EXTRACTION OF SAMARIUM, EUROPIUM AND GADOLINIUM USING [TRIALKYLAMMONIUM][DI-(2-ETHYLHEXYL) PHOSPHATE]

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Pemisahan unsur-unsur nadir bumi merupakan tugas yang mencabar disebabkan oleh persamaan sifat kimia antara kumpulan lantanid. Kaedah pengekstrakan pelarut konvensional menggunakan asid fosfat di-(2-etilheksil) (DEHPA) memerlukan jumlah peringkat yang besar dan menggunakan jumlah asid dan bes yang banyak untuk pemisahan yang berkesan. Dalam kajian ini, cecair ionik bifungsi [trialkilamonium][di-(2-etilheksil)fosfat], [A336][DEHPA] telah dikaji sebagai agen pengekstrak alternatif bagi pemisahan samarium, europium, dan gadolinium. Kesan jenis asid (asid nitrik, HNO_3; asid hidroklorik, HCl; asid sulfurik, H_2SO_4) dan kepekatan (1.5M-5.0M), serta nisbah fasa organik kepada akuas (O:A) (1:1, 3:2, 7:3, 4:1, 9:1) telah dikaji menggunakan kaedah eksperimen satu faktor pada satu masa (OFAT). Metodologi permukaan respons (RSM) menggunakan rekabentuk sejarah juga digunakan untuk mengoptimumkan dan mengesahkan keputusan eksperimen. Jumlah peringkat pengekstrakan dan pengikisan kemudian dikira berdasarkan Model Teori Aliran Berlawanan dan disahkan secara eksperimen. Analisis teknonomi dilakukan bagi kedua-dua agen pengekstrak dengan menggunakan data yang diterbitkan bagi asid fosfat di-(2-etilheksil) ester mono 2-etilheksil (EHEHPA) sebagai titik rujukan. Keadaan terbaik untuk pemisahan samarium dicapai dengan menggunakan [A336][DEHPA] dengan faktor pemisahan 2.93, berbanding dengan DEHPA yang mempunyai faktor pemisahan 1.93. Keadaan pemisahan samarium yang optimum ialah menggunakan HNO_3 3.0M dengan nisbah O:A 4:1 untuk [A336][DEHPA], dan HCl 2.0M dengan nisbah O:A 7:3 untuk DEHPA. Pemisahan unsur-unsur yang lain, europium dan gadolinium, menunjukkan [A336][DEHPA] mempunyai keupayaan pemisahan yang lebih baik berbanding DEHPA dengan faktor pemisahan masing-masing 3.44 dan 2.38. Keadaan pemisahan optimum untuk europium dan gadolinium ialah HNO_3 3.5M dengan nisbah O:A 4:1 untuk [A336][DEHPA] dan HCl 3.0M dengan nisbah O:A 4:1 untuk DEHPA. Jumlah keseluruhan peringkat pengekstrakan dan pemulaan bagi keseluruhan proses yang dikira dan disahkan bagi setiap agen pengekstrak ialah 30 peringkat untuk [A336][DEHPA] dan 46 peringkat untuk DEHPA. Penanda aras yang dipilih untuk perbandingan teknonomi dalam kajian ini adalah EHEHPA, sejenis pengekstrak yang telah terbukti digunakan dalam aplikasi industri. Analisis tersebut menunjukkan bahawa kos keseluruhan untuk [A336][DEHPA] adalah 46.49% lebih rendah daripada kos penanda aras (EHEHPA), menunjukkan pengurangan kos yang ketara yang dicapai oleh pengekstrak baru tersebut. Selain itu, DEHPA juga menunjukkan pengurangan kos sebanyak 34.15% berbanding dengan penanda aras. Perbezaan ini utamanya disebabkan oleh kos yang lebih tinggi untuk pembangunan pengadun settler bagi EHEHPA dan DEHPA. Kesimpulannya, [A336][DEHPA] telah menunjukkan keupayaan pemisahan yang unggul dengan faktor pemisahan yang lebih tinggi, penggunaan bahan kimia yang lebih rendah, dan kos yang lebih rendah berbanding agen pengekstrak lain. Ia merupakan alternatif yang lebih mesra alam dengan jumlah peringkat yang lebih kecil yang diperlukan untuk pemprosesan unsur-unsur tanah jarang. Oleh itu, [A336][DEHPA] memberikan harapan yang baik bagi pemprosesan mineral industri pada masa hadapan.

ABSTRACT

The separation of the rare earth elements is a difficult task owing to the chemical similarity of the lanthanides. The conventional solvent extraction method using (di-(2-ethylhexyl) phosphoric acid), DEHPA requires a large number of stages and consumes significant amounts of acid and base for effective separation. In this study, a bifunctional ionic liquid [trialkylammonium][di-(2-ethylhexyl)phosphate], [A336][DEHPA] was investigated as an alternative extractant for the separation of samarium, europium and gadolinium was compared to DEHPA. The effect of acid type (acid nitric, HNO_3, hydrochloric acid, HCl, sulfuric acid, H_2SO_4) and concentration (1.5M-5.0M) , as well as organic to aqueous (O:A) phase ratio (1:1,3:2,7:3,4:1,9:1) were studied using one-factor-at-time (OFAT) experimental method. The response surface methodology (RSM) using historical data design (HDD) was further used to optimized and validate the results. The number of extraction and scrubbing stages were then calculated according to Counter Current Theory Model and were validated experimentally. Techno-economic analysis was performed for both extractants, with the published data on 2-ethylhexyl phosphoric acid mono 2-ethylhexyl ester (EHEHPA) serving as the baseline. The best condition for samarium separation was achieved using [A336][DEHPA] with separation factor of 2.93, in comparison with DEHPA with 1.93. The optimum samarium separation condition was using 3.0M HNO_3 with O:A of 4:1 for [A336][DEHPA], and 2.0M HCl with O:A of 7:3 for DEHPA. The separation of remaining elements, europium and gadolinium, showed [A336][DEHPA] have better separation capabilities against DEHPA with separation factors of 3.44 and 2.38, respectively. The optimum condition for separation of europium and gadolinium was 3.5M HNO_3 at O:A of 4:1 for [A336][DEHPA] and 3.0M. HCl at O:A of 4:1 for DEHPA. The total number of extraction and scrubbing stages for the entire process calculated and validated for each extractant was 30 stages for [A336][DEHPA] and 46 stages for DEHPA. The benchmark selected for techno-economic comparison in this study was EHEHPA, a well-established extractant used in industrial applications. The analysis revealed that the overall cost for [A336][DEHPA] was 46.49% lower than the cost of the baseline (EHEHPA), indicating a substantial reduction in expenses achieved by the new extractant. Additionally, DEHPA demonstrated a cost reduction of 34.15% compared to the baseline. This difference was primarily attributed to the higher cost associated with the development of the mixer settler for EHEHPA and DEHPA. In conclusion, [A336][DEHPA] demonstrated superior separation capabilities with higher separation factors, lower reagent consumption, and reduced costs compared to other extractants. It offers a greener alternative with a smaller number of stages required for rare earth processing. Therefore, [A336][DEHPA] holds great promise for industrial mineral processing in the future.

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