

## **SINTESIS MCM-41-NH<sub>2</sub> BERBASIS SILIKA DARI LUMPUR LAPINDO UNTUK TRANSESTERIFIKASI MINYAK GORENG BEKAS**

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### **INTISARI**

Telah dilakukan sintesis MCM-41 secara hidrotermal berbasis Silika dari lumpur Lapindo yang difungsionalisasi dengan gugus amina dari (3-aminopropil) trimetoksisilan (3-APTMS) menjadi katalis basa MCM-41-NH<sub>2</sub>. Tujuan dari penelitian ini adalah untuk mempelajari teknik penghilangan *cetiltrimethylammonium bromide* (CTAB) yang digunakan sebagai cetakan dalam sintesis MCM-41 dan uji aktivitas katalis basa MCM-41-NH<sub>2</sub> pada transesterifikasi minyak goreng bekas. Silika diekstraksi dengan larutan HCl 6 M dan NaOH 6 M. Kandungan Silika dalam lumpur dikarakterisasi dengan XRF. Sintesis MCM-41 dilakukan secara hidrotermal dalam autoklaf pada suhu 100 °C selama 24 jam. Penghilangan CTAB dilakukan dengan 2 metode: kalsinasi pada suhu 540 °C selama 5 jam (MCM-41-K) dan refluks dengan etanol-HCl pada suhu 70 °C selama 6 jam (MCM-41-R). Fungsionalisasi MCM-41 dengan teknik *grafting* melalui penambahan 3-APTMS dengan rasio N/Si 5 % menghasilkan katalis basa MCM-41-NH<sub>2</sub>. Sampel MCM-41 dan MCM-41-NH<sub>2</sub> dikarakterisasi menggunakan XRD, FT-IR, BET, dan TEM. Produk hasil transesterifikasi dikarakterisasi menggunakan GC-MS.

Hasil analisis XRF menunjukkan kandungan silika dalam lumpur Lapindo 21,00 % (b/b) dan silika yang berhasil diekstraksi dari lumpur Lapindo 93,40 % (b/b). Analisis XRD terhadap MCM-41 sebelum penghilangan cetakan CTAB (MCM-41-C) menunjukkan puncak karakteristik MCM-41 pada  $2\theta$  antara 2-5° dengan luas permukaan spesifik dari data BET 27,63 m<sup>2</sup> g<sup>-1</sup>. Hasil FT-IR menunjukkan metode penghilangan CTAB yang paling efektif adalah menggunakan metode kalsinasi. Analisis XRD terhadap MCM-41 setelah penghilangan CTAB tidak menunjukkan puncak karakteristik MCM-41 namun dengan luas permukaan spesifik BET 777,95 m<sup>2</sup> g<sup>-1</sup> dengan diameter pori 4,53 nm serta memiliki karakter mesopori dengan pola isotherm adsorpsi-desorpsi tipe IV dan *hysteresis loops* tipe H1. Katalis MCM-41-NH<sub>2</sub>-K memiliki luas permukaan 637,26 m<sup>2</sup> g<sup>-1</sup>. Citra TEM MCM-41 sebelum dan setelah penghilangan CTAB menunjukkan distribusi pori yang teratur dengan bentuk heksagonal seperti sarang lebah. Konversi produk transesterifikasi tertinggi diperoleh menggunakan katalis MCM-41-NH<sub>2</sub>-K yakni 55,54 % (b/b).

Kata kunci: MCM-41, MCM-41-NH<sub>2</sub>, lumpur Lapindo, transesterifikasi.

## **SYNTHESIS OF MCM-41-NH<sub>2</sub> BASED ON SILICA FROM LAPINDO MUD FOR TRANSESTERIFICATION OF WASTE FRYING OIL**

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### **ABSTRACT**

The synthesis of MCM-41 based on Silica from Lapindo mud by hydrothermal method functionalized with amine group from (3-aminopropyl) trimetoxysilane (3-APTMS) to produce the MCM-41-NH<sub>2</sub> base catalysts has been done. The purpose of this research is to study the cetyltrimethylammonium bromide (CTAB) removal technique that was used as template in the synthesis of MCM-41 and to verify the catalytic activity of MCM-41-NH<sub>2</sub> as base catalyst in the transesterification of waste frying oil. The Silica was extracted from Lapindo mud using 6 M of HCl dan NaOH solution. The Silica content was characterized by XRF. The synthesis of MCM-41 was carried out by hydrothermall method in an autoclave at 100 °C for 24 h. The CTAB removal was carried out by 2 methods: calcination at temperature of 540 °C for 5 h (MCM-41-K) and reflux with ethanol-HCl 70 °C for 6 h (MCM-41-R). Functionalization with grafting technique by adding 3-APTMS with molar ratio of N/Si 5 % into MCM-41 produced the MCM-41-NH<sub>2</sub> base catalyst. The MCM-41 and MCM-41-NH<sub>2</sub> samples were characterized by XRD, FT-IR, BET, and TEM. The transesterification product was characterized by GC-MS.

The XRF analysis results showed that the content of silica in Lapindo mud was 21.00 wt% and the silica extracted from Lapindo mud was 93.40 wt%. The XRD analysis of the MCM-41 before removal of CTAB (MCM-41-C) showed characteristic peaks on 2 $\Theta$  between 2-5° with specific surface area of 27.63 m<sup>2</sup> g<sup>-1</sup>. The FT-IR result indicated that the most effective removal of CTAB was thatcarried out by calcination method. The XRD analysis of the MCM-41 after removal of the CTAB didn't show the characteristic peak of MCM-41 but showed greater spesific surface area of 777.95 m<sup>2</sup> g<sup>-1</sup> with pore diameter of 4.53 nm dan has a mesoporous character with the pattern of adsorption-desorption isotherm type IV and hysteresis loops type H1. The MCM-41-NH<sub>2</sub>-K catalyst has spesific surface area of 637.26 m<sup>2</sup> g<sup>-1</sup>. The TEM images of MCM-41 samples before and after CTAB removal showed ordered pore distribution with a hexagonal (honeycomb) shape. The highest conversion product of transesterification was obtained using MCM-41-NH<sub>2</sub>-K catalyst, ie 55.54 wt%.

Keywords: MCM-41, MCM-41-NH<sub>2</sub>, Lapindo mud, transesterification.