated along with the solvents glycerol, 4-hydroxymethyl-1,3-dioxolan-2-one, tetra(ethylene glycol) dimethyl ether, polyethylene glycol 150 dimethyl ether, polyethylene glycol 200, polyethylene glycol 300, polyethylene glycol 600 and polyethylenimine. Some properties of these substances are listed in Table 1 and their molecular structures are shown in Fig. 1.

Glycerol (98%) was obtained from Prolabo. Polyethylene glycol 600 (PEG 600) was obtained from Acros. Polyethylene glycol 200 (PEG 200), polyethylene glycol 300 (PEG 300), tetra(ethylene glycol) dimethyl ether (tetraglyme, 99%), polyethylene glycol 150 dimethyl ether (PEGDME 150), 4-hydroxymethyl-1,3-dioxolan-2-one (glycerol carbonate), and polyethylenimine (branched) were obtained from Sigma Aldrich. 1-Hexyl-3-methylimidazolium trifluoromethanesulfonate ([hmim][triflate]) was supplied by Merck. All substances were used as received.

1-Ethyl-3-methylimidazolium ethylsulfate ([emim][$EtSO_4$]) was synthesised according to a procedure described by Holbrey *et al.*[15] 1-Butyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide ([bmim][Tf_2N]) was synthesised according to a

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Table 1 Properties of the investigated substances (data from supplier)

Substance	Molar mass (g/mol)	Melting point (°C)	Boiling point (°C)	Density (g/cm^3)	Viscosity (10^{-3} Pa s)
[emim][EtSO4]	236.29			1.24	
[bmim][Tf2N]	419.36	−4[a]		1.429[a]	52[a]
[hmim][triflate]	330.37			1.04	
P[[VBTMA][PF6]]	321.24[b]				
Glycerol	92.09	20		1.25	
Glycerol carbonate	118.09			1.4	
Tetraglyme	222.28	−30	275 · · · 276	1.011	
PEGDME 150	~150		> 250		
PEG 200	~200	−55 to −40		1.124	60
PEG 300	~300				
PEG 600	~600	17 to 22		1.12	150 to 190
Polyethylenimine	~10,000[c]		250	1.03	

[a] From Bonhote *et al.*[16] [b] Molar mass of monomer unit ($C_{12}H_{18}NPF_6$). [c] Measured by GPC.

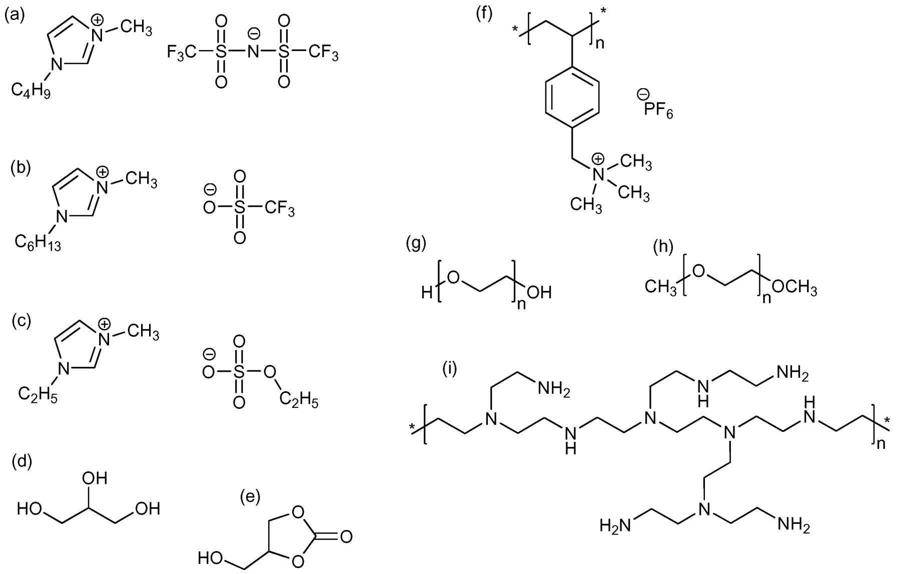

Fig. 1 Molecular structures. (a) [bmim][Tf_2N], (b) [hmim][triflate], (c) [emim][$EtSO_4$], (d) glycerol, (e) glycerol carbonate, (f) P[[VBTMA][PF_6]], (g) PEG, (h) PEGDME (tetraglyme: n = 4), (i) polyethylenamine.

