



Industrial application of ionic liquids for the recoveries of spent paint solvent



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ABSTRACT

The recovery of industrially valuable organic solvents from liquid waste, generated in chemical processes, is economically crucial to countries which need to import organic solvents. In view of this, the main objective of this study was to determine the ability of selected ionic liquids, namely, 1-ethyl-3-methylimidazolium ethylsulphate, [EMIM][ESO₄] and 1-ethyl-3-methylpyridinium ethylsulphate, [EMPy][ESO₄] to recover aromatic components from spent paint solvents. Preliminary studies done on the liquid waste, received from a paint manufacturing company, showed that the aromatic components were present in the range of (6–21)% by volume. The separation of the aromatic components was performed with the ionic liquids listed above. The phases, resulting from the separation of the mixtures, were analysed with a gas chromatograph (GC) coupled to a FID detector. Chromatograms illustrate that the chosen ZB-Wax-Plus column gave excellent separation of all components of interest from the mixtures, including the isomers of xylene. The concentrations of aromatics recovered from the spent solvents were found to be in the % ranges of (13–33) and (23–49), respectively for imidazolium and pyridinium based ionic liquids. These results also show that there is a significant correlation between π -character of ionic liquids and the level of extraction. It is therefore concluded that ionic liquids have the potential for macro-scale recovery of re-useable solvents present in liquid waste emanating from paint manufacture.

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1. Introduction

Oil paint manufacturing companies are significant users of aromatic and aliphatic hydrocarbons as solvents in their various processing units. This is evident from literature reports [1–3] which focus on the health hazards of aromatic solvents used by paint and allied industries. The pollution is not confined to the atmosphere; there is also the danger of pollution of the land if the residues from oil paint manufacture are dumped in landfill sites. The 'waste' from oil paint making processes comprises of unused pigments and solvents. In this context the solvent component is referred to as 'spent solvent'. It is noteworthy that the spent solvents contain significant amounts of aromatic species such as toluene, ethyl benzene and xylene isomers, known by the acronym, TEX. The spent solvents also contain other organic species such as aliphatic hydrocarbons. Although aromatic solvents are generally shunned because of their classification as carcinogens [4], they do play an irreplaceable role

in many chemical-based manufacturing processes. This study was therefore aimed at recoveries of aromatic solvents from waste organic mixtures emanating from oil-paint manufacture. In the interest of promoting 'green chemistry' [5,6], ionic liquids (ILs) were selected as extracting agents. ILs is emerged as a new class of solvents for the replacement of volatile organic solvents in different separation as well as industrial processes to reduce both economic and environmental pollution [7]. ILs is used in a wide range of commercial, industrial and incredible applications. Preliminary studies in our laboratories showed that the concentration of aromatic components in spent paint solvents vary between 6 and 21% by volume. Since aromatic solvents are generally costlier to manufacture than aliphatic ones, their recoveries using economically viable methods is very important. In processes where aromatic solvents are utilized as reaction media [8], it would reduce manufacturing costs if the aromatic components could be recovered on a macro scale and reused. Currently, the recovery of aromatic solvents (on an industrial scale) from mixtures containing aliphatic solvents is generally done by distillation or by using extracting solvents such as sulfolane [9,10]. It has been shown [11] that sulfolane is not effective in cases where the concentration of the aromatic components is <20%. Furthermore, distillation is not suitable for separating azeotropic mixtures.

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Although the recovery of organic solvents from spent paint solvents was performed on a laboratory scale, the experimental design for this project incorporated the following considerations:

- The chosen laboratory method [12] for synthesis of the selected IL has potential for adaption to an industrial-scale synthesis of the IL.
- The stability [13] of the IL was such that it was recovered unchanged at the end of the extraction, that is, it is unaffected by exposure to air and moisture.
- The IL meets the requirements for a 'green solvent' [14] so that it is not hazardous to the environment.
- The ILs should be selected on some rational basis [15].
- The ILs shows no loss of extraction ability on repeated reuse [16].

It is noted that there are several reports on the uses of ionic liquids for micro-extraction of BTEX [17,18] but none of them is associated with the extraction of aromatic components from non-aqueous mixtures. There are also several recent reports [19–22] on the extraction of organic liquids using ionic liquids. However they are restricted to binary and ternary systems whereas this project addresses the extraction of organic liquids from multi-component systems. Moreover, this project is an addition to our contributions [12,13,16] on the synthesis of ionic liquids and their application for the extraction of organic liquids from mixtures of organic liquids.

