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Developing a New Method to Remove Some Toxic Metal Ions from Aqueous Media Using an Ionic Liquid

Agab M. Hewas[1*], Salah R. hewas[1], Khalid M. Omemen[2]
[1]Department of Chemistry, Elemergib University Al-khomes, Libya
[2]The higher institute of science and technology Al-khums, Libya

تطوير طريقة جديدة لإزالة بعض الأيونات المعدنية السامة من الأوساط المائية باستخدام السائل الأيوني

اعقاب محمد حواس[1 *]، صلاح رمضان حواس[1]، خالد محمد اميمن[2]
[1]قسم الكيمياء، جامعة المرقب، الخمس، ليبيا
[2]المعهد العالي للعلوم والتقنية، الخمس، ليبيا

*Corresponding author: amhewas@elmergib.edu.ly

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Abstract:
This research investigates the development of sensitive method for extracting heavy metal ions, which traditionally requires the use of strong inorganic acids. The proposed method aims to be simple, rapid, accurate, and widely accessible. In this study, two types of water-immiscible ionic liquid solutions were employed: betaine [bis(trifluoromethylsulfonyl)imide] and 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF6]). Experimental findings indicate that both ionic liquids demonstrate effectiveness in extracting Ni^{2+}, Co^{2+}, and Pb^{2+} ions. The results reveal that betaine successfully extracted nickel and cobalt ions from their respective solutions but only partially extracted lead ions. Conversely, [BMIM][PF6] was effective in removing nickel and cobalt ions from water but failed to extract lead ions. These findings underscore the varying extraction efficiencies of the two ionic liquids and highlight their potential applications in targeted heavy metal removal.

Keywords: Extraction, Pollution, Immiscible Ionic Liquid.

الملخص
تركز هذه الدراسة على تطوير طريقة جديدة وحساسة لاستخلاص أيونات المعادن الثقيلة، والتي تتطلب تقليديًا استخدام أحماض غير عضوية قوية. تهدف الطريقة المقترحة إلى أن تكون بسيطة وسريعة ودقيقة ومتاحة على نطاق واسع. في هذه الدراسة، تم استخدام نوعين من المحاليل السائلة الأيونية غير القابلة للامتزاج مع الماء: البيتين [bis(trifluoromethylsulfonyl) imide] و-1بوتيل-3-ميثيل إيميدازوليوم هيكسافلوروفوسفات ([BMIM][PF6]). وتشير النتائج التجريبية إلى أن كلا السائلين الأيونيين أثبتا فعاليتهما في استخلاص أيونات Ni^{2+} وCo^{2+} وPb^{2+}. تُظهر النتائج أن البيتين نجح في استخلاص أيونات النيكل والكوبالت من محاليلهما بشكل فعال، لكنه استخلص أيونات الرصاص بشكل جزئي فقط. في المقابل، كان [BMIM][PF6] فعالاً في إزالة أيونات النيكل والكوبالت من الماء ولكنه فشل في إزالة أيونات الرصاص. تسلط هذه النتائج الضوء على كفاءة الاستخلاص المختلفة للسائلين الأيونيين وتبرز إمكانياتهما في التطبيقات المستهدفة لإزالة المعادن الثقيلة.

الكلمات المفتاحية: الاستخلاص، التلوث، السائل الأيوني غير القابل للامتزاج.

Introduction

The presence of toxic metal ions in aqueous media poses significant environmental and health challenges due to their persistence, bioaccumulation, and potential toxicity. Industrial activities, such as mining, electroplating, and chemical manufacturing, contribute to the contamination of water sources with heavy metals such as nickel (Ni^{2+}), cobalt (Co^{2+}), and lead (Pb^{2+}). Conventional methods for removing these contaminants, including precipitation, adsorption, and membrane filtration, often require harsh conditions, specialized equipment, or produce secondary waste, limiting their sustainability and efficiency [1-3]. In recent years, ionic liquids (ILs) have emerged as a promising alternative for environmental remediation due to their unique physicochemical properties, such as negligible vapor pressure, high thermal stability, and tunable solubility. Ionic liquids are composed entirely of ions, which can be tailored to target specific metal ions, enhancing their selectivity and efficiency for extraction processes. Additionally, their water immiscibility and recyclability make them attractive candidates for sustainable environmental applications [4,5].