method described by Bonhote *et al.*[16] Poly[(p-vinylbenzyl)-trimethylammonium hexafluorophosphate] (P[[VBTMA]-[PF_6]]) was synthesised as described by Tang *et al.*[17] The purity of the synthesised substances was confirmed by ^{1}H NMR.

Experimental method

A TGA7 thermogravimetric analyser (Perkin Elmer) running Pyris software was used to measure the evaporation rates of the substances. The experiments were performed at atmospheric pressure. A constant flow of gas (nitrogen or carbon dioxide, both supplied by BOC, flow rate approximately 50 cm^3/min) was maintained throughout the experiment. A small amount of the liquid sample (10–50 mg) was placed in a platinum sample pan and suspended in the furnace of the thermogravimetric analyser. The sample was heated to the experimental temperature (100–120 °C): the temperature of the sample was measured using a thermocouple placed directly under the sample pan. The sample was held isothermally at the experimental temperature for at least 30 min and the mass of the sample was recorded. After an initial period of evaporation of solvent and water impurities, the rate of mass loss of the sample remained constant and a plot of sample mass *versus* time resulted in a straight line. A linear fit over a period of 10 min was then used to obtain the evaporation rate of the pure substance.

Calculation of vapour pressures from evaporation rates

The following equation by Langmuir[18] was used for the calculation of vapour pressures from evaporation rates:

$$(dm/dt) = \alpha\sqrt{\frac{M}{2\pi RT}}P \tag{1}$$

where (dm/dt) is the evaporation rate, α is the evaporation coefficient, M is the molecular mass of the evaporating substance, R is the universal gas constant, T is the temperature, and P is the vapour pressure.

The equation is based on the kinetic gas theory and was originally derived by Langmuir for the evaporation of solids or liquids. The evaporation coefficient α depends on the experimental conditions such as pressure, temperature, presence of gas, and on the substance used. In many cases α can be regarded as being independent of the substance, and the value of α can be obtained *via* experiments using a reference substance of known vapour pressure, under the same experimental conditions.

Equation 1 can be written in the form:

$$P = kv \tag{2}$$

$$k = \frac{\sqrt{2\pi R}}{\alpha} \tag{2a}$$

$$v = (dm/dt)\bullet\sqrt{\frac{T}{M}} \tag{2b}$$

The factor v includes the experimental conditions. If α can be regarded as constant then the factor k, which contains the coefficient α, will only depend on the experimental method and not on substance or temperature. Price and Hawkins applied this equation to four different substances with known vapour pressures.[14] They found a linear relationship between P and v, with the same factor k valid for all the substances investigated. The results for other substances were also in good agreement with literature data. This indicates that the coefficient α can indeed be regarded as constant.

In order to be able to regard the coefficient α as a constant, Phang *et al.* suggested choosing an appropriate reference substance for each substance under investigation.[10] The reference substance should be similar to the experimental substance in terms of melting and boiling points as well as molecular structure.[10] Under identical experimental conditions, the value of α can then be assumed to be constant and α can be eliminated from equation 1. The vapour pressure of a substance S can then be calculated according to the following equation:

$$P_s = \left(\frac{M_{ref}}{M_s}\right)^{0.5} P_{ref} \frac{(dm/dt)_s}{(dm/dt)_{ref}} \tag{3}$$

In this study, glycerol was used as the reference substance (see ESI†). Queste *et al.* investigated the vapour pressures of various substances[19] based on ethylene glycol, propylene glycol and glycerol in the temperature range of 25 to 50 °C. They also measured the evaporation rates of these substances in relation to butyl acetate as a reference. The evaporation rates were measured using the TGA method, whereas the vapour pressures were measured independently using a different method. The comparison of the vapour pressures with the relative evaporation rates shows a correlation between the vapour pressures for each temperature and the relative evaporation rates measured using the TGA method. This confirms that the calculation of vapour pressures using TGA evaporation data is reliable.

