



Leaching performance of imidazolium based ionic liquids in the presence of hydrogen peroxide for recovery of metals from brass waste

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ABSTRACT: The application of ionic liquids (ILs), 1-methylimidazolium hydrogen sulfate (HmimHSO₄), 1-ethyl-3-methylimidazolium hydrogen sulfate (EmimHSO₄) and 1-butyl-3-methylimidazolium chloride (BmimCl) as leaching agents was investigated in the leaching of copper and zinc from brass waste in the presence of an oxidant, hydrogen peroxide (H₂O₂). Factors that affect copper and zinc dissolution rates such as ionic liquid concentration, time and temperature were investigated. The results indicated that zinc was dissolved in leach solutions with EmimHSO₄ and HmimHSO₄, completely. Temperature had no considerable influence on copper dissolution rate whilst the rate increased with decreasing IL concentration. In the EmimHSO₄ system, higher copper recoveries were achieved with 40% and 60% IL concentrations compared with IL concentrations of 20% and 80% at 40 °C leaching temperature. Copper dissolution rates decreased with EmimHSO₄ concentration at 60 °C and 80 °C in the following order; 40%>20%>60%>80%. On the other hand the leaching system with BmimCl generally resulted in poor extractions of copper and zinc.

KEYWORDS: Hydrogen peroxide; Ionic liquid; Leach; Metal; Recovery; Waste

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RESUMEN: *Rendimiento de la lixiviación con líquidos iónicos basados en imidazolio en presencia de peróxido de hidrógeno para la recuperación de metales a partir de residuos de latón.* Este trabajo investiga el uso de líquidos iónicos (LIs), hidrogenosulfato de 1-metilimidazolio (HmimHSO₄), hidrogenosulfato de 1-etil-3-metilimidazolio (EmimHSO₄) y cloruro de 1-butil-1-metilimidazolio (BmimCl), como agentes de lixiviación de cobre y zinc a partir de residuos de latón en presencia de un oxidante, peróxido de hidrógeno (H₂O₂). Se estudiaron distintos factores que afectan a la velocidad de disolución del cobre y el zinc, como la concentración del líquido iónico, el tiempo y la temperatura. Los resultados indican que el zinc se disuelve completamente en las disoluciones que contienen EmimHSO₄ y HmimHSO₄. La temperatura no tiene un efecto significativo en la velocidad de disolución del cobre, mientras que dicha velocidad aumenta al disminuir la concentración del líquido iónico. En los sistemas que contienen EmimHSO₄, los mejores resultados de recuperación del cobre se obtuvieron con concentraciones del 20% y el 80% de LI, a una temperatura de lixiviación de 40 °C. Sin embargo, la velocidad de disolución del cobre disminuye con la concentración de EmimHSO₄ en los sistemas a 60 °C y 80 °C en el siguiente orden: 40%>20%>60%>80%. Por otro lado, el sistema de lixiviación con BmimCl generalmente dio como resultado extracciones pobres tanto de cobre como de zinc.

PALABRAS CLAVE: Líquido iónico; Lixiviación; Metal; Peróxido de hidrógeno; Recuperación; Residuos

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1. INTRODUCTION

Wastes which occur during brass production are considered to be significant copper and zinc resources because of their high copper and zinc content (Nesbitt and Xue, 1995; Welter and Revet, 2001; Kahvecioglu *et al.*, 2003). Some previous researchers (Abdel Basir and Rabah, 1999; Timur *et al.*, 2000; Ahmed *et al.*, 2012; Kilicarslan and Saridede, 2015a) have proposed hydrometallurgical recycling processes for the recovery of metals from brass waste using the conventional acid leaching process.

Recently, ionic liquids (ILs) have been focused on as alternative solvents for the sustainable dissolution, extraction and separation of metal ions (McCluskey *et al.*, 2002; Dandan and Kyung, 2010). ILs are organic salts composed of two components, anions and cations and they generally have low melting points ($<100\text{ }^{\circ}\text{C}$). It is possible to design an IL with the desired specifications to meet the requirement of a specific process (Earle and Seddon, 2000). Their unique properties such as low vapor pressure, non-flammability, wide liquid range and thermal stability to solubilize a wide range of solutes including metal salts and complexes make them attractive for hydrometallurgical applications. Especially, their ability to dissolve a variety of components at low temperatures makes the ILs promising and green alternatives to the traditional aqueous acid/alkaline solutions used in hydrometallurgical processes (Tian *et al.*, 2010).

