

Synthesis of novel ionic liquids and evaluation of their leaching performance in the recovery of copper and zinc from industrial brass slag

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ABSTRACT: Imidazolium-based ionic liquids, 1,3-dibenzylimidazolium trifluoroacetate, 1-benzyl-3-ethylimidazolium trifluoroacetate and 1-benzyl-3-propylimidazolium trifluoroacetate were prepared by simple methods, when compared with those in literature sources, and characterized by IR, NMR and GC-MS and LC-MS. These ILs (Ionic Liquids) were employed as leaching agents in the treatment of industrial copper and zinc bearing slag for the recovery of metals. Results showed that synthesized all novel ionic liquids meet the standard specifications of an ionic liquid. Metal recovery rates decrease with time except for zinc dissolution in 1,3-dibenzylimidazolium trifluoroacetate. 1,3-dibenzylimidazolium trifluoroacetate, when compared with others, is a more effective leaching agent for the treatment of copper and zinc bearing brass slag. Metal recovery rates achieved with this IL are 62.58% for zinc and 24.95% for copper.

KEYWORDS: Copper; Ionic liquid; Leach; Recovery; Synthesis; Zinc

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RESUMEN: *Síntesis de nuevos líquidos iónicos y evaluación de su rendimiento de lixiviación en la recuperación de cobre y zinc de escorias industriales de latón.* Los líquidos iónicos a base de imidazolio, trifluoroacetato de 1,3-dibencilimidazolio, trifluoroacetato de 1-bencil-3-etilimidazolio y trifluoroacetato de 1-bencil-3-propilimidazolio se prepararon mediante métodos sencillos, comparados con los de fuentes bibliográficas. Se caracterizaron mediante IR, NMR, GC-MS y LC-MS. Los líquidos iónicos se emplearon como agentes de lixiviación en el tratamiento de una escoria industrial de cobre y zinc para la recuperación de dichos metales. Los resultados mostraron que todos los nuevos líquidos iónicos sintetizados cumplen con las especificaciones estándar de un líquido iónico. Las tasas de recuperación de metales disminuyen con el tiempo, excepto para la disolución de zinc en trifluoroacetato de 1,3-dibencilimidazolio. El trifluoroacetato de 1,3-dibencilimidazolio, cuando se compara con otros, es un agente de lixiviación más eficaz para el tratamiento de escorias de latón. Las tasas de recuperación de metales logradas con este líquido iónico son 62,58% para el zinc y 24,95% para el cobre.

PALABRAS CLAVE: Cobre; Líquido iónico; Lixiviación; Recuperación; Síntesis; Zinc

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1. INTRODUCTION

Ionic liquids (ILs) have attracted a great deal of attention due to their excellent chemical and physical properties, low melting point around room temperature, good stability in air and in moisture, high solubility of various materials, and negligible vapor pressure (Hajipour and Rafiee, 2009). Room temperature ionic liquids (RTILs) are molten salts in liquid form at room temperature in comparison to high-temperature molten salts (Keskin *et al.*, 2007). Generally, they are composed of anion and cation components and can be designed for a specific purpose by setting particular properties which is why they are called “designer solvents” (Earle and Seddon, 2000). Furthermore, their acidic or basic properties can be adjustable (Ma *et al.*, 2015). In addition to these unique features, they are also known as green solvents (Laus *et al.*, 2005).

The 1,3-dialkylimidazolium cations which have generally been used as green solvents in organic synthesis, organometallic catalysis and enzymatic catalysis are widely employed in ionic liquid synthesis. These cations were first used in electrochemical studies which exhibited wider liquid properties. In addition, the imidazolium ionic liquids display favorable physical properties such as high conductivity, solvation ability and wide range of Lewis acidity. Because of that, they have many different applications such as in separation processes, nano-chemistry and electrochemistry as well as new materials.

Using ionic liquids as leachate in the leaching process of metallurgical ores or wastes is possible and they have great potential as environmentally-friendly media and can be used as alternatives to conventional hydrometallurgical processes. The production of non-ferrous metals from ores has serious drawbacks such as high energy and acid consumption and high environmental pollution and damage. Hence, development of IL processes with less environmental impact and offering high efficiency emerges as a necessity (Tian *et al.*, 2010).

