Leaching Behaviour of Langkawi Black Sand for the Recovery of Titanium
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Abstract
Leaching behaviour of Langkawi black sand was carried out in this study for the recovery of titanium. In order to determine the maximum leaching condition various parameters like effect of leaching time, temperature, concentration of sulfuric acid, solid to liquid ratio and volume of hydrogen peroxide were studied. The experimental data reveals that the optimum temperature and time were 80 °C and 120 min, respectively. In addition, 5M sulfuric acid, 2 mL of hydrogen peroxide and solid liquid ratio of 0.032 W/V showed the best leaching result.

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1. Introduction
Titanium is a hard, malleable, ductile and lustrous metal and has high strength, low density and excellent corrosion resistance which allow this metal to be used for parts of aircraft, spacecraft, missiles, ships and prosthetic devices.

The concentrated sources of titanium are the minerals ilmenite, titanomagnetite, rutile, anatase and brookite. Black sand is one of the sources which contain ilmenite, magnetite, rutile, garnet and zircon [1]. The economic importance of black sand depends on the type of the occurrence, the extractability of elements and

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the exploitation of particular natural products of industrial interest such as rutile, zircon and ilmenite [2, 3].

Titania was separated from ilmenite fraction of Egypt and Greece black sand by several researchers using hydrochloric acid, sulfuric acid, fused KOH and NaOH. [1, 4, 5]. But there are no reports on the treatment of Langkawi, Malaysia black sand except one study which reported the isotope identification of Langkawi black sand using neutron activation analysis [6].

This study investigates the dissolution of Langkawi black sand by acid leaching. Leaching was performed by sulfuric acid with oxidative reagent. In a through study the dissolution of titanium varies by varying the temperature, acid concentration and solid liquid ratio.

2. Experimental technique

2.1. Reagents

Black sand was collected in a single lot from the Langkawi Island, Malaysia. Its median grain size was 75–300 μm. It was dry ground and used in this study. The chemicals were either of Merck (Germany) or BDH (England) grade and used without further purification.

2.2. Analytical

X-ray diffraction studies was carried out on a Shimadzu X-Ray Diffractometer (XRD 6000) using Co-Kα radiation at a scan rate of 2°/min. Chemical analysis of titanium was determined by spectrophotometric method after dissolution of sand. The spectrophotometric method used for Ti (IV) was sulfuric acid–hydrogen peroxide at 420 nm using UV-2100 UNICO Spectrometer [7].

2.3. Procedure

The titanium content of black sand was measured by baking it with 0.5g of ground anhydrous Na₂CO₃ in a platinum boat which was then heated for 6 hours in a Muffle furnace, at 1000 °C. The baked sand was then contacted with sulfuric acid at 80 °C. The concentration in the resulting solution was measured by spectrophotometric method and the data were used for calculating the dissolution percentage of titanium in different parameters of this experiment.

In order to carry out the experiment, black sand was stirred with sulfuric acid and hydrogen peroxide. Then the solid residue was filtrate out and concentrations in the filtrate were determined spectrophotometric method. In the case of temperature parameter, thermo stated water bath equipped with a thermometer was used.

3. Result and Discussion

3.1. Characterization

The XRD spectra of the black sand sample used in this study are shown in Fig. 1. The auto-matching indicates the presence of two phases of black sand ilmenite and rutile.

3.2. Analysis

3.2.1. Effect of time
Figure 2 shows the effect of leaching time on titanium dissolution. The leaching is increased with increasing the time up to 120 minutes and the value is 57%. After that a pronounced decrease of leaching is observed by a further increase in time. This decrease may be due to the elongation of time of reaction leads to an evaporation of a significant portion of hydrogen peroxide from the reaction mixture [5] However, for subsequent parameters 120 minutes of contact time was adopted.

3.2.2. Effect of temperature

The temperature dependence of Titanium dissolution was studied between the temperature ranges of 50°C to 100 °C. It can be seen from the graph (Fig. 3) that the increase of temperature from 50°C - 80°C leads to an increase of dissolution of black sand and continuing with decreasing after 80 °C. This phenomenon may be ascribed to the thermal transformation of complex bonding which expected to be less soluble in acids [5].
3.2.3. Effect of sulfuric acid concentration

In order to observe the influence of sulfuric acid concentration, the study was conducted varying the sulfuric acid concentration from 1 to 5 M, where other conditions were constant. The result is presented in Fig. 4 and indicated that the dissolution percentage of black sand was increased with the increasing of acid concentration.

3.2.4. Effect of solid liquid ratio

The effect of solid to liquid ratio (S/L, w/v), i.e. the ratio of the weight of black sand to the volume of solution, on the dissolution of titanium was studied between 0.006-0.064 g/mL at a constant sulfuric acid and...
hydrogen per oxide concentration. The result is given in Fig. 5 which is suggesting that efficient dissolution of titanium from black sand was attained around 60% in the S/L ratio of 0.032 g/mL.

3.2.5. Effect of volume of hydrogen peroxide

To evaluate the effect of hydrogen peroxide, the volume of hydrogen per oxide was varied between 0.25 to 2.5 mL, while the other leaching parameters were kept constant. The dissolution percentage vs. volume of hydrogen per oxide curve is shown in Fig. 6. The figure showed that the dissolution percentage was increased with the increasing of hydrogen peroxide volume.

4. Conclusions

The investigation was demonstrated the technical feasibility of Langkawi black sand by leaching with sulfuric acid. Suitable process conditions were 120 min contact time; solid-liquid ratio of 0.032 g/mL; Temperature of 80°C; 5 M sulfuric acid concentration and 2 mL of hydrogen peroxide for titanium dissolution. The dissolution percentage was increased with increasing Temperature up to 80°C after that it decreased. At optimum condition, about 60% Titanium was leached out.

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