Up to now, ILs have been studied as solvents in various leaching systems for the dissolution of transition metals, gold and silver from their concentrates. McCluskey *et al.*, (2002) studied the IL 1-butyl-3-methyl-imidazolium tetrafluoroborate (BmimBF_4) for leaching of chalcopryrite and found that after 8 hours, 90% of copper extraction was achieved at $100\text{ }^{\circ}\text{C}$. Whitehead *et al.*, (2004) studied the extraction of gold and silver from ores by 1-butyl-3-methyl-imidazolium hydrogen sulfate (BmimHSO_4) in the presence of iron (III) sulfate as an oxidant and thiourea. 87% of gold, and $\geq 60\%$ of silver recovery were achieved with the relatively selective dissolution with regard to the other metals (Cu, Zn, Pb and Fe) present in the concentrate. Dong *et al.*, (2009) investigated the leaching of chalcopryrite using BmimHSO_4 as leaching solution and reported that the leaching efficiency of copper increased from 51.8% to 87.8% when the IL concentration in the leaching solution increased from 10% (v/v) to 100%. Apart from these, dissolution of copper from waste printed circuit boards using the same IL (BmimHSO_4) has been studied by Huang *et al.*, (2014) and they reached almost 100% of copper recovery in 80% (v/v) of the IL concentration with the addition of 10 mL of hydrogen peroxide.

In this study, the leaching of brass waste for the recovery of copper and zinc was investigated by using

various ionic liquids, 1-methylimidazolium hydrogen sulfate (HmimHSO_4), 1-ethyl-3-methylimidazolium hydrogen sulfate (EmimHSO_4) and 1-butyl-3-methylimidazolium chloride (BmimCl). The leaching characteristics and the leaching rate of the metals were examined as a function of time. In addition, temperature and IL concentration, as affecting factors, were investigated in the leaching processes.

2. MATERIALS AND METHODS

2.1. Raw materials

Brass waste used in the present study was obtained from Ozer Metal Company (Tekirdag, Turkey). In the plant, brass melting slag is treated to separate coarse (metallic values) and fine fractions as shown in Fig. 1. Coarse fraction is recharged to the melting furnace while the remaining part of the slag (brass ash) is stored for retreatment via a pyro/hydrometallurgical process. Total sample (2 kg) taken from the company was split into representative subsamples using a rotary cone sample divider for characterization and dissolution tests. ICP-OES method was used to determine the chemical analyses of the brass waste sample and the results are presented in Table 1. Moreover, mineralogical compounds were determined by X-ray diffraction analysis (XRD) and the results are given in Fig. 2. The results of the chemical and mineralogical analyses showed that the waste is rich in metallic copper, copper oxide, and zinc oxide. Also it contains calcium and silicium compounds.

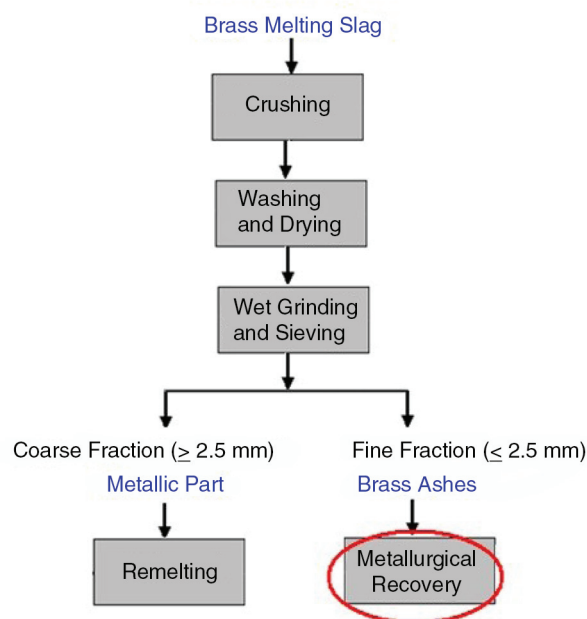


FIGURE 1. Schematic presentation of brass melting slag treatment procedure.

