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Synthesis of a new ionic liquid for efficient liquid/liquid extraction of lead ions from neutral aqueous environment without the use of extractants

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Abstract: This work introduces a new air and moisture stable ionic liquid which is tested for the extraction of Pb2+ from neutral aqueous solution. Here, no chelating agents were used. Grafting coordinating functional groups on the cation of the ionic liquid was not necessary. Very small ionic liquid to aqueous phase ratio was used. The ionic liquid used for this purpose was N-hexyl-4,4-bipyridinium bis(trifluoromethylsulfonyl)imide ([C6byp][Tf2N]). Its synthesis is characterized by spectrometry (1H, 13C, and 19F NMR, ESI-MS, FTIR) as well as Carbon, Hydrogen and Nitrogen (CHN) elemental analysis. Differential scanning calorimetry (DSC) has been used to analyze in detail the thermal behavior in the temperature range of −20 to 200 °C. Interestingly, the ionic liquid demonstrated nearly complete removal of the metal ion from the aqueous phase (98.16%). Furthermore, the reusability (recyclability) investigation demonstrated that the ionic liquid can be used at least for four cycles with undiminished efficiency (98.16% for the second cycle, 97.64% for the third and fourth cycles). Its cycle ability reduces the concern arising from the high cost of ILs. This result indicates that the use of this ionic liquid...
A new IL was synthesized from 4,4ʹ-bipyridil, 1-bromohexane and lithium bis(trifluoromethansulfonyl)imide. 1-Bromohexane converts the symmetric molecular 4,4ʹ-bipyridil to an asymmetric cation by quaternization on one of the nitrogen atoms. The asymmetric and cationic natures provide low melting point and salt characters, respectively. On the other hand, lithium bis(trifluoromethansulfonyl)imide is used to develop a low melting point and hydrophobic properties by replacing the bromide anion from the first salt. The hydrophobic property is very important in creating a biphasic system. Functionalizing neither the cation nor the anion and pH modification was necessary. Moreover, a significantly small IL to aqueous phase ratio was also used. The hydrophobic salt was tested for the extraction of Pb²⁺ from aqueous phase.
2. Experimental

2.1. Materials and methods

1-Bromohexane (Fairfield, OH, USA), 4,4′-Bipyridine (99%), 1,4-dioxane and acetonitrile are (Sigma–Aldrich) are used as received; Lithium bis(trifluormethylsulfonyl)imide (from Alfa Aesar); lead ion standard solution (1000 ppm) (Merck, Germany).

The structures and the purity of the synthesized ionic liquid were checked from $^1$H and $^{13}$C NMR obtained using Bruker AM-270 (270 MHz) spectrometer. $^{19}$F NMR spectra were recorded on a 400 MHz Bruker 400 Ultrashield NMR with operating frequencies of 376 MHz. Infrared (FT-IR) spectrum was recorded using a Perkin Elmer spectrum BX spectrophotometer in 400–4000 cm$^{-1}$ range in KBr pellets. ESI MS was used to determine the molecular ion mass of the cation and anion of the ionic liquid using Bruker Micro TOF. CHN elemental analysis was made by the employment of 5ECHN2200 elemental analyzer taking 10 mg sample.

Inductively coupled plasma optical emission (ICP-OES) spectroscopy (“Perkin Elmer, Optima 8000 ICP-OES spectrometer”) was employed in the investigation of the extraction efficiency. The operating parameters of Perkin Elmer, Optima 8000 ICP-OES spectrometer for the analysis of Pd$^{2+}$ is summarized as in the table below (Table 1).

3. Synthesis

3.1. $[\text{C}_{6}\text{byp}][\text{CF}_{3}\text{SO}_{2}]_{2}N$

The target IL N-hexyl-4,4′-bipyridinium bis(trifluormethylsulfonyl)imide ([$\text{C}_{6}\text{byp}][\text{CF}_{3}\text{SO}_{2}]_{2}N$) was synthesized using simple metathesis reaction from a precursor organic salt N-hexyl-4,4′-bipyridinium bromide ([$\text{C}_{6}\text{byp}]\text{Br}$). The later was prepared based on literature reports (Abebe & Tamiru, 2018). To 5 g (0.0156 mol) [$\text{C}_{6}\text{byp}]\text{Br}$ dissolved in 100 mL distilled water in double-neck round bottomed flask fitted with a condenser being stirred in an oil bath at 65°C, 100 mL aqueous solution of slightly excess molar equivalent of Li([$\text{CF}_{3}\text{SO}_{2}]_{2}N$) (4.50 g, 0.016 mol) was added dropwise. It was stirred for 1 h and oily suspensions were observed. The mixture was left to cool to room temperature and a yellowish oily separate phase was obtained. The oily phase was decanted and decolorized by stirring its methanolic solution in activated charcoal for about 1 h. The methanol was removed by vacuum evaporation and a colorless viscous liquid was obtained (yield: 5.89 g, 85.68%). (Scheme 1).