This study focuses on developing a novel method for the removal of toxic metal ions from aqueous media using ionic liquids. Two types of water-immiscible ionic liquids**,** betaine [bis(trifluoromethylsulfonyl)imide] and 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF6])**,** are investigated for their potential in extracting heavy metal ions. The proposed approach aims to provide a rapid, efficient, and environmentally friendly alternative to conventional methods, addressing key challenges in water purification and metal recovery [6,7]. Base on exploring the extraction efficiency of these ionic liquids, this study contributes to the development of advanced materials and methods for sustainable water treatment technologies [8-10]. The primary objective of this research is to develop a novel, sensitive method for the extraction of heavy metal ions such as Ni^{2+}**,** Co^{2+}**,** and Pb^{2+}. This method is intended to be simple, rapid, accurate, and widely accessible**,** offering an efficient solution for addressing metal contamination in aqueous media. To achieve this goal, the investigation will proceed through the following key stages:

1. Preparation of Ionic Liquids**:**
   Synthesize two types of ionic liquids that are immiscible with water, ensuring their suitability for selective extraction processes.
2. Metal Ion Exposure and Extraction Capability Assessment**:**
   Subject solutions containing the target metal ions to the prepared ionic liquid electrolytes to evaluate their efficiency in extracting each metal ion.
3. Verification of Extraction Processes:
   Confirm the successful extraction of each metal ion by employing colorimetric indicators to visually and analytically validate the outcomes.

This systematic approach aims to establish a reliable and innovative methodology for heavy metal extraction, contributing to advancements in environmental remediation and sustainable water treatment technologies.

Extraction Procedure

The extraction process involves introducing a water-immiscible ionic liquid into a solution containing the target metal ions. This results in the formation of two distinct phases:

1. Upper Layer: The aqueous phase, which contains the remaining metal ions that have not been extracted.
2. Lower Layer: The room-temperature ionic liquid (RTIL) phase, which selectively extracts and binds the targeted metal ions.

These two phases are clearly separated by a well-defined interface, as illustrated in the subsequent Figure 1. This separation facilitates the efficient recovery of metal ions from the aqueous phase into the RTIL phase, enabling a straightforward and effective extraction process.

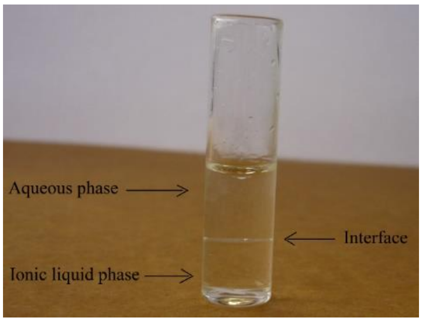

Figure 1. The binary mixture (IL & water) used in the extraction of metal ions.

Method

To perform the extraction:

1. Preparation of the Solution:
   A metal ion solution with a concentration of 200 ppm was prepared.
2. Addition of the Ionic Liquid:
   The ionic liquid was carefully introduced into the solution by tilting the container (e.g., a test tube) and gently adding the liquid along the inner wall of the tube. This ensured minimal disturbance and avoided any mixing of the phases.
3. Phase Separation:
   Upon addition, two distinct phases formed:
   - The upper aqueous phase.
   - The lower ionic liquid phase (RTIL).
4. Indicator Utilization:
   The presence of a colorimetric indicator, such as dimethylglyoxime (DMG), facilitated clear visual distinction between the phases and aided in monitoring the extraction process.

This gentle and straightforward approach ensured the integrity of the extraction process, allowing efficient separation of the ionic liquid from the aqueous solution.

Results and Discussion

Extraction Process Using Betaine

The initial stages of the extraction process demonstrated a clear separation of phases:

- The upper aqueous phase was pink, attributed to the DMG:Ni complex present in the solution.
- The lower ionic liquid phase (betaine) was colorless.