TABLE 1. Chemical composition of brass waste 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

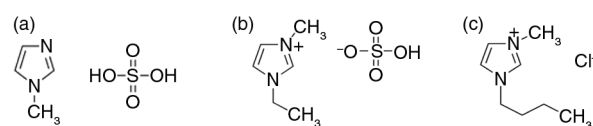
2.2. Ionic liquids

In order to perform the leaching tests of brass waste in imidazolium based ionic liquids, 1-methylimidazolium hydrogen sulfate (HmimHSO₄), 1-ethyl-3-methylimidazolium hydrogen sulfate (EmimHSO₄) and 1-butyl-3-methylimidazolium chloride (BmimCl) were used because of their water soluble character which is crucial for the leaching process. The structures of the ionic liquids purchased from Sigma Aldrich (Germany) are shown in Fig. 3.

Both ILs, HmimHSO₄ and EmimHSO₄ contain hydrogen sulfate anions but BmimCl contains a chloride anion. They all have acidic characters. EmimHSO₄ is in liquid form but HmimHSO₄ and BmimCl present as solid at room temperature and their melting points are 39 °C and 70 °C, respectively

2.3. Dissolution tests

A closed glass reactor with 250 mL capacity was used in the leaching tests. It was placed in a water bath on a magnetic stirrer. Ionic liquids 1-methylimidazolium hydrogen sulfate (HmimHSO₄), 1-ethyl-3-methylimidazolium hydrogen sulfate (EmimHSO₄) and 1-butyl-3-methylimidazolium chloride (BmimCl) and their aqueous solutions were used as leaching solutions in 200 mL volume. Distilled water was used to prepare IL aqueous solutions. Hydrogen peroxide (H₂O₂) was added as the oxidizing agent at constant volume 20% (v/v) into all leaching solutions to enhance particularly copper dissolution (Kilicarslan *et al.*, 2014). When the solution reached

FIGURE 3. Chemical structure of: (a) HmimHSO₄, (b) EmimHSO₄ and (c) BmimCl.

the desired temperature, 20 g of brass waste was poured into the reactor. This amount of waste in 200 mL solution provides 1/100 solid liquid ratio. Sample solutions were taken during the leaching test periodically and then solutions were separated from the residues by filtration to obtain clean solutions for the analysis of metals (Cu and Zn). Metal contents of solutions were determined using atomic absorption spectrometer (AAS). The effects of temperature and IL concentration on copper and zinc dissolutions were investigated.

3. RESULTS AND DISCUSSION

3.1. HmimHSO₄ leaching

3.1.1. Zinc Dissolution

The effect of the HmimHSO₄ ionic liquid concentration on zinc dissolution for temperatures of 40, 60 and 80 °C is shown in Fig. 4. In 20 minutes, all zinc in the waste was dissolved, due to higher solubility of ZnO in the ionic liquid solution for 20, 40, 60, and 80% concentrations of HmimHSO₄ at 40 °C. It is evident that the ionic liquid concentration did not influence the zinc recovery rate significantly at moderate temperatures. At 60 °C and 80 °C, the amount of dissolved zinc in the HmimHSO₄ solution was negligible when the mixture of 80% HmimHSO₄ (v/v) and 20% H₂O₂ (v/v) was used as leach solution. The reason for this limited zinc solubility is no additional water used. It can be said that