Results and discussion

Vapour pressures of molecular solvents

The vapour pressures of several substances at 95 °C were calculated from isothermal TGA data according to the above equations. The results are shown in Table 2. The literature values for glycerol are included as it is the reference material in the vapour pressure determinations. Because of this no experimental values are given for glycerol. The temperatures for all the material evaluations chosen were based on expected process temperatures for a number of reactions. Data for ionic liquids are difficult to obtain and an extensive search of databases has resulted in a very small number of citations. Where available, these are reported.

Table 2 Vapour pressure data obtained from experiments and literature values

Substance	Temperature (°C)	Vapour pressure (Pa)	Literature values (Pa)
Glycerol	95	—	15.8[a]
	100	—	23.0[a]
	120	—	92.1[a]
Tetraglyme	95	60.5	87[b]
		—	190[c]
		—	90[d]
PEGDME150	95	78.8	N/A
Glycerol carbonate	95	7.6	7.2[e]
PEG200	95	9.9	N/A
PEG250	95	—	0.0002[d]
PEG300	95	1.5	N/A
PEG600	95	0.2	N/A
Polyethylenimine	95	0.2	N/A
[emim][$EtSO_4$]	120	2.0	N/A
[hmim][triflate]	120	1.0	0.007[f]
	172	—	0.007[f]
	200	—	0.2[f]
[bmim][Tf_2N]	120	1.2	0.004[f]
[bmim][Tf_2N]	165	—	0.004[f]
[bmim][Tf_2N]	244	—	0.5[f]
P[[VBTMA][PF_6]]	100	0.9[g]	N/A

[a] Vapour pressure values for the reference substance glycerol are literature values. [b] Extrapolated from data, Chaudhari[20] 1995. [c] Extrapolated from data, Kuczynski.[21] [d] Taken from graph, Hartel.[22] [e] Extrapolated from data, Chernyak.[23] [f] Experimental and extrapolated data from Dong, 2007[24–25] [g] Calculated using molar mass of monomer unit.

The polyethylene glycol ethers have the highest vapour pressures. The vapour pressure of polyethylene glycol 150 dimethyl ether is higher than that of tetraglyme, as tetraglyme has a higher molecular mass due to longer hydrocarbon chains, 4 monomer units per molecule, compared to an average of only 2 monomer units per molecule in PEGDME 150.

The vapour pressures of the other molecular solvents investigated in this study are comparatively low. The increase in molecular mass in the order PEG 200-PEG 300-PEG 600 results in a decrease in vapour pressure of approximately one order of magnitude. The lowest vapour pressures were found for PEG 600 and polyethylenimine, a branched polymer with a high molecular mass.

Literature data are available for some of the substances investigated, albeit at different temperatures. The vapour pressure of tetraglyme was investigated at temperatures above 100 °C.[23] and above 150 °C.[26] Extrapolation of the respective equations to the experimental temperature of 95 °C results in vapour pressures of 87 Pa and 190 Pa, respectively, compared to the value of circa 60 Pa obtained in this study. While the vapour pressure obtained in this way from the data in Kuczynski *et al.*,[21] 190 Pa, is significantly higher than the value obtained in this study, the value of 87 Pa calculated from Chaudhari *et al.*[20] is in fair agreement with the result of this study. The experimental data measured by Chaudhari *et al.* are much closer to the experimental temperature in this study and thus the respective extrapolation is more accurate than for the data by Kuczynski *et al.* This may explain the discrepancy of the vapour pressure results in this study from Kuczynski *et al.* The vapour pressure of tetraglyme was also reported in graphic form by Hartel[22] in the temperature range used in this study. At the temperature of 95 °C a vapour pressure of circa 90 Pa is obtained, which is also in good agreement with the results of this study.