Few studies have been performed on processing ores and wastes with ILs for leaching of metals. Electric arc furnace dust consists mainly of metal oxides which have been treated with choline chloride in the presence of urea and hence ZnO, Cu₂O and PbO were selectively leached out (Abbott *et al.*, 2006; Abbott *et al.*, 2011). BmimHSO₄ has been applied to leaching of brass ash, chalcopryrite, sulphidic gold and silver ore. 99% Zn and 24.82% Cu have been recovered from brass ash in aqueous IL solution. Cu recovery was up to 82% with the addition of oxidant (H₂O₂) (Kilicarşlan *et al.*, 2014). Furthermore, high copper extraction (90%) from chalcopryrite was achieved with BmimHSO₄ after 8 h at 100 °C (Dong *et al.*, 2009).

The aim of this paper is to synthesize novel ionic liquids with trifluoroacetate anions and to

examine their leaching behaviors. The first stage of the present work describes preparation of environmentally-friendly functionalized imidazolium-based ionic liquids by using simple methods. For this purpose, three different ionic liquids namely, 1,3-dibenzylimidazolium trifluoroacetate, 1-benzyl-3-ethylimidazolium trifluoroacetate and 1-benzyl-3-propylimidazolium trifluoroacetate were synthesized and characterized in detail for the first time. In the second stage, they were used as leaching agents for copper and zinc dissolution from brass melting slag and the effects of some important parameters (ionic liquid concentration, temperature and time) on metal dissolution were investigated.

2. EXPERIMENTAL

2.1. Synthesis of ionic liquids

The universal method of preparing ionic liquids is the alkylation of a suitable heteroatom containing organic molecules to form the cation (Chai *et al.*, 2012). After the alkylation, the anion is changed to the one desired. 1-benzylimidazole was chosen as the starting material in a new way. Then, alkylation with ethyl, propyl and benzyl bromides were produced unlike sources in good yields. These compounds combined with sodium trifluoroacetate as the anion, due to its acidic property. Compound 6 was prepared in different ways as in the reference (Kulkarni *et al.*, 2007). Their structures were investigated by IR (Infrared Spectroscopy), NMR (Nuclear Magnetic Resonance), GC-MS (Gas chromatography-Mass spectrometry) and LC-MS (Liquid Chromatography Mass Spectrometry).

Reactions were carried out under argon atmosphere unless otherwise indicated. Reactions were monitored using thin-layer chromatography (TLC). Visualization of the developed chromatogram was performed under UV light or KMnO₄ stain. Resulting residue was purified by silica gel chromatography with a solvent mixture to afford the title compounds. IR spectra were obtained with a Perkin Elmer, FT-IR system and are reported in terms of frequency of absorption (cm⁻¹). NMR spectra were determined with a Bruker Avance III-500 MHz NMR with TMS as the internal standard. Mass spectra were measured on an Agilent 6890N GC-System-5973 MSD and or Agilent LC-MSD Trap SL.

2.1.1. General procedure of 1-benzyl-3-alkyl (or aryl)-1H-imidazol-3-ium bromides (1, 3, 5)

1-Benzylimidazole (1 mmol) was placed in a two neckless flask with acetonitrile (10 mL) under argon atmosphere. The solution was stirred and heated at reflux at 75 °C for half an hour. Then, alkyl (or aryl) bromide (3 mmol) was added drop wise over

a 20 min period by injector. After, it was heated for 5 h and stirred at room temperature for one day. After removal of the solvent, it was dried under vacuum to afford the products 1, 3 and 5. Table 1 shows the preparation and analysis results of the 1-Benzyl-3-ethyl-1H-imidazol-3-ium bromide, 1-Benzyl-3-propyl-1H-imidazol-3-ium bromide and 1,3-Dibenzyl-1H-imidazol-3-ium bromide.