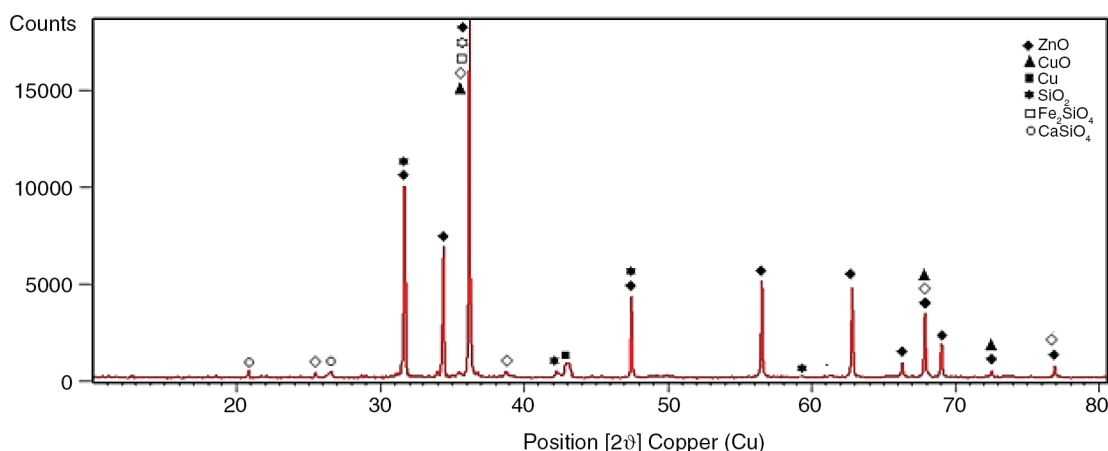


FIGURE 2. XRD pattern of waste sample.

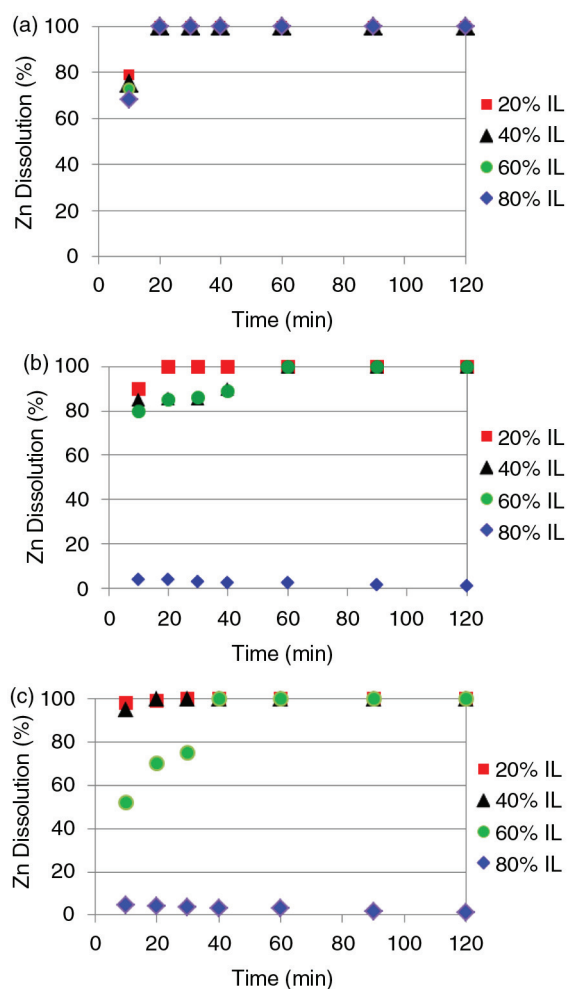


FIGURE 4. Effect of temperature and HmimHSO₄ concentration on zinc dissolution: (a) 40 °C, (b) 60 °C and (c) 80 °C.

the extraction of zinc in aqueous HmimHSO₄ system is significantly higher than in the non-aqueous system with higher HmimHSO₄ concentrations.

All the zinc was dissolved in concentration of 20% IL (v/v) after 20 minutes leaching time. However, 60 minutes were needed for 40% and 60% IL (v/v) concentration at 60 °C and 20 minutes for 40% and 40 minutes for 60% IL (v/v) concentration at 80 °C to dissolve all zinc in the waste. Zinc dissolution rate increased significantly from 2.20% to 100% when the ionic liquid concentration decreased from 80% to any other concentration after 60 minutes at 60 °C. Likewise, it increased remarkably from 3.20% to 100% with the same IL concentrations at 80 °C after 40 minutes.

