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SCRUBBING STUDY ON RARE EARTH LOADED ORGANIC FROM EXTRACTION PROCESS OF YTTRIUM

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ABSTRACT

Separation process of rare earth elements is normally accomplished using solvent extraction that consists of extraction, scrubbing stage and stripping stage. Since the separation of rare earth elements is difficult due to their chemical similarities, improvement of the process need to be done to increase separation factors and to reduce chemical consumption thus will reduce chemical waste. This study focused on scrubbing stage for extraction of yttrium from Malaysian Xenotime. The objective of this study is to find the best scrubbing agent in order to remove inorganic element from loaded organic phase as much as it can. Three different media were chosen as scrubbing agent which is deionized water, nitric acid and ammonium nitrate to scrub calcium, sulfur and chlorine produced from rare earth loaded organic solution. Results from X-Ray Fluorescence (XRF) shows that 70°C deionized water with contact time of 10 minutes have a tendency to scrub most sulfur (15.2 ppm), chlorine (0.56 ppm) and calcium (26.2 ppm)from loaded organic solution.

Keyword: Scrubbing, Rare Earth, Xenotime, Solvent Extraction , XRF.

INTRODUCTION

Rare earth elements (REEs) have growing as a worldwide demand in recent years. After many years of research and development, rare earth elements had proved their advantages in green technologies such as next generation wind turbine and electric vehicles. Rare earths also used in the manufacture of optical sensors (yttrium), ceramic (lanthanum), permanent magnets (neodymium) and catalyst in oil refining (lanthanum). More recently, the term 'rare earths' has been used to designate the element themselves. Scandium and yttrium tend to occur in the same ore deposits as lanthanides and exhibit similar chemical properties (Balaram et al., 2019).

In Malaysia, major source of rare earth in mineral is xenotime (heavy rare earth) and monazite (light rare earth). Xenotime (YPO_4) is classified as radioactive mineral. Malaysian xenotime had been assumed consist of 30% of yttrium and 40% rare earth. Xenotime is fused with sodium hydroxide at 350°C to help phosphate removal from the rare earth more efficient compared than other route such as acid digestion (Roshasnorlyza et al., 2025). A part of the precise leaching process, the leach solution usually contain dissolved impurities such as chlorine, phosphorus and calcium which are removed by precipitation before proceeding to solvent extraction to separate rare earths.

Solvent extraction begins by separating different group of rare earths from leach solution. Individual rare earths are difficult to separate from each other due to their similar physical and chemical properties. Separation processes based on solvent extraction and ion-exchange technique had been developed to produce high purity single rare earth solutions or compounds (Peiqiang Fan et al., 2023) (Youssef et al., 2023). Solvent extraction is used as commonly the most appropriate commercial technology for separating rare earths (Feng Xie et al., 2014) (Abreu et al., 2014) (Hiskeya et al., 2018) (Wu et al., 2018) due to the need to be able to handle larger volumes of leach solution. Also, solvent extraction is being developed as environmental friendly technique since there is no use of high temperature compared than pyrometallurgy studies.

Separation and purification of rare earth using solvent extraction processes have been reviewed. There are four classes of extractant, namely as cation exchangers, solvation extractants, anion exchangers and chelating extractants that have been utilized for separating rare earths . In this study, cation exchanger which is di-ethylhexyl phosphoric acid (DEHPA) is used as extractants for separation of rare earth in kerosene and aqueous chloride solution. DEHPA can extract 100% of thorium at low concentration (Nur Aqilah et al, 2019) but because of the distribution coefficient and separating factors that lies strongly in the organic solution, it is difficult to strip the loaded organic. Therefore, scrubbing technique is proposed to remove inorganic co-extracted element in order to make stripping become more efficient.

Several investigations have been reported in the literature to study the behavior of scrubbing technique towards removal of inorganic element. Jorjani et al., 2012 studied the behavior of loaded organic phase (from loaded Tri-Butyl Phosphoric Acid, TBP) that had been scrubbed with hot deionized water and ammonium nitrate. The results showed that three stages of scrubbing with a phase ratio (Va/Vo) of five removed about 80%, 30%, 27%, and 15% of Ca, Mg, Fe, and P, respectively, from loaded TBP, while less than 9% of total REEs was lost.

