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RESEARCH ARTICLE

Synthesis and characterization of zeolite NaX from Bangka Belitung Kaolin as alternative precursor

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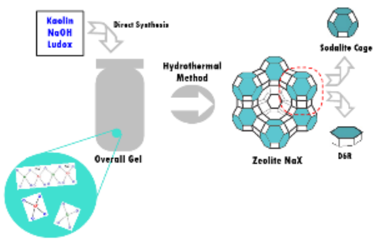
Graphical abstract



Abstract

The potential use of kaolin as silica and alumina precursor for the synthesis of zeolite NaX was investigated in this study. The synthesis involved three steps of reactions; the preparation of seed gel, the formation of feedstock gel using kaolin and the combination of overall gel followed by hydrothermal treatment at 105°C for 12 hours. Analysis using X-ray Diffraction (XRD) method indicated the transformation of kaolin into pure phase zeolite NaX with a small amount of kaolin was still visible. Detail microscopic analysis showed the morphology of zeolite X consisted of octahedral particles with a crystallite diameter of 20-30 µm. Analysis of surface acidity using pyridine as probe molecule indicated the zeolite X has high Brønsted acidity with 0.181 mmol/g of acid sites, significantly higher than Lewis acidity ~0.053 mmol/g. The N_2 adsorption-desorption measurement indicated a type IV material with both microporous and mesoporous structures with an average pore size of 1.47 nm for micropore and 3.41 nm for mesoporous.

Keywords: Kaolin, zeolite NaX, porous materials, hydrothermal method

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INTRODUCTION

Zeolite consists of four connected AlO_4 and SiO_4 tetrahedrons connected via oxygen to form porous aluminosilicates framework. The intracrystalline channel in the zeolite framework is occupied with water molecule and cation to neutralise the negative charge of AlO_4. The mobility of the cation and the flexibility of the size, shape and pore structure of the zeolite allows modification to accommodate catalytic desire. Zeolite is commonly produced from hydrogels aluminate and sodium silicate, however, production of zeolite from alternative silica and alumina source such as bagasse fly ash [1] [2], rice hush ash [3] and kaolin [4] have received considerable attention since the past few decades. Kaolin is naturally occurring minerals that abundantly available in Indonesia particularly in south Sumatra, Bangka Belitung and Java island. Kaolin from Bangka Belitung has low iron and titanium content but rich with silica (54.9 wt.%) and alumina (36 wt.%). Studies were previously carried out for the synthesis of zeolite X using kaolin originated from Bulgaria [5] and also the synthesis of zeolite Y from natural kaolin [6], kaolin obtained from China [7] and Iran [8]. Kaolin is an ideal alternative candidate to replace commercial silica and alumina precursors due to a high level of silica and alumina with relatively low iron content. The use of kaolin as raw material for the synthesis of zeolite offers an alternative economical route by utilising naturally abundant resources rather than commercially available chemical.

The aim to synthesis zeolite NaX is due to its unique three-dimensional pore structure and surface acidity that can be utilised as a solid acid catalyst [9], ion exchanger [10], and adsorbent. Zeolite NaX belongs to faujasite family that composes of sodalite cage with 6-rings (D6R) to form hexagonal framework.

Studies that were carried out on the synthesis of zeolite X from kaolin required acid or base leaching treatment to reduce its resistance towards chemical transformation to zeolite [11]. Kaolin also required thermal treatment at high temperature ~ 700–900°C to form amorphous metakaolin before it can be used for zeolite synthesize [12]. The additional pre-treatment process has an indirect effect on the production cost and also detrimental to the environment i.e., production of greenhouse gases.

Here we investigate the potential of kaolin as alternative silica and alumina precursors without the need for pre-treatment to produce zeolite NaX via hydrothermal method. The physical properties of zeolite X were analysed using XRD, SEM, FTIR and N_2-gas adsorption-desorption methods to obtain its crystal morphology, framework structure, pore structure and surface acidity.

EXPERIMENTAL

Materials

Kaolin from Bangka Belitung contained 36 wt.% Al_2O_3, 54.9 wt.% SiO_2, 3.34 wt.% Fe_2O_3 and 1.88 wt.% K_2O. The materials used in the synthesis were sodium hydroxide (99 wt.% NaOH Merck), sodium aluminate (53 wt.% $NaAlO_2$ Merck), silica colloidal (LUDOX) (30 wt.% SiO_2 and 70 wt.% H_2O Merck), ammonium acetate (Merck) for ion exchanged and acidity characterization. Demineralized water was used for cleaning and chemical preparation.

Synthesis of zeolite NaX

Bangka Belitung kaolin used in this study was obtained from Bangka Belitung (Sumatra, Indonesia). Chemical and mineralogical composition of the received kaolin is listed in Table 1. The as-received kaolin contains a low level of metal oxide impurities with Si to Al molar ratio of 1.53. Kaolin was used for the synthesis of zeolite X without prior pretreatment. The mixture kaolin and other precursors were

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