3.1.2. Copper Dissolution

Figure 5a shows the effect of HmimHSO₄ ionic liquid concentration on copper recovery rates at 40 °C as a function of leaching time. The copper

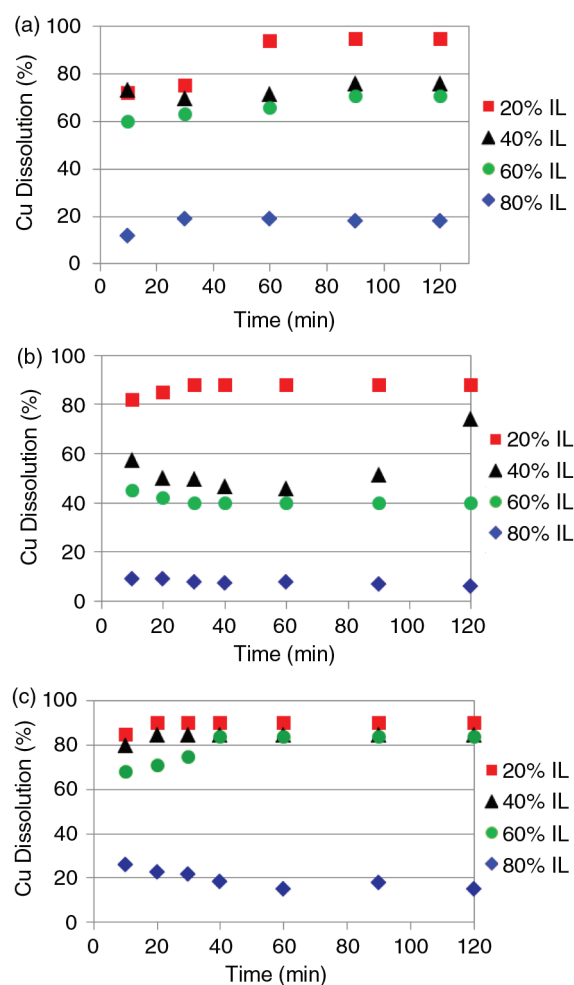


FIGURE 5. Effect of temperature and HmimHSO₄ concentration on copper dissolution: (a) 40 °C, (b) 60 °C and (c) 80 °C.

recovery rate increases considerably with decreasing ionic liquid concentration from 80 to 20% (v/v). When ionic liquid concentration was 80% (v/v), copper recovery rate was about 20% at the end of total leaching time. Moreover, a rapid increase was observed with decreasing ionic liquid concentration between 60% and 20% (v/v). Copper dissolution increased from 17.98% to 70.69% with decreasing of the ionic liquid concentration from 80% to 60% (v/v) in 120 minutes. A smaller difference was observed between 60% and 40% ionic liquid concentrations during leaching time. But, importantly, copper dissolution increased from 76.16% to 95% with decreasing ionic liquid concentration from 40% to 20% (v/v).

The effect of HmimHSO₄ concentration on the copper recovery at 60 °C and 80 °C is given in Fig. 5b and c respectively. The extraction of copper at the temperature of 60 °C and 80 °C showed a similar trend as in Fig. 5a, that is the copper recovery rate increased remarkably as the HmimHSO₄

concentration decreased from 80 to 20% for both leaching temperatures. After 60 minutes, increasing the leaching time has no effect on the dissolution rate of zinc and copper for each temperature and each IL concentration.

The ionic liquid, HmimHSO₄ acted like an acid in aqueous solution, and the hydrogen peroxide played an important role as an oxidant in the study. The oxygen coming from the decomposition of hydrogen peroxide reacts with metallic copper to form cupric oxide, which can easily react with ionic liquid to form cupric compounds or dissolved copper.

3.2. EmimHSO₄ leaching

3.2.1. Zinc Dissolution

Dissolution of zinc in the presence of hydrogen peroxide 20% (v/v) was investigated in various concentrations of 1-ethyl-3-methylimidazolium hydrogen sulfate (EmimHSO₄) ionic liquid at temperatures of 40, 60 and 80 °C. Zinc dissolution was