3.2. Sample preparation for the extraction process

Exposure to low amounts of lead greater than 0.01 mg/L (10 µg/L) results adverse health effects (Bellinger et al. 1987; Hara et al., 2015; Water, 2008; Hussain et al. 2017; Miller-Schulze et al. 2019). Based on this, a 1.903 mg/L (1903 µg/L) Pb$^{2+}$ solution was prepared by diluting a 1000 mg/L stock solution which was prepared by dissolving 1.5980 g of Pb(NO$_3$)$_2$ in 1 L distilled water.

| Parameters                              | Value     |
|-----------------------------------------|-----------|
| Plasma flow                             | 12 L/min  |
| Auxiliary flow                          | 0.5 L/min |
| Nebulizer flow                          | 0.2 L/min |
| RF power                                | 1500 watt |
| Sample flow rate                        | 1 mL/min  |
| Sample replicates                       | 3         |
| Spectra line (analytical wave length)   | 220.353 nm|
3.3. Extraction of Pb$^{2+}$ from the aqueous phase by [C$_6$byp][(CF$_3$SO$_2$)$_2$N]

To 0.05 g [C$_6$byp][(CF$_3$SO$_2$)$_2$N] in 25 mL conical-flask, 10 mL of a 1.903 mg/L lead ion (Pb$^{2+}$) solution was transferred at 298 K and two phases were formed. The mixture was shaken using a shaker at 216 RPM for 1 h at room temperature and then allowed to stand for 1 h to attain equilibrium and phase separation. The shaking speed was selected by starting from 200 RPM and increasing by 2 at which the IL dispersed better into the aqueous phase. Then, all the aqueous phase was taken from the mixture and diluted to 50 mL using distilled water. The concentration of lead ion that was left in the aqueous phase of the first cycle (not extracted by the IL) was determined by ICP-OES. The IL phase was placed in a clean and dry conical flask, to which 10 mL, 69–72% HNO$_3$ was added and the content was heated gently until a few drops remained in the flask and it was repeated three times until the entire organic portion was removed. Then, the residue was dissolved and diluted using distilled water in a 50 mL volumetric flask. For the control solution, exactly 10 mL of 1.903 mg/L of Pb$^{2+}$ solution was taken, diluted to 50 mL using distilled water and analyzed by ICP-OES. Each experiment was repeated three times. The precision of the method was evaluated by calculating the standard deviation (SD) and relative standard deviation (RSD) of the replicate measurements. For all the data obtained, the%RSD values were below 5% which signifies the repeatability of the procedure.

The extraction ability of the [C$_6$byp][(CF$_3$SO$_2$)$_2$N] from the aqueous phase was investigated by measuring the concentration of Pb$^{2+}$ in the aqueous phase before and after the extraction process and in the ILs after the extraction. Extraction efficiencies (%E) were calculated by:

$$\%E = \frac{(C)_aq - (Cf)_aq}{(C)_aq} \times 100$$

3.4. Recyclability of the [C$_6$byp][(CF$_3$SO$_2$)$_2$N]

Ionic liquids are expensive to use compared to the traditional materials. Thus, recycling and reusing them is necessary to reduce costs. The reusability of the ionic liquid was investigated by taking 10 mL of 1.903 mg/L solution of lead metal ion and 0.05 g of [C$_6$byp][(CF$_3$SO$_2$)$_2$N]. The ionic liquid sample was used repeatedly four times stripping Pb$^{2+}$ by 3 mL of 0.1 M HNO$_3$ acid solution from the ionic liquid after each extraction step. The recovered ionic liquid was washed three times using 5 mL distilled water to remove the acid which was checked by measuring the pH of the distilled water used using a universal indicator. The extraction procedure was exactly the same as the previous one and the lead metal ion concentration in the aqueous phase was determined.
3.5. Calibration of the ICP-OES instrument

Standard solutions of lead ion were used to calibrate the ICP-OES and the result is indicated in Table 2. A calibration curve with a good regression coefficient was obtained.

4. RESULTS AND DISCUSSION

4.1. Characterization of \([C_6\text{byp}]\[(\text{CF}_3\text{SO}_2)\text{N}\]

The synthesis of the IL composed of un-functionalized ions and yet employed for the extraction by coordination experiment was confirmed using proton (\(^1\text{H}\)), carbon (\(^{13}\text{C}\)), fluorine (\(^{19}\text{F}\)) NMR, FTIR spectroscopic techniques as well as CHN elemental analysis.