2.1.2. General procedure of 1-benzyl-3-alkyl (or aryl)-1H-imidazol-3-ium 2,2,2-trifluoroacetates (2, 4, 6)

Compound 1, 3 or 5 (1 mmol) and sodium trifluoroacetate (1 mmol) were dissolved with acetonitrile (7 mL), and the resulting mixture was stirred and heated at reflux for 2 h. Then, it was stirred at room temperature for one day. The solid (NaBr) was filtered and the solvent was removed in vacuum to obtain compound 2, 4 or 6. Table 2 shows the synthesis and analysis results of the 1-Benzyl-3-ethyl-1H-imidazol-3-ium 2,2,2-trifluoroacetate, 1-Benzyl-3-propyl-1H-imidazol-3-ium 2,2,2-trifluoroacetate and 1,3-Dibenzyl-1H-imidazol-3-ium 2,2,2-trifluoroacetate.

2.2. Leaching procedure

Industrial copper and zinc based slag were supplied from a brass manufacturer in Turkey and their chemical composition detected with Atomic Absorption Spectrometry (AAS) as given in Table 3.

All leaching experiments were carried out in closed glass beakers with magnetic stirring (360 rpm) at 100 g.L⁻¹ pulp density. Leaching temperature was selected as 40 °C and 60 °C for all experiments. The ionic liquid concentrations (v/v) used in the aqueous leach solutions were 20%, 40% and 60%. Hydrogen peroxide was added as the oxidant in a concentration of 20% in all tests. Solution samples were taken at 30, 60, 90 and 120 min during dissolution and

analyzed by atomic absorption spectrometer to determine the content of copper and zinc.

3. RESULTS AND DISCUSSION

3.1. 1,3-dibenzylimidazolium trifluoroacetate leaching

The relationship between leaching time and copper recovery rate with changing ionic liquid concentration after the leaching process of brass melting slag with 1,3-dibenzylimidazolium trifluoroacetate 40 °C is shown in Fig. 1a. It indicates that the copper leaching rate increased as the ionic liquid concentration increased from 20% to 60%. The highest value of the copper recovery rate was 16.5% after 30 min leaching when 60% ionic liquid concentration was used. It is interesting that the copper dissolution rate decreases with time. This may be due to the precipitation of dissolved copper as copper sulfate in the IL leaching system. On the other hand, when 20% and 40% ionic liquid concentrations were used, the copper recovery rate remained below 5%.

Figure 1b presents the effect of ionic liquid concentration on copper recovery in 1,3-dibenzylimidazolium trifluoroacetate solution at 60 °C. Similar to the recovery rates in a temperature of 40 °C, the copper recovery rate increase depends on the ionic liquid concentration increment. The maximum copper dissolution yield was 24.95% with 60% ionic liquid concentration after a leaching time of 30 min. However, it decreases with increasing leaching time due to the predicted copper sulfate precipitation of 60% and 40% IL concentrations. Furthermore, copper was not able to be dissolved when the IL concentration was 20%.

Comparing Fig. 1 (a, b), the copper extraction increased when the temperature was raised from 40 °C to 60 °C. However, increasing the leaching

TABLE 1. Preparation of 1,3,5 and analysis results

Preparation of 1, 3, 5			
	1, 3, 5		
	1	3	5
	R = ethyl propyl benzyl		
No	1	3	5
Name	<i>1-Benzyl-3-ethyl-1H-imidazol-3-ium bromide</i>	<i>1-Benzyl-3-propyl-1H-imidazol-3-ium bromide</i>	<i>1,3-Dibenzyl-1H-imidazol-3-ium bromide</i>
Condition	Colorless liquid (71 %)	Colorless liquid (82 %)	Colorless solid (97 %)
R _f (20:1 dichloromethane/methanol)	0.18	0.21	0.24
GC-MS (EI, 70 eV)	<i>m/z</i> = 267 (M ⁺), 172, 91	<i>m/z</i> = 281 (M ⁺), 172, 158, 110, 91	<i>m/z</i> = 329 (M ⁺), 171, 91, 80