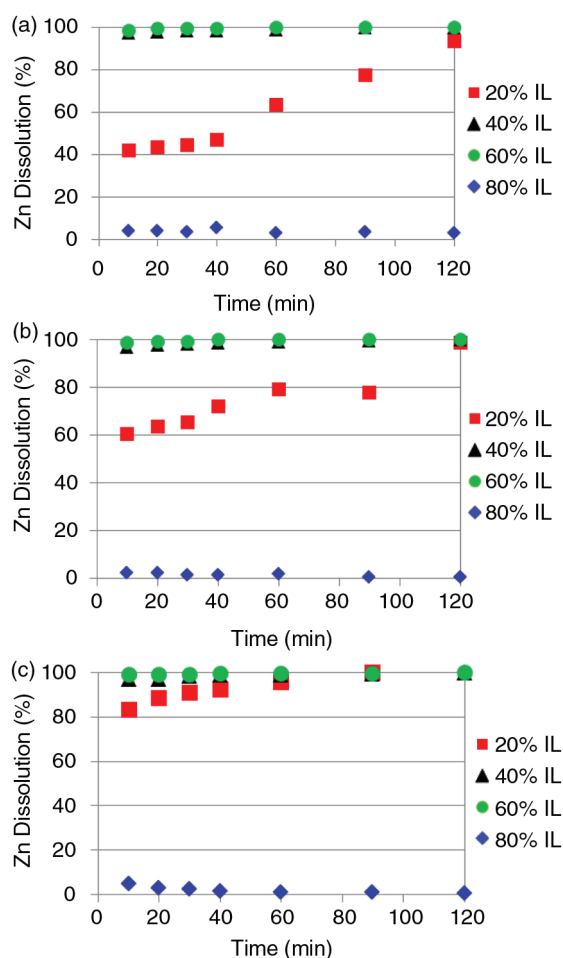


FIGURE 6. Effect of temperature and EmimHSO₄ concentration on zinc dissolution: (a) 40 °C, (b) 60 °C and (c) 80 °C.

completed at all temperatures in 10 minutes when 40 and 60% (v/v) IL concentrations were used (Fig. 6). Dissolution rate values in 20% (v/v) IL concentration increased significantly with increasing temperature in the first 10 minutes giving 41.93%, 60.23% and 83.23% dissolution rates at 40, 60 and 80 °C, respectively. Furthermore, dissolution of zinc in 20% (v/v) IL concentration showed a general increment with increasing leaching time at studied temperatures achieving 100% dissolution at the end.

On the other hand, it is notable that the 80% IL did not give any satisfactory results for zinc dissolution at any temperature or time. Because this solution only consists of 80% IL and 20% oxidant and no water, while solutions of 20, 40 and 60% IL contain calculated amounts of water with oxidant addition. When EmimHSO₄ concentration of 20%, 40%, 60% (v/v) in aqueous solutions with 20% H₂O₂ (oxidant) is present, the hydrogen sulfate anion can dissociate releasing H⁺ ions and hence the EmimHSO₄ plays the role of an acid as reported in a previous study for ionic liquid BmimHSO₄ by Whitehead *et al.*, (2007). In the case of 80% (v/v) EmimHSO₄, the dissociation capability of the hydrogen sulphate anion and ability to act as an acid is reduced because proton hydration cannot occur in the mixture of ionic liquid and oxidant. Consequently, zinc dissolution is higher in 40% and 60% IL concentrations compared with ionic liquid rich (80% IL) and water rich (20% IL) media.

3.2.2. Copper Dissolution

The influence of temperature, EmimHSO₄ concentration and leaching time on copper dissolution in the brass waste is given in Fig. 7. Higher copper recoveries were achieved in 40% and 60% IL (v/v) concentrations admixed with water and oxidant compared with IL concentrations of 20% and 80% (v/v) at 40 °C leaching temperature. Maximum copper recovery was obtained as 48.82% in 40% IL (v/v) at the end of 120 minutes. 20% and 80% IL concentrations gave very limited copper dissolution and no marked changes were observed with increasing time during 120 minutes under the studied conditions (Fig. 7a).

The dissolution of copper at the temperature of 60 °C and 80 °C showed a similar trend (Fig. 5b and c) in that the copper dissolution rates increased with EmimHSO₄ concentration in the following order: 40% > 20% > 60% > 80%. Maximum copper recovery rates were 51.53% and 52.27% for 60 °C and 80 °C leaching temperatures, respectively at 40% IL aqueous solution. Especially, 80% IL (v/v) mixed with 20% H₂O₂ was unsuccessful as leachant compared to the other mixtures in 60 °C and 80 °C though higher dissolution rates were obtained than at 40 °C. This is the result of a lack of additional water in the initial leach solution. Changing of dissolution rates with time was in the very narrow range.

