RINGKASAN

Limbah *Fly ash* batubara menjadi salah satu limbah berbahaya baik untuk lingkunngan ataupun manusia. Kandungan logam-logam berat dengan ukuran partikel yang sangat kecil menjadi penyebabnya karena dapat mencemari lingkungan dan masuk dalam tubuh manusia melalui rantai makanan ataupun pernapasan. Selain logam berat, terdapat kandungan alumina dan silika dalam jumlah besar sehingga dapat dimanfaat sebagai bahan material berpori salah satunya ialah zeolit ZSM-5. Alumina dan silika dari *fly ash* batubara diekstraksi menggunakan metode refluks dengan larutan alkali. Ekstrak *fly ash* batubara (EFA) digunakan sebagai bahan utama sintesis zeolite menggunakan metode hidrotermal pada suhu 150°C dengan mevariasikan waktu hidrotermal (10, 24, 48, 72 dan 120 jam) dan waktu *aging* (0, 6, 12 dan 24 jam).

Melalui karakterisasi XRF, diketahui komposisi alumina dan silika dalam EFA meningkat dari 16,7669% dan 30,8462% menjadi 42,04% dan 53,42%. Hasil sintesis pada variasi waktu hidrotermal dan *aging* telah menunjukan keberhasilan terbentuknya struktur zeolit melalui hasil spektra FTIR yang muncul serapan pada rentang bilangan gelombang 950-1250 cm^{-1} (vibrasi ulur asimetri TO_4), 650-720 cm^{-1} (vibrasi ulur simetri TO_4), dan 500-650 cm^{-1} (vibrasi cincin ganda zeolit) pada semua sampel hasil sintesis. Analisa data XRD menggunakan *software X'Pert Highscore* menunjukan keseluruhan sampel memiliki puncak difraksi pada 2θ 13,95°; 24,29°; 34,63° dan 42,75°. Dimana pola difraksi tersebut mengindikasiakn fasa zeolite ZK-14 (SOD) berdasarkan ICSD no. 201587, COD no.96-152-9731 dan IZA *database.* Namun pada waktu *aging* 6 dan 12 jam terdeteksi puncak difraksi ZSM-5 pada 2θ 22,93° dan 22,94°.

Perbedaan waktu hidrotermal dan *aging* sangat berpengeruh terhadap *%yield*, kristalinitas dan ukuran partikel yang dihasilkan. Dimana ketiga hal tersebut berbanding lurus dengan meningkatnya waktu hidrotermal, namun berbanding terbalik dengan waktu *aging* kecuali pada *%yield* yang tidak terpengaruh secara signifikan. Dengan %*yield* tertinggi 37,83% pada waktu hidrotermal 48 jam, kristalinitas tertinggi 50,73% pada waktu hidrotermal 72 jam dan ukuran partikel terbesar 23,82 nm pada waktu hidrotermal 120 jam. Pada variai waktu *aging* cenderung membentuk fasa amorf karena pengaruh dari laju pembentukan nukleasi pada periode induksi yang tidak diikuti dengan peningkatan waktu hidrotermal sehingga membentuk ion aluminosilikat amorf dalam jumlah yang banyak.

Kata Kunci: *Fly ash* Batubara, ZSM-5, Zeolit, Waktu Hidrotermal, Waktu *Aging*

SUMMARY

Fly ash from coal combustion is considered one of the hazardous wastes, posing significant risks to both the environment and human health. The presence of heavy metals in finely particulate form is a major concern, as it can contaminate the environment and enter the human body through the food chain and respiration. Additionally, the substantial content of alumina and silica in fly ash makes it a valuable source for porous materials, such as zeolite ZSM-5. The extraction of alumina and silica from coal fly ash is accomplished using a reflux method with an alkaline solution. The resulting extract, known as Extracted Fly Ash (EFA), serves as the primary material for synthesizing zeolite. This synthesis process occurs via a hydrothermal method at 150°C, with variations in hydrothermal time (10, 24, 48, 72, and 120 hours) and aging time (0, 6, 12, and 24 hours).

Through X-ray Fluorescence (XRF) analysis, it is evident that the composition of alumina and silica within EFA increases substantially, from 16.7669% and 30.8462% to 42.04% and 53.42%, respectively. The results of the synthesis under different hydrothermal and aging conditions indicate the successful formation of zeolite structures. This is confirmed by Fourier Transform Infrared (FTIR) spectra, which reveal absorptions within the wavenumber ranges of 950-1250 cm^{-1} (asymmetric stretching vibrations of TO_4), 650-720 cm^{-1} (symmetric stretching vibrations of TO_4), and 500-650 cm^{-1} (double ring vibrations characteristic of zeolites) in all synthesized samples. X-ray Diffraction (XRD) analysis, conducted using the X'Pert Highscore software, demonstrates that all samples exhibit diffraction peaks at 2θ values of 13.95°, 24.29°, 34.63°, and 42.75°. These diffraction patterns suggest the presence of the zeolite ZK-14 (SOD) phase, as indicated by the ICSD no. 201587, COD no. 96-152-9731, and IZA database references. Notably, during the aging process of 6 and 12 hours, additional diffraction peaks associated with ZSM-5 are detected at 2θ values of 22.93° and 22.94°.

The variation in hydrothermal and aging times significantly influences the %yield, crystallinity, and particle size of the synthesized zeolites. In general, these three parameters are directly proportional to the increase in hydrothermal time. However, aging time exhibits an inverse relationship with these parameters, with the exception of %yield, which remains relatively unaffected. The highest %yield, 37.83%, is achieved after 48 hours of hydrothermal treatment, while the highest crystallinity, 50.73%, is attained following 72 hours of hydrothermal treatment. The largest particle size, 23.82 nm, is observed after 120 hours of hydrothermal treatment. During the aging process, particularly in cases of 6 and 12 hours, an amorphous phase tends to form due to the impact of nucleation rate during the induction period, which is not followed by an extended hydrothermal treatment period. This leads to the substantial formation of amorphous aluminosilicate ions.

Keywords: Coal fly ash, ZSM-5, Zeolite, Hydrothermal time, Aging time