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A selective preparation of phillipsite and sodalite from perlite

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Abstract

The preparation of commercially valuable materials, i.e. phillipsite, sodalite octahydrate and faujasite from perlite via inexpensive direct treatment with sodium hydroxide solution, at low temperatures of 70–100 °C under autogeneous pressure, is reported. The formation of phillipsite and sodalite is shown to be selective, whereas the occurring of faujasite is always found with phillipsite. The influences of sodium hydroxide solution concentration, reaction temperature and time on the type of the solid products, and the amount of Na^+ and K^+ incorporated in the zeolitized solids were investigated. The relation between the type of the synthesized zeolites and the uptake of Na^+ and the leaching of K^+ is described, and shown to be significant for further investigation as to expand the application as a cation exchanger.

Keywords: Minerals; Perlite; Phillipsite; Sodalite; Faujasite; X-ray diffraction

1. Introduction

Thailand has considerable mineral resources producing more than 50 different minerals and processed mineral products in 2001. The production of overall industrial minerals has been increasing, 700 metric ton in 1997 to 9915 metric tons in 2001 in the case of perlite [1]. Most of these minerals are commercially low in price due to the exploitation mostly as raw materials. According to the policy of sustainable exploration and development of mineral resources of Thailand [2], a novel way to increase value-added mineral products needs to be explored.

Perlite is a rhyolitic glass making up of more than 70% by weight of silica and 13% of alumina occurring in various types and forms depending on locations of formation. There are three types of natural perlite found in Thailand; banded, classical and pumicious, all of which mostly found in Lopburi province of about one million metric tons in total [3]. The conversion of perlite to zeolitic materials occurring naturally under humid atmosphere and low to medium temperatures (75–250 °C) has been reported [4,5] and hence revealed a novel way in enhancing the commercial value of the mineral. Zeolites have been applied successfully in the chemical industry and environmental protection over the last 35 years due to their remarkable physical and chemical properties, which include molecular sieving, adsorbing and cation exchange capability [6–12]. The attempts to prepare zeolites from perlite in laboratories have been developed continually leading to the formation of various types of zeolites, for example, gismondine, heulandite, zeolite-Pc and zeolite-V. [5–13] The utilization of the zeolitized materials though is limited according to the uncontrolled variation in degree of purity and selectivity.

Here we report the preparation of zeolites, i.e. phillipsite, sodalite octahydrate, which can be prepared selectively, and faujasite, from perlite employing an inexpensive and simple treatment with sodium hydroxide solution at low temperature.

2. Experimental procedure

The perlite from Amphur Lamnarai, Lopburi province, Thailand, was employed as-receive in a powder form with no prior pretreatment in the zeolitization experiments. The zeolitizations were carried out by mixing the perlite powder with aqueous solution of sodium hydroxide (Thasco, 98%) in a weight by volume ratio of 1:5. A range of 1 to 5 mol dm^{-3} solution concentrations was investigated. The reactions were performed under autogeneous pressure in closed containers with 18% filling factor, at two different temper-

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atures, 70 and 100 °C, for various durations between 5 and 20 days. These were to study the influences of reaction temperature and time on the final solid products. The products were then recovered by filtration and washing with distilled water until the pH of the filtrate was lower than 8, followed by rinsing with acetone before leaving to dry in air. Powder X-ray diffractometer (Siemen D500/D501, CuKα, Ni filter, $\lambda = 1.54$ Å) and scanning electron microscope (JEOL JSM-6335 F, Japan) were used to characterize the crystalline solid products and to investigate the morphology of the solids, respectively. The existence of zeotype framework was confirmed by Fourier Transform Infrared (FTIR) spectroscopy (Nicolet 510).

The determinations of the exchangeable alkali metal ions, Na^+ and K^+, which were found in the starting materials (X-ray fluorescent spectrometer, Horiba MESA-500 W), and also used in the zeolitization process in the Na^+ case, were conducted on the zeolitized solids employing atomic absorption spectroscopy (Shidmadzu AA670). The zeolitized solids were treated with concentrated hydrochloric acid in the sample weight by solution volume ratio of 1:10 at 80 °C for 4 h prior to the measurements. This was to confirm the complete exchange of H^+ ions in the solution with the occluded Na^+ or K^+ ions in the zeolitized solids.

