

INTISARI

PREPARASI DAN KARAKTERISASI FISIKO KIMIA ZEOLIT TERAKTIVASI ASAM SULFAT DAN BENTONIT BERPILAR ALUMINA DENGAN TEKNIK KALSINASI GELOMBANG MIKRO

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Telah dilakukan sintesis dan karakterisasi alumina silikat yaitu zeolit dan bentonit terpillar Al_2O_3 , serta uji katalitiknya dalam reaksi esterifikasi. Zeolit alam diaktivasi menggunakan H_2SO_4 , kemudian dikalsinasi dengan *oven* gelombang mikro (*microwave*), sedang bentonit alam dilanjutkan pula dengan pilarisasi menggunakan ion Keggin Al_{13} . Dengan demikian pada penelitian ini dipelajari pengaruh perlakuan konsentrasi H_2SO_4 , suspensi Keggin, rasio OH/Al, dan waktu kalsinasi terhadap sifat fisiko kimiawinya. Selain itu dipelajari pula aktivitasnya pada sintesis etil asetat dari reaksi antara etanol dan asam asetat. Katalis yang diperoleh dikarakterisasi dengan metode difraksi sinar X, *Transmission electron Microscopy* (TEM), analisis serapan gas N_2 (BET), sedang untuk keasaman permukaan menggunakan metode adsorpsi amonia, piridin dan analisis spektroskopi infra merah (FTIR).

Hasil difraktogram XRD, Spektre FTIR yang diperoleh menunjukkan bahwa tidak terjadi perubahan struktur permukaan, sementara SEM dan TEM memperlihatkan pembukaan kanal dan pori. Sifat fisikokimiawi Zeolit teraktivasi lebih rendah dibanding bentonit terpillar, yaitu pada keasaman dan porositas, sedang luaspermukaan dan diameter pori lebih tinggi.

Optimasi zeolit teraktivasi tercapai pada asam sulfat 2 M dan waktu kalsinasi 20 menit yaitu dengan *basal spacing* 9,08 Å, luas permukaan 251,00 m^2/g , volume pori 0,12 cm^3/g , diameter pori 9,89 nm daya adsorpsi masing-masing 0,90 mmol amonia/g dan 0,038 mmol piridin/g., sedang bentonit terpillar terjadi pada konsentrasi asam sulfat 2 M dan waktu kalsinasi 20 menit, rasio Keggin 2,2 dan suspensi 10 mmol Al_{13} /g dengan *basal spacing* 14,19Å, luas permukaan 154,64 m^2/g , volume pori 0,13 cm^3/g , diameter pori 3,38 nm dan daya adsorpsi masing-masing sebesar 11,75 mmol amonia/g dan 2,44 mmol piridin/g. Uji aktivitas menunjukkan bahwa persentase etil asetat yang dihasilkan menggunakan katalis zeolit alam aktif dan bentonit terpillar Al_{13} masing – masing sebesar 20,53 dan 19,76%.

Kata kunci : Zeolit, Bentonit, Aktivasi, Pilarisasi, Karakterisasi.

ABSTRACT

PREPARATION AND PHYSICO-CHEMICAL CHARACTERIZATION OF SULFURIC ACID-ACTIVATED ZEOLITE AND ALUMINA-PILLARED BENTONITE USING MICROWAVE CALCINATION TECHNIQUE

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Preparation and characterization of aluminosilicate compounds as H-zeolite and Al_2O_3 -pillared bentonite have been conducted as well as their catalytic activities on esterification reaction. The aim of this research were to study the effect of H_2SO_4 and Keggin ion concentration, the ratio OH/Al, and calcination time to the physicochemical properties of the aluminosilicates. Natural zeolite and bentonite were activated by H_2SO_4 then calcined with microwave oven, meanwhile natural bentonite was followed by pillarization using Keggin ion (Al_{13}). Next, the compounds were characterized by X-ray diffraction, Transmission Electron Microscopy (TEM) and N_2 gas sorption analysis (BET) methods. The surface acidities were characterized using ammonia and pyridine adsorption as well as infrared spectroscopy (FTIR) methods. The activated catalysts were also tested their catalytic activities on the synthesis of ethyl acetate from ethanol and acetic acid.

XRD-Diffractograms and FTIR spectra obtained show that there is no change in the surface structure of the compounds, The characterization results exhibit also that pillarization on bentonite was successful. SEM and TEM images show the opening of channels and pores for both compounds. The nature of the activated zeolite was lower than pillared bentonite, especially in terms of acidity, surface area and porosity.

Optimized value was achieved at a concentration of 2 M sulfuric acid and calcination time of 20 minutes, i.e on basal spacing 9,08 Å, surface area of 251.00 m^2/g , pore volume of 0.12 cm^3/g , a pore diameter of 9.89 nm, ammonia and pyridine adsorption capacities of 0.90 mmol/g and 0.038 mmol/g, respectively, for pillared bentonite occurred at a concentration of 2 M sulfuric acid, calcination time of 20 minutes, mol ratio of 2.2 and concentration of Al_{13} Keggin of 10 mmol/g with a basal spacing 14,49 Å, surface area of 154.64 m^2/g , basal spacing of 1,42 nm, pore volume of 0,13 cm^3/g , pore diameter of 3, 8 nm and the adsorption capacities of 11.75 mmol ammonia /g and 2.44 mmol pyridine/g, respectively. The catalytic activities showed that the percentage product of ethyl acetate using acidified natural zeolite and alumina pillared bentonite catalysts is 20.53 and 19.76% respectively.

Keywords: Zeolite, Bentonite, Activation, Pillarization, Characterization.

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