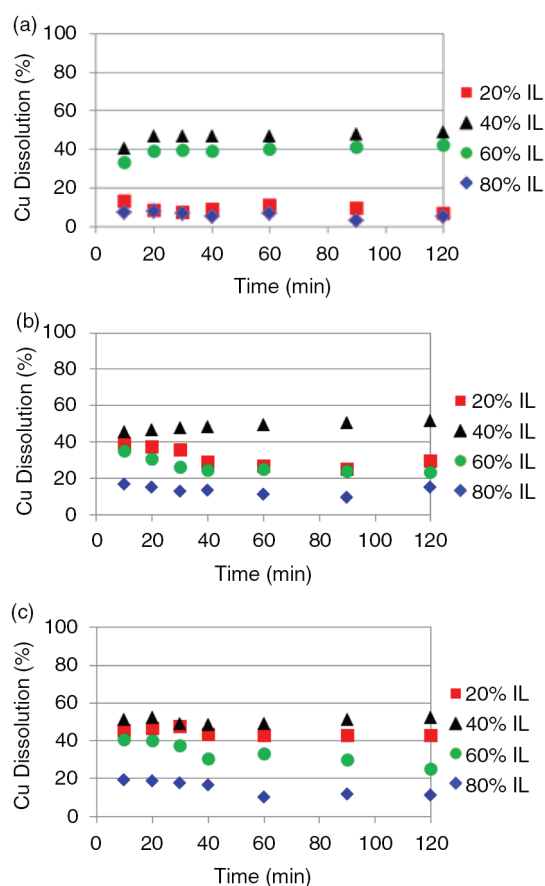


FIGURE 7. Effect of temperature and EmimHSO₄ concentration on copper dissolution: (a) 40 °C, (b) 60 °C and (c) 80 °C.

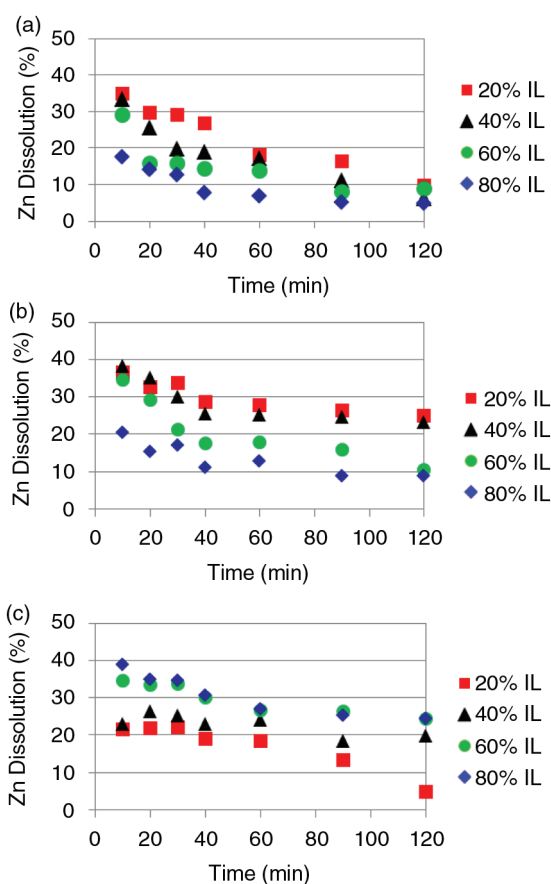


FIGURE 8. Effect of temperature and BmimCl concentration on zinc dissolution: (a) 40 °C, (b) 60 °C and (c) 80 °C.

3.3. BmimCl dissolution

3.3.1. Zinc Dissolution

As shown in Fig. 8, zinc has only partial solubility in various BmimCl concentrations admixed with water and oxidant at 40, 60 and 80 °C leaching temperatures. Increase in the BmimCl concentration resulted in a serious decrease in the dissolution of zinc at both 40 and 60 °C (Fig. 8a and b). However, when the temperature was increased to 80 °C, then higher BmimCl concentrations provided higher zinc dissolution yields (Fig. 8c). While the maximum zinc dissolution rate was 33.38% in 20% IL (v/v) at 40 °C, it was 38.84% at 80% IL (v/v) at 80 °C. Furthermore, a notable decrease in the dissolution rates of zinc with time was observed during the leaching process for all concentrations and temperatures.