Vapour pressure data for temperatures above 150 °C can also be found for glycerol carbonate.[23] Extrapolation gives a value of 7.2 Pa at 95 °C. This is in good agreement with the value of 7.6 Pa obtained in this study.

Hartel[22] also reported vapour pressure data for PEG 250 at temperatures above 140 °C. Extrapolation results in a value of circa 0.0002 Pa at 95 °C. This value is several orders of magnitude lower than the vapour pressures obtained in this study for PEG 200, PEG 300 and even PEG 600. Hartel does not give any explicit experimental values, nor does he explain the experimental method or cite any original literature. However, it cannot be concluded that Hartel's values are wrong, as the values obtained by Hartel for tetraglyme seem to be in line with the results in this study. The fact that the data by Hartel had to be extrapolated from graphic data at higher temperatures in order to compare them to the experimental data in this study may well contribute to the high discrepancy.

It cannot be completely ruled out that the results are not the vapour pressures of the pure substances, but of the substances containing small amounts of impurities such as water. In the presence of water, vapour pressure is expected to be higher than for the pure substance.[26] As most of the substances in this study are hygroscopic, this might apply to almost all the experimental data. This is a particularly important aspect for the reference substance glycerol, as any error in the reference experiment will result in errors in all the other calculated vapour pressures. Therefore it has to be ensured that the glycerol samples are pure at the time of measuring the evaporation rate. For this reason, experiments with a longer drying time of 60 min were carried out with glycerol at the same temperature. No significant difference was found in the evaporation rates obtained after 60 min compared to the values obtained after 30 min.

Water impurity and therefore too high evaporation rate for the PEG samples in this study might be a possible explanation for the deviation of the respective vapour pressure data from the literature value for PEG 250.[22] However, this would mean that the PEG samples used in this study contained a rather high amount of water. This would be very surprising, particularly because the other investigated substances are also hygroscopic but do not seem to contain much water impurity after the same pre-treatment procedure, as the vapour pressures seem to be in good agreement with literature values.

Queste *et al.* investigated the vapour pressures of various substances based on ethylene glycol, propylene glycol and glycerol in the temperature range of 25 to 50 °C.[19] These substances have a similar structure to the ones used in this study, but Queste *et al.* used only substances with low boiling points. Their vapour pressures reached values of 60 Pa at 50 °C for the glycerol-based substances with the lowest volatility.

It has to be taken into account that hydrogen bonds have an impact on the evaporation rate.[19,27] Therefore one has to be careful when comparing substances with different extent of hydrogen bonding whose vapour pressures were measured from evaporation rates with the TGA method. All in all, however, the results of this study agree with available literature data, and so the experimental method seems to give reasonably accurate results for different substances using a quick and simple method.

Vapour pressures of ionic liquids

Evaporation rates for several ionic liquids were measured using the TGA method at 120 and 100 °C. The ionic liquids chosen were those routinely used in our research group as reaction solvents or adsorbents for carbon dioxide capture. The results for the vapour pressures of the ionic liquids calculated from the experimental data are also shown in Table 2. It can be seen that the vapour pressures of the ionic liquids are in the range 1–2 Pa at 120 °C. This is in the order of magnitude of the polyethylene glycols with higher molecular weights (PEG 300–600).

As the molar mass of the poly(ionic liquid) is unknown, the mass of the monomer unit was used instead for calculation of the vapour pressure. This means that in reality the vapour pressure may be even lower than the value given in Table 2, depending on how many monomer units form one average molecule in the gas phase.

