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Influence of microwave power and temperature on the silica leaching process

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Abstract. This research presents a study for the silica leaching from peat clay in alkalic sodium hydroxide under microwave-heated condition. Microwave bases leaching was implemented using the Iwaki Pyrex glass reactor in a modified microwave oven. The characterization of peat clay was specified by X-ray diffraction (XRD) analysis and X-ray Fluorescence (XRF) analysis. An investigation was made of the influence of microwave power, temperature, and reaction time on the silica leaching recovery. An enhancement on the silica recovery for 12 M sodium hydroxide, solid/liquid ratio 0.03 g/mL, and 15 min reaction time was obtained 30.62 %-wt and 27.85%-wt for 900 W and 90 °C respectively. The microwave assisted leaching is more efficient regarding overall silica dissolution.

1. Introduction

Indonesia, especially South Kalimantan, has a lot of peat clays. Peat clay contains the main chemical compounds in the form of silica oxide, aluminum oxide and iron oxide which have the potential to be applied as an adsorbent, coagulant, and catalyst in the chemical industry. Based on the results of the investigation by Mirwan et al. [1,2], the composition of peat clay in the Peat Village of South Kalimantan contains 31.9 - 38.8% by weight of silica oxide, 11-17.9% by weight of aluminum oxide, and 11.4 - 27.3% by weight of iron oxide. Silica oxide or SiO₂ is the most abundant material in the earth's crust. It is a valuable multipurpose inorganic chemical compound available in gel, crystalline and amorphous forms. However, the recovery process of pure silica requires a lot of energy. Many conventional industrial processes require highly combustion temperatures in excess of 700 °C.

The leaching process used microwaves has several advantages, namely a fast, selective, flexible heating process, and is uniformly distributed in the particle cavity [3]. In addition, the process is energy efficient and consumes low energy compared to conventional processes. This microwave technology has been used in copper dan chromium leaching [4], Au(III) and Cu(II) [5], zinc ore [6], and palladium, platinum, rhodium and ruthenium [7]. Recently, microwaves have been applied to the aluminum leaching process from peat clay with aluminum ratios of 67% and 46.6% for 100 W and 80 W, respectively [8].

Considering several positive factors for the use of microwaves such as fast, selective, even heating in the particle cavity, and energy saving, research on the application of microwaves to leaching silica from peat clay using variations in microwave power, temperature, reaction time has been carried out and the results are discussed. Microwave-assisted aluminum leaching of peat clay was informed



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previously [8] however, efforts are being made to evaluate its role in silica leaching of the same material first being undertaken.

2. Materials and Method

2.1. Materials

The peat clay sample used in the present studies was collected from Peat Village, South Kalimantan, Indonesia and in the depths approximately 3.0 meters from the surface of the earth. After washing and drying under sunlight directly, the peat clay was crushed and ground using crusher to +200-325 mesh size based on the American Society for Testing and Materials (ASTM). The next step, peat clay was calcined at 700 °C during 2 h for thermal treatment. Sodium hydroxide pellets was obtained from Merck.

Identification of various phases in the peat clay was analyzed with the model Philips X-pert powder X-ray diffractometer (XRD, Netherlands) using the support of International Centre for Diffraction database (ICDD) and X'Pert High Score Plus software to search and match mineralogical composition qualitatively. The chemical composition of peat clay minerals quantitatively was determined using ANalytical type miniPAL4 X-ray fluorescence (XRF, Netherlands).

2.2. Microwave silicate leaching method

The experimental apparatus used for the microwave leaching process is shown in Figure 1. This leaching study has used a household microwave oven capable of providing 900 W of power at a frequency of 2.45 GHz. A 500 mL three neck Iwaki Pyrex glass reactor was placed in a modified microwave oven. To prevent evaporation, a spiral condenser is attached to the glass reactor. The temperature control of the solution in the reactor is measured using a modified thermocouple sensor. In this experiment the process conditions were kept constant, namely 12 M sodium hydroxide concentration, 300 mesh particle size, 300 rpm stirrer speed, 0.03 g/mL solid to liquid ratio, and 60 min reaction time.