TABLE 2. Synthesis of 2, 4, 6 and analysis results

Synthesis of 2, 4, 6			
	1, 3, 5		2, 4, 6
	1	3	5
	R = ethyl	propyl	benzyl
No	2	4	6
Name	<i>1-Benzyl-3-ethyl-1H-imidazol-3-ium 2,2,2-trifluoroacetate</i>	<i>1-Benzyl-3-propyl-1H-imidazol-3-ium 2,2,2-trifluoroacetate</i>	<i>1,3-Dibenzyl-1H-imidazol-3-ium 2,2,2-trifluoroacetate</i>
Condition	Yellow liquid (68 %)	Colorless liquid (73 %)	Yellow solid (53 %)
R _f (20:1 dichloromethane/methanol)	0.64	0.22	0.68
GC-MS (EI, 70 eV)	<i>m/z</i> = 300 (M ⁺), 281, 271, 252, 208, 158, 91. LCMS (ESI-QTOF) <i>m/z</i> : calcd for C ₁₄ H ₁₅ F ₃ N ₂ O ₂ 300.1086; Found 301.1162 [M+H] ⁺	<i>m/z</i> = 314 (M ⁺), 281, 271, 252, 208, 171, 91. LCMS (ESI-QTOF) <i>m/z</i> : calcd for C ₁₅ H ₁₇ F ₃ N ₂ O ₂ 314.1242; Found 315.1322 [M+H] ⁺	<i>m/z</i> = 362 (M ⁺), 362, 271, 252, 171 158, 91. LCMS (ESI-QTOF) <i>m/z</i> : calcd for C ₁₉ H ₁₇ F ₃ N ₂ O ₂ 362.1242; Found 363.1353 [M+H] ⁺ .
FTIR (ATR)	ν = 3430, 3131, 3069, 2984, 1678, 1562, 1495, 1455, 1422, 1156, 715, 694 cm ⁻¹	ν = 3416, 3131, 3064, 2970, 2935, 1679, 1562, 1498, 1455, 1157, 709, 662 cm ⁻¹	ν = 3175, 3153, 3113 ve 3073, 2984, 1790, 1608, 1592, 1498, 1444, 1163, 703, 691 cm ⁻¹
¹ H NMR (500 MHz, CD ₃ COCD ₃)	δ = 1.56 (t, J=7.25 Hz, 3H, CH ₃), 4.41-4.45 (q, J=7.25 Hz, 2H, CH ₂), 5.70 (s, 2H, CH ₂), 7.39-7.42 (m, 3H, H _{ar}), 7.65 (d, J= 6.94 Hz, 2H, H _{ar}), 7.88 (s, 1H, =CH), 7.90 (s, 1H, =CH), 10.24 (s, 1H, CH) ppm	δ = 0.92 (t, J=7.25Hz, 3H, CH ₃), 2.06-2.08 (m, 1H, CH ₂), 3.27-3.29 (m, 1H, CH ₂), 4.36 (t, J=7.25 Hz, 2H, CH ₂), 5.72 (s, 2H, CH ₂), 7.38-7.43 (m, 3H, Har), 7.65 (dd, J= 1.57, 7.25 Hz, 2H, Har), 7.89 (s, 1H, =CH), 7.94 (s, 1H, =CH), 10.25 (s, 1H, CH) ppm	δ = 5.67 (s, 4H, CH ₂), 7.41-7.45 (m, 6H, Har), 7.58-7.60 (m, 4H, Har), 7.87 (s, 1H, =CH), 7.88 (s, 1H, =CH), 9.97 (s, 1H, CH) ppm
¹³ C NMR (125 MHz, CD ₃ COCD ₃)	δ = 10.7 (CH ₃), 50.9 (CH ₂), 53.1 (CH ₂), 122.7 (CAr), 128.6 (CAr), 129.8 (CAr), 129.9 (CH), 130.1 (CH), 133.2 (CH) 135.4 (Cq), 205.9 (C=O) ppm	δ = 10.8 (CH ₃), 51.8 (CH ₂), 53.2 (CH ₂), 123.3 (CAr), 123.6 (CAr), 128.4 (CAr), 129.2 (CAr), 129.5 (CAr), 129.7 (CH), 129.7 (CH), 129.9 (CH), 135.9 (Cq), 206.4 (C=O) ppm	δ = 53.6 (CH ₂), 123.7 (CAr), 127.4 (CAr), 127.5 (CAr), 128.9 (CAr), 129.6 (2xCAr), 129.9 (2xCAr), 130.0 (2xCH), 135.3 (Cq), 206.0 (C=O) ppm