Upon gently shaking the binary mixture for a few seconds, a noticeable transfer of the DMG:Ni complex occurred. The pink color, characteristic of the complex, migrated from the aqueous phase to the ionic liquid phase. This resulted in the aqueous phase turning colorless, while the betaine ionic liquid phase adopted a pink coloration, as depicted in the accompanying figure (a & b).

Interestingly, the pink coloration in the ionic liquid phase began to fade gradually, eventually disappearing entirely within a few minutes (c). This change likely indicates the formation of a new and more stable complex between nickel and betaine. This complex is presumed to supersede the stability of the DMG:Ni complex, thereby explaining the observed disappearance of the pink color.

These findings suggest that betaine ionic liquid exhibits a strong affinity for nickel ions, facilitating efficient extraction through complexation and highlighting its potential as a powerful medium for selective metal ion removal.

Figure 2 illustrates the extraction process of nickel ions using betaine ionic liquid, depicting: (a) the system prior to agitation, (b) the system immediately after shaking, and (c) the system a few seconds following phase settling.

A similar experimental procedure was conducted for cobalt ions, with the addition of a single drop of pyridine to enhance the extraction process. The transition of the yellow-brown coloration, characteristic of cobalt complexes, from the upper aqueous layer to the lower ionic liquid layer was clearly observed. This transition is effectively depicted in Figure 3 (a & b), providing a visual representation of the results.

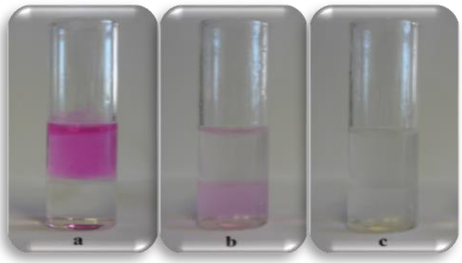

Figure 2. The extraction process of nickel ions using betaine ionic liquid, depicting: (a) the system prior to agitation, (b) the system immediately after shaking, and (c) the system a few seconds following phase settling.

A similar outcome was observed for lead using the rhodizonic acid indicator. Upon gently shaking the binary mixture, the colored complex transitioned from the aqueous phase to the ionic liquid phase. After allowing the system to settle, the separation of phases and the movement of the complex were evident, as depicted in Figure 4.

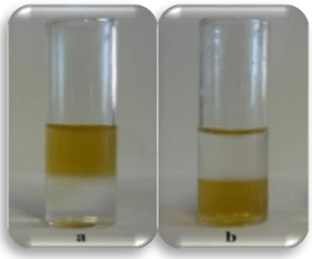

Figure 3. The extraction of cobalt ions by using betaine a) before shaking and b) after shaking.

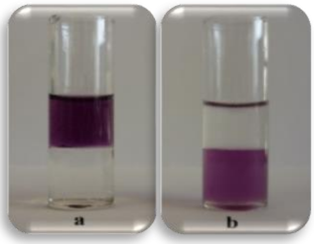

Figure 4. The extraction of lead ions by using betaine a) before shaking and b) after shaking.

In the case of lead, the extraction process using betaine was observed to be incomplete. When a single drop of the reagent was added to the post-extraction aqueous layer, the resulting color remained consistent with the original, albeit with reduced intensity. This observation, as illustrated in Figure 5, indicates that a portion of the lead ions remained unextracted. Overall, it can be concluded that while lead was only partially extracted, betaine demonstrated complete removal efficiency for nickel and cobalt ions under the same experimental conditions.

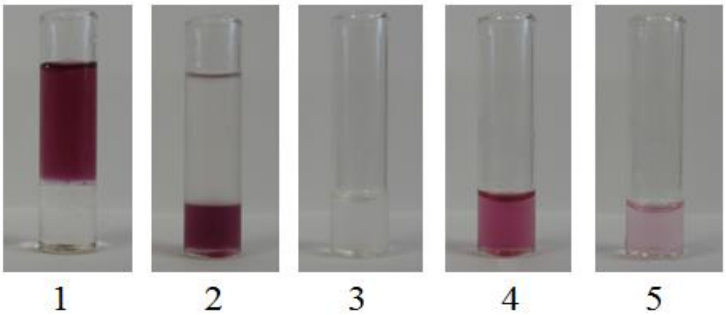

Figure 5. A positive result of lead ions in the extracted aqueous layer by using the rhodizonic acid indicator: 1) the test runs for 200 ppm of lead solution; 2) after one extraction step; 4) after repeating the extraction process in two steps; and 5) after repeating the extraction process in three steps.