The synthesis of the target IL was achieved by intentional monohexylation of one of the nitrogen atoms of \(4,4^\prime\)-bipyridyl using 1-bromohexane (Scheme 1). The deliberate monohexylation resulted in a monopositively charged coordinating cation. This was achieved by the systematic selection of a solvent (1,4-Dioxane) in which the first alkylation results an insoluble product consequently alkylation of the second nitrogen atom is prevented and will be available for coordination. This phenomena resulted in a product with reduced symmetry which helped for the identification of the product as new peaks appeared in the \(^1\text{H}\) and \(^{13}\text{C}\) NMR spectra of the cation of the product (Figure 1(a,b)). The later argument was strengthened by the characteristic ESI MS\(^+\) spectrum at \(M/z = 241.1703\) for the molecular ion of the cation, \([C_6\text{byp}]^+\) (Figure 2(a)). Furthermore, the asymmetric monopositively charged nature of the cation is a paramount precondition to prepare an ionic liquid with a low melting point. The organic salt with the bromide anion, \([C_6\text{bpy}]\text{Br}\]), was found non-ionic liquid for having a melting point far greater than 100 \(^\circ\text{C}\) which disqualifies it to serve for extraction purpose. Therefore, exchanging the bromide by bis(trifluoromethylsulfonyl)imide anion \(([(\text{CF}_3\text{SO}_2)\text{N}]^-)\)via a metathesis reaction rendered an ionic liquid as a product with a melting point of less than room temperature and is called room temperature ionic liquid (RTIL). The later was confirmed from its DSC thermogram obtained in the temperature range \(-20\) to \(200\) \(^\circ\text{C}\). It is evident that the IL remained in the liquid state for the entire range of study reported here. We do not observe the cold crystallization (Tc), melting temperature (Tm) and glass transition (Tg) in the DSC measurement in this range (Figure 4).

This may be attributed to the free rotation of the cation via C-C single bond connecting the two pyridine molecules and the flexibility of the anion. The combination of these two properties significantly minimizes the vander Waals interactions. Tm, Tc and Tg may be obtained if the scanning start far below \(-20\) \(^\circ\text{C}\) (Chavan & Mandal, 2015).

The anion exchange took place successfully which is evident from the appearance of four new peaks in \(^{13}\text{C}\) NMR at \(\delta\) ppm 112.82–127.00 assignable to the carbon in \([(\text{CF}_3\text{SO}_2)\text{N}]^-\) (Figure 1b). The coupling with the three bonded fluorine atoms to carbon is responsible for the quartet. Moreover, the characteristic ESI MS\(^-\) spectrum for the molecular anion at \(M/e = 279.9165\) for \([(\text{CF}_3\text{SO}_2)\text{N}]^-\) supports that the exchange occurred (Figure 2(b)).

Furthermore, the complete replacement of bromide ion by \([(\text{CF}_3\text{SO}_2)\text{N}]^-\) is substantiated by the appearance of a single signal at \(\delta = -78.75\) ppm in the \(^{19}\text{F}\)NMR spectra which is a characteristic for fluorine (Figure 1©). Similarly, the FTIR spectrum of this IL is shown in Figure 3 and the vibrational

### Table 2. Working standards of the calibration curve for determinations of Pb\(^{2+}\) using ICP-OES

| Standards | Concentration (mg/L) | Intensity | Regression equation |
|-----------|---------------------|-----------|--------------------|
| 1         | 0.05                | 1269.8    |                    |
| 2         | 1.05                | 29,261.3  |                    |
| 3         | 2.05                | 56,616.5  | \(y = 27730x - 1.7\) |
| 4         | 3.05                | 84,787.5  |                    |
| 5         | 4.05                | 112,640.8 | \(R^2 = 0.999991\) |
| 6         | 5.05                | 139,752.2 |                    |
assignments are summarized in Table 3. The weak bands occurring between 2870 and 3132 cm$^{-1}$ correspond to CH stretching characteristic of the cation. The bands at 3132 and 3072 cm$^{-1}$ are due to the bipyridine ring C-H stretching of the cation. The bands at 2950 and 2870 cm$^{-1}$ are assigned to C-H stretching from the hexyl portion of the cation (Talaty et al., 2004). Furthermore, the cation is signaled in terms of very weak overtone bands at 1960 and...
Figure 2. ESI MS+ of a) the cation, b) anion of [C₆bpy] [(CF₃SO₂)₂N].

Figure 3. FTIR spectrum of [C₆bpy][(CF₃SO₂)₂N].
1849 cm\(^{-1}\) obtained by the ring C-H bending in addition to the aromatic C = C and C = N stretching appeared at 1636 and 1464 cm\(^{-1}\), respectively. It is evident that the IR spectrum is strongly dominated by contributions from the anion \([(CF_3SO_2)_2]^−\). This is attributed most probably to its more freedom of motion than the cation. The strong bands appeared at 1050, 1121 as well as 1353 and 1191 cm\(^{-1}\) are assigned to the stretching movements by S-N-S, O = S = O and CF\(_3\), respectively (Reye et al., 1998).