TABLE 3. Chemical composition of the slag sample (wt. %)

Zn	Cu	Ca	Al	Mg	Na	Ca	S	Si
53.92	22.36	5.81	1.84	0.10	0.30	0.95	0.32	3.51

time mainly decreased the copper recovery rate. Leaching experiments with concentration of 20% IL gave poor results. The effect of ionic liquid concentration on the leaching efficiencies of zinc when 1,3-dibenzylimidazolium trifluoroacetate was used as leachant is given in Fig. 2 (a, b) at 40 °C and 60 °C, respectively.

It is clear from the figures that increasing the ionic liquid concentration has a positive effect on zinc

dissolution. This situation is true for both leaching temperatures but a difference between concentrations is more clearly seen at 60 °C. After 120 min, the zinc recovery rate was raised from 11.78% to 32.56% as the ionic liquid concentration increased from 20% to 60% at 40 °C (Fig. 2a). The highest value of the zinc recovery rate reached 62.58% using 60% ionic liquid concentration at the end of 2 h leaching at 60 °C (Fig. 2b).

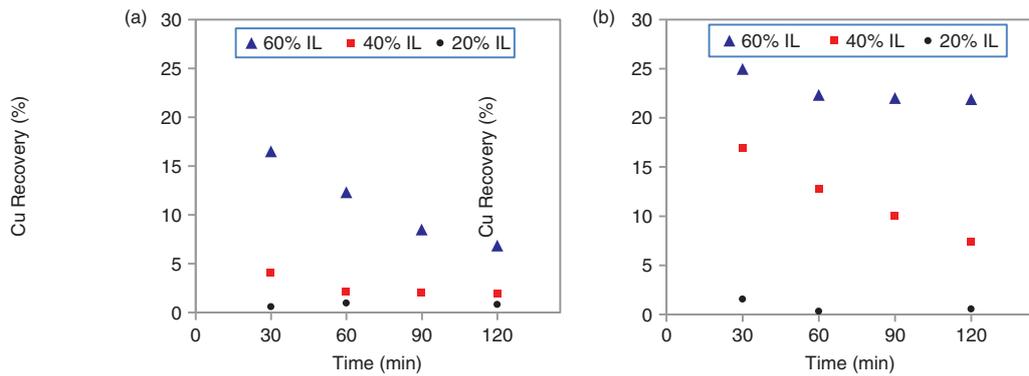


FIGURE 1. The effect of ionic liquid concentration on copper recovery rate in 1,3-dibenzylimidazolium trifluoroacetate solution at a) 40 °C and b) 60 °C.

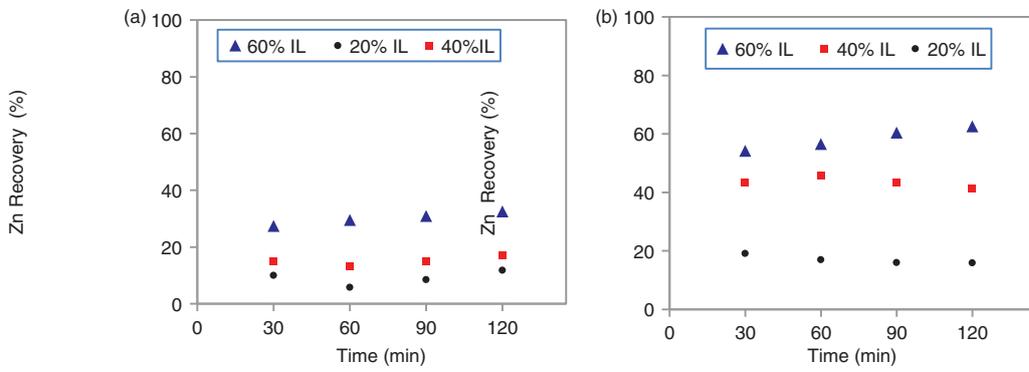


FIGURE 2. The effect of ionic liquid concentration on zinc recovery rate in 1,3-dibenzylimidazolium trifluoroacetate solution at a) 40 °C and b) 60 °C.