A particular amount of dry peat clay sample and 250 mL of sodium hydroxide solution were put into the reactor which is connected to each other. The influences of microwave power (540, 720, and 900 W) and microwave leaching temperature (30, 50, 70, and 90 °C) were studied with a reaction time of 60 minutes and the stirring speed set to 300 rpm. At specific time intervals, the sample solution is picked up with a syringe and filtered for analysis to determine the silicate content using a UV-Vis spectrophotometer (Cary 100 Conc UV-Vis, Agilent CrossLab, US). Silicate concentration was determined using UV-Vis absorbance at a wavelength of 410 nm. Using a calibration curve for silica, the silicate concentration (in ppm) can be determined. The actual silicate concentration, in % by weight, is calculated by the following equation.

$$\text{Actual silicate conc. (wt)} = \left(\frac{\text{conc. (ppm)} \times \text{dilution factor}}{1 \times 10^{-4}} \right)$$

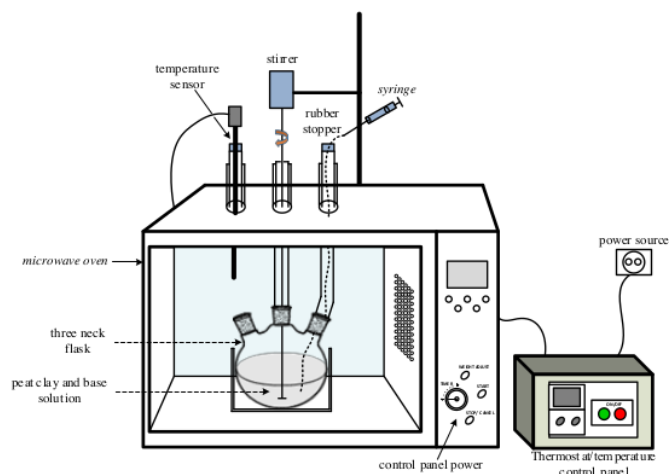


Figure 1. The microwave leaching experiment apparatus

1

3. Results and Discussion

3.1. Characterization of peat clay

XRD analysis indicated that the peat clay has some main compound phases, specifically albite, kaolinite, and montmorillonite (Fig. 2). At different depths and areas, peat clay contains dominant compound phases, namely kaolinite, albite, sillimanite, and montmorillonite [1,2]. The chemical analysis of peat clay with XRF is shown in Table 1. The mineralogical composition of peat clay qualitatively consists of 30.4% SiO₂, 21.7% Al₂O₃, 18.3% Fe₂O₃, and 5.8% MoO₃.

3.2. Effect of microwave power

One of the factors that greatly affects the rate of microwave heating is power. The microwave power used in the study was to determine the effect of different microwave power, namely 540, 720, and 900 W. However, the temperature of the leaching process was kept constant at 90 °C using a thermocouple which was installed in the microwave unit and connected to the temperature control unit. This was done so that the temperature of the leaching reaction can be maintained by adjusting the magnetron power output in the microwave. The results of this study are shown in Figure 3. The actual silicate was increased with the increase in microwave power in the range 540 - 900 W. Peat clay at a microwave power input of 900 W provide silicate leaching ability of about 30.63% by weight for 15 min reaction time. The rate of microwave heating is greatly influenced by power and high microwave power has an effect on increasing metal dissolution [8,9]. The increasing the microwave power is increased the leaching yield for copper and chromium from the spent catalyst phase in a short time of 2 minutes [4].