The extraction of yttrium from Xenotime is carried out with DEHPA in kerosene. In this study, the form of yttrium and rare earth oxalate is produced after precipitation of leached solution from Alkaline Fusion of Malaysian Xenotime (Meor Yusoff MS et al., 2015). The pregnant liquor or rare earth loaded organic with DEHPA is then scrubbed with deionized water, nitric acid and ammonium nitrate.

MATERIALS AND METHOD

Preparation of pregnant liquor

The sample used in this study was prepared from alkaline fusion process of xenotime obtained from the stockpile of Nuklear Malaysia where sodium hydroxide pellets is used to mix with xenotime and fused in furnace for 350°C. Washing and leaching process is introduced as previous study (Meor Yusoff MS et al., 2015) and mixed rare earth oxalate is obtained. Mixed rare earth oxalate is then dissolved in 3M hydrochloric acid for it has optimum molarity for yttrium's extraction as Figure 1.

The extractants used for two stage extraction process were Diethylhexyl Phosphoric Acid (DEHPA), which was from Sigma-Aldrich. DEHPA is used without further purification. Kerosene, which was used as a diluent, was supplied by LabChem. Deionized water and hydrochloric acid (from RCI Labscan) is used in all the experiments for aqueous solutions. Nitric acid (from RCI Labscan) and ammonium nitrate (from Sigma-Aldrich) were used as scrubbing agent. The organic solutions from this extraction process were called rare earth loaded organic or pregnant liquor and were analyzed by Energy Dispersive X-Ray Fluorescence (EDXRF), Shimadzu EDX-7000 model as Table 1.

Table 1: Concentration of organic element and inorganic element in rare earth loaded organic

Organic	**Y**	**Gd**	**Dy**	**Ho**	**Er**	**Tm**	**Yb**
Concentration (ppm)	52,310	363.9	3,540	1,320	3,350	739.0	6530

Inorganic	**P**	**Ca**	**Na**	**Si**	**S**	**Cl**	**Mg**
Concentration (ppm)	125,190	1970	129	133	2080	7010	41

Scrubbing of rare earth loaded organic

This study were conducted with three different scrubbing agent which is deionized water, nitric acid (from RCI Labscan) and ammonium nitrate from Sigma-Aldrich to remove undesirable loaded elements such as magnesium, sodium, chlorine, calcium, sulfur, iron and phosphorus. Rare earth loaded organic and scrubbing agent (aqueous) were mixed in a beaker with magnetic stirrer speed at scale two (IKA MAG). The concoction is transferred into a conical separating funnel when the mixing is completed, for two-phase separation. Then, the aqueous phase is used for analysis. The elements in aqueous phase (chlorine, calcium and sulfur) were analyzed by Energy Dispersive X-Ray Fluorescence (EDXRF), Thermo Scientific QUANT'X model. Parameters studied at this stage were different type of scrubbing media which consist of variable of concentration.

RESULTS AND DISCUSSION

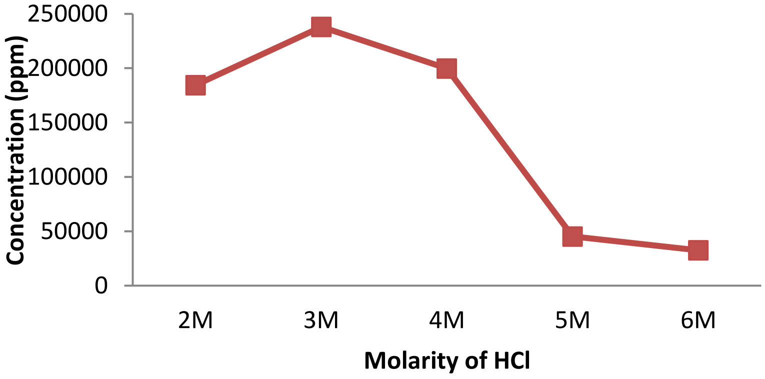

Figure 1: Concentration of extracted yttrium with different hydrochloric acid molarity.

The results are shown in Figure 1. The concentration of extracted yttrium decreases rapidly as the molarity of HCl increases up to 3 M. Beyond this point, the concentration of yttrium rises, possibly due to the availability of more extraction sites for yttrium. However, further increases in HCl molarity do not lead to any significant improvement in the extraction percentage. Overall, the highest concentration of extracted yttrium is observed at 3 M HCl.