To date, very few literature data exist on the vapour pressures of ionic liquids. Most of the available data are based on binary or ternary mixtures with conventional organic solvents and so deal with the vapour pressures of the solvents rather than the ionic liquids themselves. However, some studies on the evaporation of ionic liquids have been performed.[1] The estimation of ionic liquid vapour pressures from calculated and experimental calorimetric data resulted in values of circa 10^{-10} to 10^{-11} Pa at room temperature and circa 10^{-4} Pa at 125 °C for 1-butyl-3-methylimidazolium hexafluorophosphate ([bmim][PF_6]).[27,28] These values are several orders of magnitude lower than the results of this study. However, these literature results are mainly based on calculations and estimations and therefore not very accurate. None of the data available are derived from effusion studies except those from Zaitsau *et al.*,[9] who used the Knudsen method. The only data for a pure ionic liquid is for 1-alkyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imides over a range of elevated temperatures.[9,24–25]

Measurements of the vapour pressures of 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([emim]-[NTf_2]) as well as 1-butyl-3-methylimidazolium dicyanamide ([bmim][DCA]) resulted in values of 0.1 to 2 Pa for the vapour pressure at temperatures between 180 and 260 °C, and 2×10^{-6} and 12×10^{-6} Pa respectively at room temperature.[3]

Boiling points of some ionic liquids were estimated from experimentally obtained surface tensions and densities.[4] For ionic liquids with the same cation there was a tendency of decreasing boiling point with anions in the order BF_4 > PF_6 > NTf_2. Longer alkyl chains in the methylimidazolium cation resulted in a decrease in the boiling point, indicating an increase in vapour pressure. However, according to a later study with the same type of ionic liquids, longer alkyl chains in the cation reduced the evaporation rate and therefore the vapour pressure.[7] The same trend was found by Zaitsau *et al.*[9] in an experimental investigation of the vapour pressures of 1-alkyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imides. The vapour pressure decreased with increasing alkyl chain length in the cation. This is in agreement with the results in this study (Table 2) where the vapour pressure decreases for the ionic liquids in the order of the cations emim > bmim > hmim. However, in this study the anions were different for each ionic liquid. As the anion can affect the vapour pressure, no conclusions about the influence of the alkyl chain in the cation can be drawn from the comparison of the ionic liquids in this study.

For the ionic liquid [bmim][NTf$_2$], Paulechka *et al.* found a vapour pressure of 7×10^{-5} Pa at 120 °C,[29] and Zaitsau *et al.*[9] obtained a vapour pressure of 8×10^{-5} Pa at the same temperature. Both values agree well with each other and are significantly lower than the 1.2 Pa found in this study.

Conclusion

The TGA method is a useful screening method for vapour pressure measurements with only small amounts of substance required. The experiments are quick and easy and yet result in reasonably accurate results for comparing different substances.

Room temperature ionic liquids can have detectable vapour pressures at temperatures of 100–120 °C. This has to be considered when using ionic liquids as alternative solvents in reaction and separation processes. The vapour pressures of ionic liquids are, however, very small even compared to molecular solvents with low vapour pressures such as glycerol.

Considering their low vapour pressures, polymeric substances with high molecular weight such as PEG 600 and poly(ethylenimine) could be even more suitable for processes where a low vapour pressure is favourable. However, these substances also have the highest viscosities. Both poly(ethylene imine) and PEG 600 are highly viscous at room temperature. This would cause problems with pumping the liquid, and pipes would have to be heated in order to maintain the fluidity of the substance.

Polyethylene glycols with molecular masses in the range of 300–400 Da might be a suitable alternative solvents, however. Their viscosity is in the order of magnitude of ionic liquids and their vapour pressure is comparable to those of the ionic liquids investigated in this study. Furthermore, they are available at a comparatively low cost. Of course, the solvent properties of the substance are also of importance when choosing an appropriate solvent for a process.

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