2. Experimental

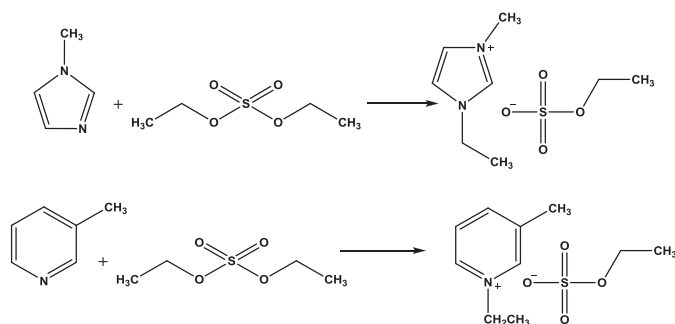
2.1. Materials

Diethylsulphate ($\geq 99\%$), 1-methylimidazole ($\geq 99\%$), and 3-methylpicoline were purchased from Fluka. Toluene ($\geq 99.9\%$), benzene ($\geq 99.9\%$), ethyl benzene, *m*-xylene ($\geq 99\%$), *o*-xylene ($\geq 99\%$), *p*-xylene ($\geq 99\%$) and *n*-hexane ($\geq 99\%$) were purchased from Merck. Karl-Fischer reagent was purchased from Riedel-de-Haen. Samples of spent solvents were obtained from Plascon Paints, one of the major manufacturers of paints in South Africa. The ionic liquids 1-ethyl-3-methylimidazolium ethylsulphate, [EMIM][ESO₄] and 1-ethyl-3-methylpyridinium ethylsulphate, [EMpy][ESO₄] were synthesized in our laboratory using modifications of documented methods.

2.2. Procedures

2.2.1. Synthesis and characterization of ionic liquids

The ionic liquid, 1-ethyl-3-methylimidazolium ethylsulphate, was synthesized using modifications of reported procedures [23,24] while 1-ethyl-3-methylpyridinium ethylsulphate was produced by adapting a documented [25] method. Since the project was aimed at establishing procedures suitable for industrial use, the most amenable method was chosen. For the synthesis of imidazolium and pyridinium-based ionic liquids, quarternization reactions, whereby the ionic liquid cations are formed from the tertiary amines using alkylating agents such as alkyl halides and dialkyl sulphates, proved to be very suitable. Quarternization of 1-ethyl-3-methylimidazolium ethylsulfate and 1-ethyl-3-methylpyridinium ethylsulfate proceeds as follows:



In illustration of the principles described above, the following procedure was used for the synthesis of 1-ethyl-3-methylimidazolium ethyl sulphate:

Diethylsulphate (0.44 mol, 58 mL) was slowly added drop-wise to an ice-cooled mixture of 1-methylimidazole (0.44 mol, 35 mL) and toluene (1.88 mol, 200 mL) under an inert atmosphere of argon. When the addition of diethylsulphate was complete, the reaction mixture was stirred for 5 h at room temperature to allow the reaction to go to completion. At the end of this period, the lower layer (ionic liquid phase) was separated from the mixture by using a separating funnel and then washed three times with a total volume of 100 mL toluene. The crude product was dried at 70 °C under reduced pressure for 3 h using a rotary evaporator; to remove organic solvents and water before the characterization measurements.

2.2.2. Determination of water content in the ionic liquids

The water content of the ionic liquid was determined using a Karl-Fischer Coulometer [Metrohm 756]. Ionic liquids were tested immediately after drying under high vacuum for 48 h and it was found that the water content was below 850 ppm for both the ionic liquids used in this work.

2.2.3. Analysis of spent solvent waste mixtures

Spent paint solvent mixtures were analysed for the concentration by volume of aromatic components present in each. A known volume of each spent solvent mixture was distilled using a rotary evaporator (to collect all volatile organic components including aromatic and aliphatic hydrocarbons as the most important ones). The volume of the distilled liquid was carefully measured and then analysed for the percentage (by volume) of each aromatic component present in the distillate, using GC fitted with capillary column and an FID detector. The concentrations in the raw samples were calculated using the original volume of the raw sample. The results for the above are given in Table 1.

2.2.4. Extraction in stages using freshly-prepared ionic liquids

Aliquots of distillates from spent paint solvents were extracted with freshly-prepared ionic liquids under controlled conditions, using purpose-made water jacketed reaction vessels through which water from a thermostatically controlled tank was circulated. The extraction of aromatic components (toluene, ethyl-benzene and isomers of xylenes, known by the acronym TEX) from the measured samples, using the ionic liquids listed above, was carried out at constant temperature of 40 °C and atmospheric pressure. Aliquots (10 mL) of the spent solvent were placed in the reaction vessels (at 40 °C), followed by addition of ionic liquid (20 mL). The mixture was agitated vigorously using a magnetic stirrer for 5 h in order to allow for intimate contact between the two phases. The equilibrium mixture was then transferred into a separating funnel and allowed to settle for at least 12 h at room temperature to ensure that complete phase separation had occurred. The phases were then separated and stored in suitable sample vials for analysis. The ionic liquid-rich phase is referred to as the extract phase and the alkane-rich phase as the raffinate phase. The ionic liquid was recovered from the mixtures using a rotary evaporator to remove organic solvents

Table 1
Concentrations of aromatic components in spent paint solvent samples.

Aromatic components	Spent solvent mixture	
	Peak area	% (v/v)
Benzene	Nil	Nil
Toluene	18493790	21.0
Ethyl benzene	6168467	7.1
<i>p</i> -Xylene	6652490	7.7
<i>m</i> -Xylene	15830365	18.0
<i>o</i> -Xylene	7260821	8.2
Total aromatics		62.0

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