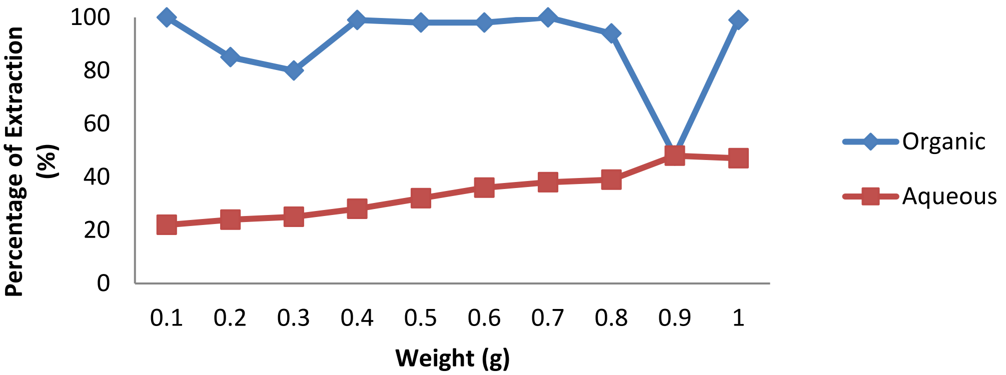

Figure 2: First stage extraction behavior of yttrium as a function of rare earth oxalate weight, contact time: 15 minutes, phase ratio: 1:1

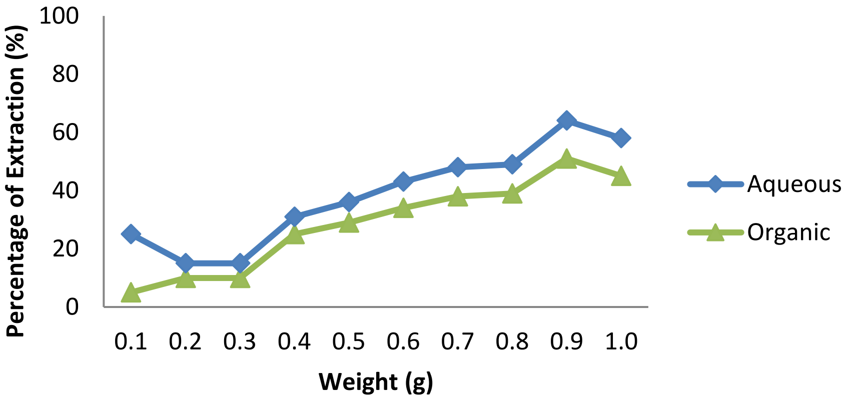

Figure 3: Second stage extraction behavior of yttrium as a function of rare earth oxalate weight, contact time: 15 minutes, phase ratio: 1:1

Figures 2 and 3 illustrate the behavior of yttrium extraction during the first and second stages after the pregnant liquor was produced. The results indicate that the percentage of extraction increases with the weight of rare earth oxalate. In the second stage of extraction, however, the percentage of extraction with 1.0 gram of rare earth oxalate shows a slight decrease, likely due to the limited availability of extraction sites for yttrium. Therefore, 1.0 gram of rare earth oxalate is identified as the optimum amount for yttrium extraction when using 30% DEHPA.

Scrubbing of rare earth loaded organic

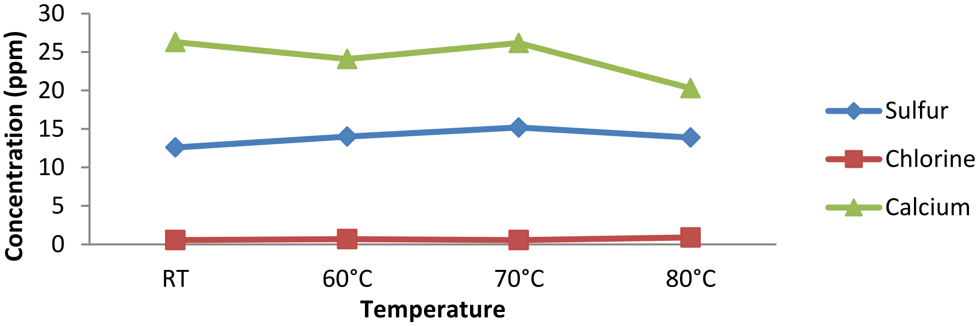

Figure 4: Effect of deionized water to rare earth loaded organic on the removal of inorganic elements.