Extraction Process Using [BMIM][PF6]

The second phase of the study investigated the extraction capability of another ionic liquid, [BMIM][PF6], for the removal of the same metal ions. This ionic liquid also demonstrated success in extracting nickel ions. However, unlike the previous case with betaine, the colored complex formed during the extraction process exhibited greater stability, maintaining its intensity even after an extended period. This observation is clearly depicted in Figure 6, highlighting the effectiveness and stability of [BMIM][PF6] in nickel ion extraction.

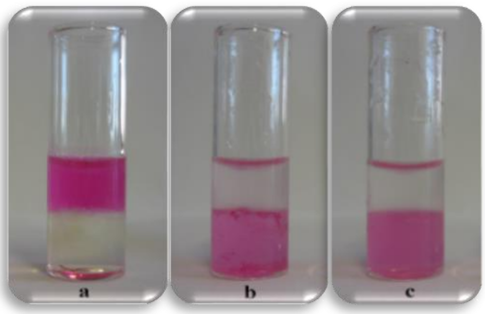

Figure 6. The extraction of nickel ions by using [BMIM][PF6], a) before shaking, b) a few seconds after shaking, and c) a few minutes after settling down.

A positive outcome was also achieved in the extraction of cobalt ions using [BMIM][PF6]. During the process, a yellow oily complex identified as DMG:Co:Py successfully transferred from the upper aqueous phase to the lower ionic liquid phase. This result demonstrates the effectiveness of [BMIM][PF6] in cobalt extraction and is visually represented in the subsequent figure 7.

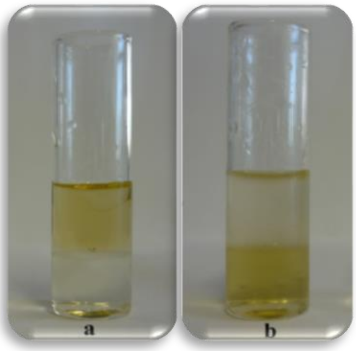

Figure 7. The extraction of cobalt ions by using [BMIM][PF6], a) before shaking and b) after shaking.

However, 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF6]) failed to extract lead ions, as illustrated in the following figure: (a) prior to shaking the tube, and (b) after shaking it. The extraction behavior of these metal ions was systematically examined under identical conditions and at room temperature, highlighting the differential efficiency of [BMIM][PF6] for various metal ions.

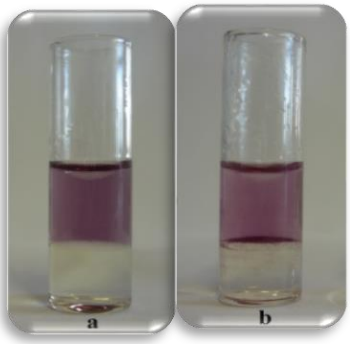

Figure 8. The extraction of lead ions by using [BMIM][PF6], a) before shaking and b) after shaking.

Based on the experimental results presented, it can be concluded that the ionic liquid betaine effectively extracted nickel and cobalt ions from their respective solutions, although it only partially extracted lead ions**.** Similarly, [BMIM][PF6] demonstrated success in removing nickel and cobalt ions from aqueous solutions but failed to extract lead ions under the same experimental conditions. These findings highlight the varying extraction efficiencies of the two ionic liquids for different metal ions.

Conclusion

The extraction process using ionic liquids has been demonstrated to be highly effective, achieving significant extraction rates in a single attempt without requiring repeated procedures. The use of colorimetric indicators facilitated the straightforward tracking of the extraction process by visually observing the transition of the colored complexes between the immiscible phases. This method highlights the potential of ionic liquids as efficient and reliable agents for the removal of metal ions from aqueous solutions.

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