3. Results and discussion

3.1. Conversion of perlite to phillipsite, sodalite octahydrate and faujasite

The perlite powder obtained from Amphur Lamnarai, Lopburi province, which is light grey in color with elemental composition in weight percentage; SiO_2 71.67, Al_2O_3 13.45, Fe_2O_3 1.43, Na_2O 2.33, K_2O 4.31, TiO_2 0.39, MnO_2 0.06, H_2O 5.16, is mostly amorphous by powder X-ray diffraction as shown in Fig. 1. This is consistent with the

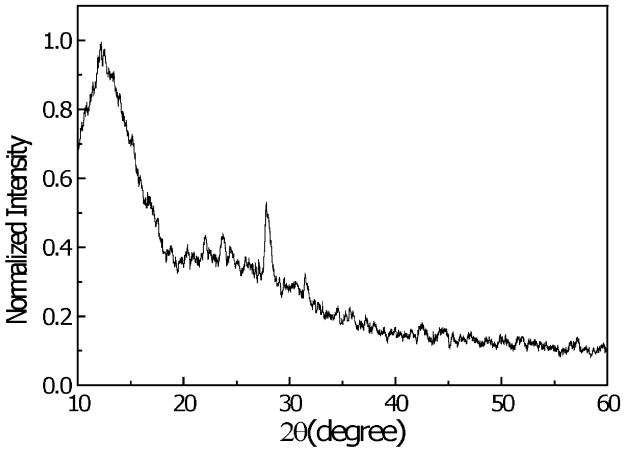

Fig. 1. Powder X-ray diffraction pattern of the perlite powder from Amphur Lamnarai, Lopburi province, Thailand, the starting material for zeolitization experiments, suggesting a very low crystallinity.

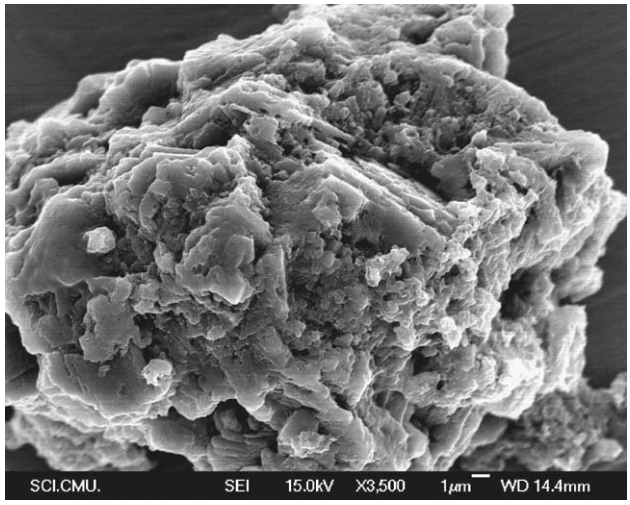

Fig. 2. A scanning electron micrograph of Lamnarai perlite shows an aggregate of melt with porous fragments located on the surface.

electron micrograph (Fig. 2) showing no distinguishing shape, but an aggregate of melt. Porous fragments with no discrete shape can be observed on the surface of the melt. This may correspond to the porous solids altered naturally from perlite, and hence the peaks appearing in the powder X-ray pattern (Fig. 1). These peaks, though, could not be identified due to the insufficient number of the data.

The treatment of perlite under the investigated conditions as summarized in Table 1 led to selective formation of either phillipsite or sodalite octahydrate. Fig. 3a and b

Table 1
Summary of the conditions employed in the treatments of perlite with sodium hydroxide solutions and corresponding solid products identified by powder X-ray diffraction, and the exchangeable Na^+ and K^+ ion contents

[NaOH(aq)] (mol dm^{-3})	Temperature (°C)	Reaction time (day)	Product	Ions content (mmol g^{-1})	
				Na^+	K^+
1	100	5	PHI	1.64(2)	0.70(1)
		10	PHI	1.30(2)	0.58(1)
		15	PHI	1.64(2)	0.72(1)
	70	5	amorphous	–	–
		10	amorphous	–	–
		15	amorphous	–	–
3	100	5	PHI	2.80(2)	0.75(1)
		10	PHI	3.14(6)	0.76(1)
		20	PHI	2.74(3)	0.77(1)
	70	5	PHI	1.71(1)	0.18(1)
		10	PHI	2.41(3)	0.48(1)
		20	PHI	2.95(4)	0.48(1)
5	100	5	SOD	5.98(3)	0.14(1)
		10	SOD + UN	6.50(2)	0.15(1)
		15	PHI	2.96(4)	0.56(1)
	70	5	PHI + FAU	2.85(2)	0.15(1)
		10	PHI + FAU	4.45(3)	0.26(1)
		15	PHI + FAU	4.15(5)	0.26(1)

PHI, SOD, FAU and UN stand for phillipsite, sodalite, faujasite and unidentified phases, respectively. Standard deviations are shown in brackets.