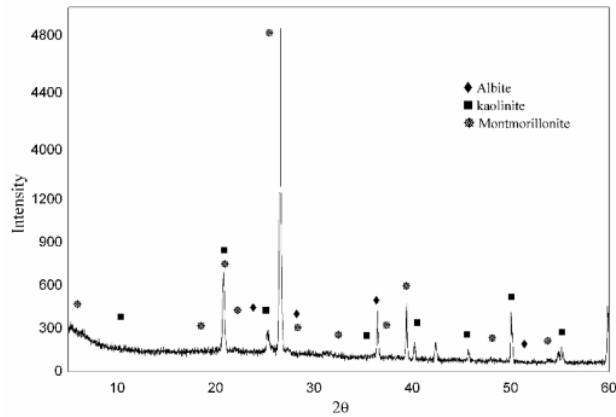


Figure 2. XRD analysis of peat clay

Table 1. The mineralogical composition of peat clay

Elements (oxides)	Weight (%)
SiO ₂	30.4
Al ₂ O ₃	21.7
Fe ₂ O ₃	18.3
K ₂ O	1.12
MnO	0.15
CaO	0.62
TiO ₂	1.82
V ₂ O ₅	0.14
Cr ₂ O ₃	0.22
ZnO	0.08
NiO	0.11
CuO	0.14
MoO ₃	5.8
Re ₂ O ₇	0.19
LOI	19.21

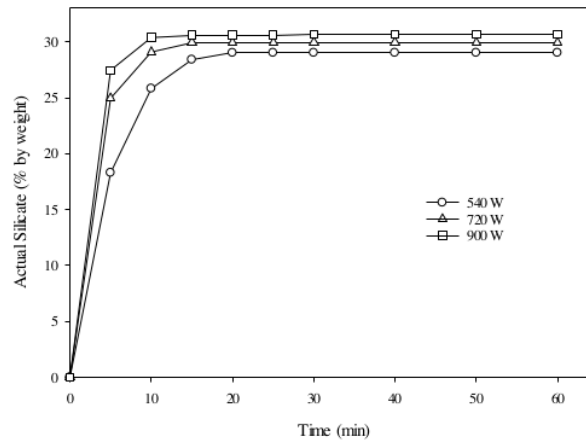


Figure 3. Effect of microwave power on silicate leaching as a function of reaction time

3.3. Effect of temperature

The effect of temperature on silicate mineral leaching in the peat clays was studied by varying the reaction temperature from 30 to 90 °C using a 900 W microwave power input. Figure 4 shown that the the rate of actual-silicate extraction was rapid and overall leaching was increased at initial leaching time 5 min. Afterwards, actual-silicate leaching followed steady and becomes plateau after 20 min of leaching. The leaching behavior shows that with increase in temperature, the actual-silicate leaching yield increases. The actual-silicate obtained by about 27.87 % by weight at 90 °C. According to Mirwan et al. [8] the kinetic energy of peat clay molecules changes to heat energy due to molecular interactions so that the temperature of the leaching process increases.

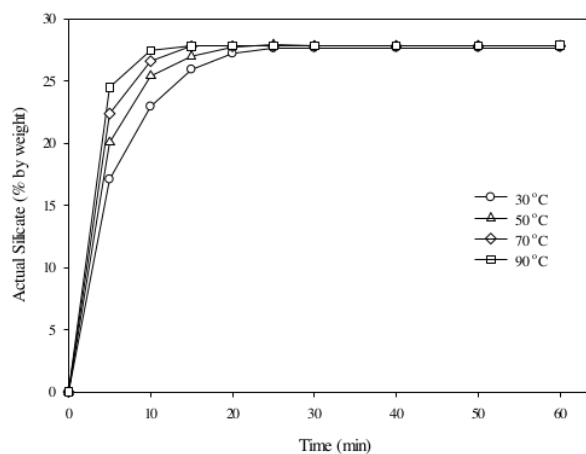


Figure 4. Effect of temperature on silicate leaching as a function of reaction time

4. Conclusions

A research on microwave assisted leaching of silicate from peat clay was investigated. The peat clay for silicate mineralization is albite, kaolinite, and montmorillonite with the main mineralogical composition of SiO₂, Al₂O₃, Fe₂O₃, and MoO₃. The optimum microwave assisted leaching at 12 M sodium hydroxide concentration, 90 C temperature, and 15 min reaction time was achieved 30.63 % by weight for 900 W. The high microwave absorption capability of the silicate mineral matrix was provided a rapid rise in local temperature thereby increased dissolution.

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