3.3.2. Copper Dissolution

Figure 9 reveals that the extraction of copper in the BmimCl system remained under 10%. Negligible copper solubility in this system is due to the

insolubility of copper ion in chloride media in contrast to the sulfate systems. Changing IL concentration in water did not lead to higher dissolution yields of zinc. Increasing of temperature from 20 to 80 °C did not improve the results either. Also, copper dissolution rates decreased with increasing leaching time. At the end of the test time of 120 minutes, there was no difference among the dissolution rates between 40 and 60 °C for any concentration especially in longer leaching times. Hence, use of BmimCl with H₂O₂ as leaching media is not ideal to dissolve copper and zinc. It is clear from a comparison of the HmimHSO₄, EmimHSO₄ and BmimCl leaching systems that, although the hydrogen sulfate systems give high extractions of copper and zinc, it is far less for the chloride system. The systems containing HSO₄ are clearly better than the Cl system and has high potential for recovery of copper and zinc efficiently.

The applications of ionic liquids are available for the electrodeposition of metals from leach liquors after solid-liquid separation (Whitehead *et al.*, 2004; Kilicarslan and Saridede, 2015b). Electrodeposition of metal present in an ionic liquid leach liquor may

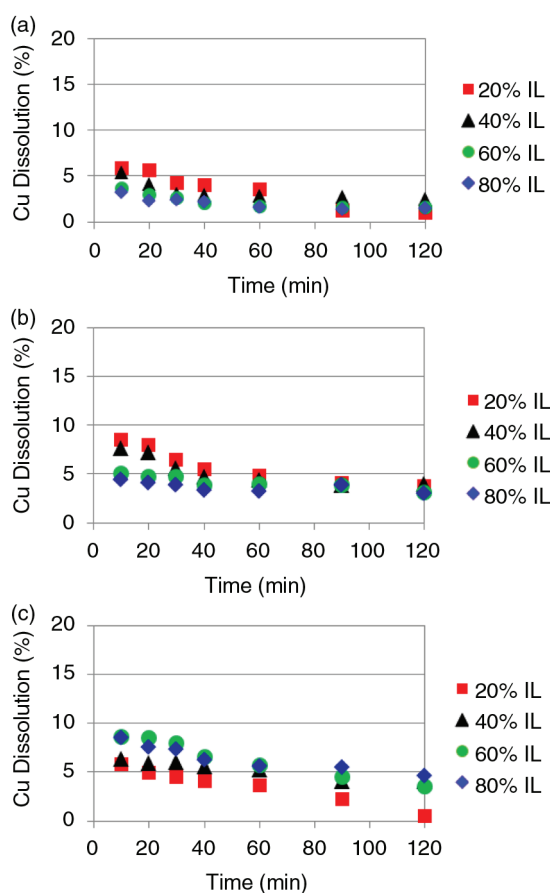


FIGURE 9. Effect of temperature and BmimCl concentration on copper dissolution: (a) 40 °C, (b) 60 °C and (c) 80 °C.

also be considered an advantages method due to the ability of reusing the non-volatile stripped ionic liquid solution for further leach cycles which makes the process cost effectiveness and less harmful to the environment.

4. CONCLUSIONS

In the present work, dissolution of copper and zinc in industrial brass ash was investigated using ionic liquids (ILs); 1-butyl-3-methylimidazolium chloride (BmimCl), 1-ethyl-3-methylimidazolium hydrogen sulfate (EmimHSO₄), and 1-methylimidazolium hydrogen sulfate (HmimHSO₄) as leaching media with hydrogen peroxide as oxidant. 100% Zn recovery was achieved in EmimHSO₄ and HmimHSO₄ leaching systems. Furthermore, HmimHSO₄ was found to be the most efficient ionic liquid for recovery of copper among the studied ionic liquids and the maximum copper dissolution rate is 95% under the following conditions: 20% (v/v) HmimHSO₄, 40 °C and 2 hours. However, leaching systems with BmimCl generally resulted in poor extractions of copper and zinc.

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