Moreover, the bands at 504 as well as 737 and 605 are assigned to the bending motions of CF\(_3\) and O = S = O, respectively. Finally, the band appeared at 789 cm\(^{-1}\) corresponds to the stretching of CS (Heimer et al., 2006).

Moreover, the purity of the synthesized \([C_6bpy][(CF_3SO_2)_2 N]\) was confirmed from the CHN elemental analysis where a very good agreement between the calculated and experimental values of the elements was obtained. Calculated (experimental) C, 41.46(41.18); H, 4.06(3.98); N, 8.06(7.83).

\(\nu\), stretching; \(\delta\), bending

### Table 3. Frequencies and vibrational assignments of \([C_6bpy][(CF_3SO_2)_2 N]\)

| Assignment         | Frequency, cm\(^{-1}\) |
|--------------------|------------------------|
| 1 \(\nu\) C-H, aromatic | 3132, 3072 |
| 2 \(\nu\) C-H,hexyl   | 2950, 2870 |
| 3 \(\delta\) C-H, aromatic | 1960, 1849, overtone |
| 4 \(\nu\) C = C, aromatic | 1636 |
| 5 \(\nu\) C = N, aromatic | 1464 |
| 6 \(\nu\) SO\(_2\) and \(\delta\) SO\(_2\) | 1353, 1121 and 605, respectively |
| 7 \(\nu\) CF\(_3\) and \(\delta\) CF\(_3\) | 1191 and 737, 504, respectively |
| 8 \(\nu\) SNS        | 1050 |
| 9 \(\nu\) CS         | 789 |

Figure 4. DSC of \([C_6bpy][(CF_3SO_2)_2 N]\).
4.2. Extraction efficiency of \([C_6byp][(CF_3SO_2)_2N]\]

Unlike previous efforts, nearly complete extraction of \(Pb^{2+}\) by \([C_6byp][(CF_3SO_2)_2N]\) was achieved without pH modification, employment of coordinating substances to reduce the hydrophilicity of the metal ion, and modification of the ILs by grafting coordinating appendages on the cation (Table 4) (Domańska & Rękawek, 2009). Moreover, a very small ionic liquid to aqueous phase ratio was used. Interestingly, the extent of the extraction in four cycles is appreciably high and nearly the same. The efficiency of extraction for the successive four steps are 98.16% for the first and second steps and 97.64% for the third and fourth steps. In the later experiment, the moisture and air stability of the IL was also confirmed. This is an opportunity to use this ionic liquid since it minimizes the concerns arising from high cost and elongated time required for synthesis. The \(Pb^{2+}\) ions are transferred to the IL most probably being coordinated to the non-alkylated nitrogen found on the cation of the IL as \([Pb(C_6bpy)_n][(CF_3SO_2)_2N]_n(NO_3)(n=1\text{-}6)\) (Abebe & Tamiru, 2018).

5. Conclusions

\([C_6byp][(CF_3SO_2)_2N]\) is a new ionic liquid intentionally designed for biphasic extraction purpose by coordination through its donor nitrogen. It avoids much more complicated process design and extraction mechanisms. Its synthesis procedure is simple. Its recycle ability is also very simple. It is efficient in liquid/liquid extraction of \(Pb^{2+}\) from a neutral aqueous phase for at least four cycles with undiminished efficiency. Its actions are very straightforward that it requires no chelation procedure to decrease the hydrophilicity of the metal ion. Moreover, functionalization on its cation and anion is not necessary. Thus, it is a good opportunity to employ this ionic liquid for liquid/liquid extraction since it minimizes pressures arising from additional steps in modifying the ionic liquids as well as the use of extractants which incur the additional synthetic cost and elongated time.

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**Table 4. The concentration of \(Pb^{2+}\) before and after the extraction in the aqueous phase for each cycle at 298 K and IL**

| Cycle | \((C_i)^aq\)(mg/L) | \((C_f)^aq\)(mg/L) | \(C_{IL}^{**}\)(mg/L) | RSD % | %E |
|-------|-----------------|-----------------|-----------------|-------|----|
| I     | 0.3806          | 0.007           | 0.372           | 0.1543| 98.16|
| II    | 0.3806          | 0.007           | -               | 0.1804| 98.16|
| III   | 0.3806          | 0.009           | -               | 0.1154| 97.64|
| IV    | 0.3806          | 0.008           | -               | 0.1013| 97.90|

\((C_i)^aq\): concentration of the \(Pb^{2+}\) in the aqueous phase before extraction; \((C_f)^aq\): concentration of the \(Pb^{2+}\) in the aqueous phase after extraction; \(C_{IL}^{**}\): the concentration of \(Pb^{2+}\) was made only in the first round of extraction.

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