Figure 4 shows that deionized water at 70 °C can scrub the highest amounts of sulfur (15.2 ppm), chlorine (0.56 ppm), and calcium (26.2 ppm) compared to room temperature, 60 °C, and 80 °C deionized water. The concentrations of sulfur and calcium increase as the temperature rises from room temperature (RT) to 70 °C. In contrast, chlorine removal is more effective at 80 °C, where a greater amount is scrubbed from the rare earth–loaded organic phase. These results were obtained after 10 minutes of contact time with an aqueous-to-organic ratio of 1:1.

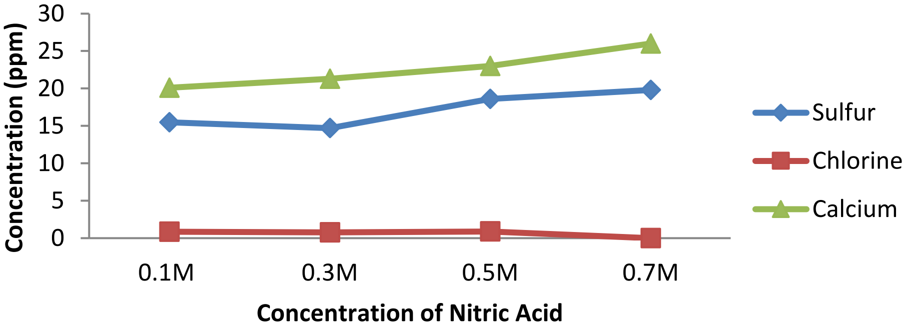

Figure 5: Effect of nitric acid to rare earth loaded organic on the removal of inorganic elements

Figure 5 shows that 0.7 M nitric acid, used as a scrubbing agent, effectively removes sulfur and calcium from the loaded organic phase, with concentrations of 19.8 ppm and 26.0 ppm respectively after 10 minutes of contact time at an aqueous-to-organic ratio of 1:1. For chlorine, the concentration decreases slightly, from 0.89 ppm at 0.5 M nitric acid to 0.00 ppm at 0.7 M.

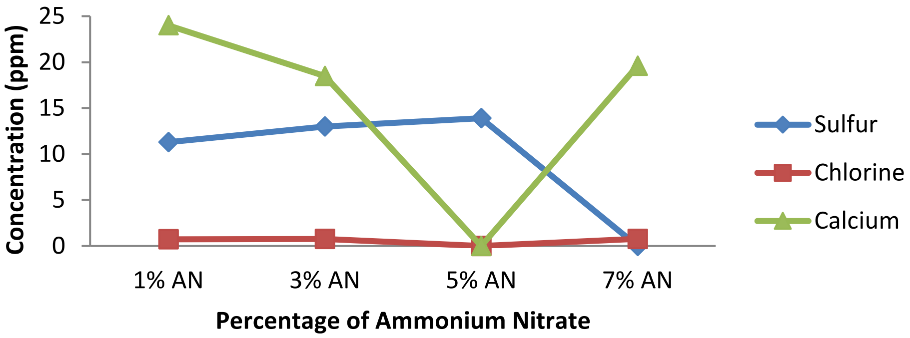

Figure 6: Effect of ammonium nitrate to rare earth loaded organic on the removal of inorganic elements

Figure 6 shows that the concentrations of sulfur and calcium increase rapidly with the percentage of aqueous solution, reaching their highest values at 3% ammonium nitrate after 10 minutes of contact time with an aqueous-to-organic ratio of 2:1. Beyond this point, the concentrations of chlorine and calcium decrease sharply at 5%, likely due to fewer available sites on the scrubbing agent to remove these elements. For chlorine, the concentration rises slightly from 0.73 ppm at 1% ammonium nitrate to 0.76 ppm at 3%, before dropping to 0.00 ppm at 5%.

CONCLUSION

This study proved that yttrium can be extracted from Malaysian Xenotime by extraction using DEHPA (30%) in hydrochloric acid solution. Scrubbing of the rare earth loaded organic phase to remove inorganic (impurities) were performed using deionized water, nitric acid and ammonium nitrate. Hot deionized water with 70°C scrub most sulfur (15.2 ppm), chlorine (0.56 ppm) and calcium (26.2 ppm) compared than other media.

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