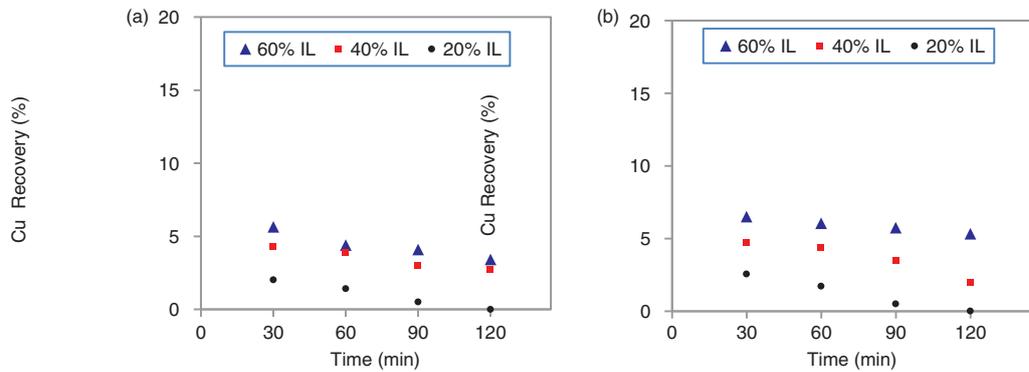


FIGURE 3. The effect of ionic liquid concentration on copper recovery rate in 1-benzyl-3-ethylimidazolium trifluoroacetate solution at a) 40 °C and b) 60 °C.

3.2. 1-benzyl-3-Ethylimidazolium trifluoroacetate leaching

As seen in Fig. 3, the IL concentration has a positive effect on copper dissolution for both leaching temperatures of 40 °C and 60 °C. Nevertheless, the dissolution efficiencies obtained were not satisfactory since the highest values remained below

5.65% for 40% IL concentration and 6.52% for 60% IL concentration after 30 minutes at 40 °C and 60 °C, respectively. Time also has a negative effect on the copper recovery rate for any concentration or temperature.

Changes to the zinc recovery rate with ionic liquid concentration, temperature and time for 1-benzyl-3-ethylimidazolium trifluoroacetate leaching are given

in Fig. 4. The zinc dissolution yield increased when ionic liquid concentration rise from 20% to 60% for both temperatures. But there is no significant impact of ionic liquid concentration on the zinc dissolution despite the zinc recovery rate increasing with concentration. The highest zinc recovery rate reached was 7.74% with 60% IL concentration after 60 min at 60 °C. Time increases adversely affected the leaching efficiency of zinc, as with copper.

It is possible to say that the zinc and copper recovery rate increased with leaching temperature and IL concentration but the achieved recovery rates are very low.

3.3. 1-benzyl-3-Propylimidazolium trifluoroacetate leaching

The effect of 1-benzyl-3-propylimidazolium trifluoroacetate concentration on copper and zinc recovery rate at 60 °C is given in Fig. 5 (a, b), respectively. As can be seen from Fig. 5a the copper recovery rate increased as the IL concentration rise from 20% to 60%. The difference between the

copper recovery rates is more noticeable at early leaching times. Copper dissolution decreased with increased leaching time. While the highest copper recovery rate was 9.61% in the IL solution of 60%, after 30 min, it was 3.10% under the same conditions, after 120 min.

It can be seen from Fig. 5b that the zinc recovery rate increased from 4.67% to 10.4% after 30 min when the ionic liquid concentration increased from 20% to 60%. It is evident from Fig. 5b that the zinc recovery rate increased with increasing ionic liquid concentration at 60 °C. However, increased time decreased the amount of zinc entering the solution. The obtained recovery rates of copper and zinc are at low levels as with the 1-benzyl-3-ethylimidazolium trifluoroacetate solution.

4. CONCLUSIONS

- New synthesized novel ionic liquids: 1,3-dibenzylimidazolium trifluoroacetate, 1-benzyl-3-ethylimidazolium trifluoroacetate and 1-benzyl-3-propylimidazolium

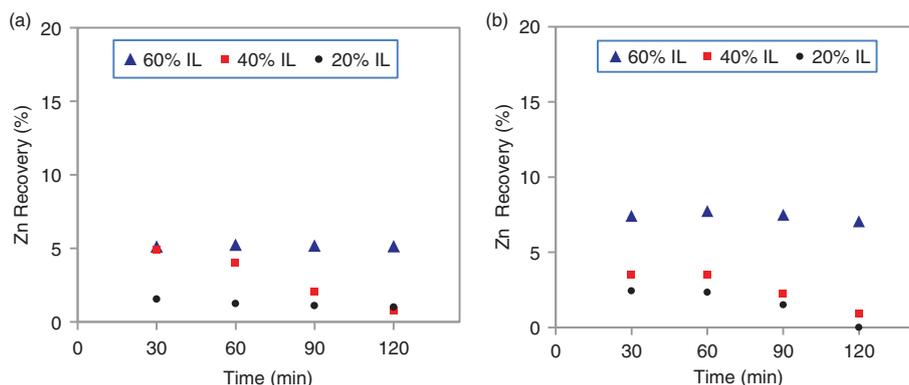


FIGURE 4. The effect of ionic liquid concentration on zinc recovery rate in 1-benzyl-3-ethyl imidazolium trifluoroacetate solution at a) 40 °C and b) 60 °C.

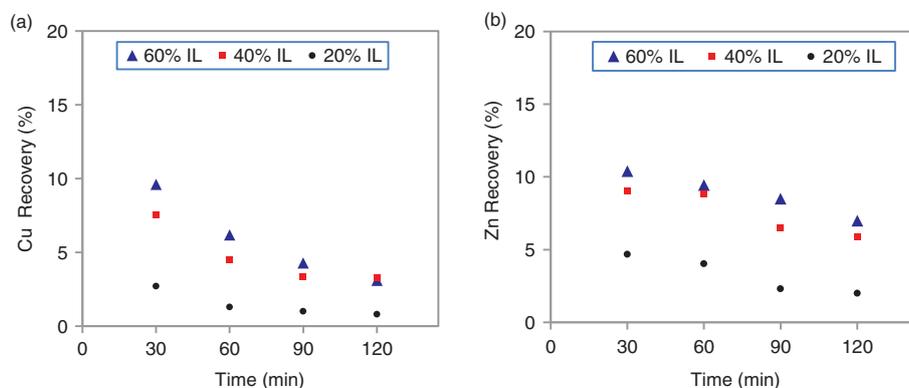


FIGURE 5. The effect of ionic liquid concentration on metal recovery rate in 1-benzyl-3-propylimidazolium trifluoroacetate at 60 °C: a) Cu and b) Zn.

- trifluoroacetate meet the standard specifications of an ionic liquid.
- IL concentration and temperature affect the metal recovery rates positively in all used IL leaching media, but metal recovery rates decrease with time except for zinc dissolution in 1,3-dibenzylimidazolium trifluoroacetate.
- 1,3-dibenzylimidazolium trifluoroacetate, when compared with others, is a more effective leaching agent for the treatment of copper and zinc bearing brass slag.
- Metal recovery which was achieved with 1,3-dibenzylimidazolium trifluoroacetate is promising with 62.58% zinc and 24.95% copper recovery rates. 1-benzyl-3-ethylimidazolium trifluoroacetate and 1-benzyl-3-propylimidazolium trifluoroacetate are quite inefficient as leaching media for both copper and zinc dissolution.

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