

KONVERSI SATU LANGKAH (ONE STEP) MINYAK SAWIT BEKAS MENJADI BIODIESEL DENGAN KATALIS BIFUNGSIONAL Ni/SILIKA MESOPORI - NH₂/SILIKA MESOPORI, DAN Ni-NH₂/SILIKA MESOPORI DARI LUMPUR SIDOARJO TERCETAK CTAB

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INTISARI

Sintesis silika mesopori dari lumpur Sidoarjo tercetak CTAB (SM) sebagai pengembangan katalis telah berhasil dilakukan. SM dimodifikasi dengan impregnasi logam nikel dan atau teknik *grafting* berupa penambahan gugus amina (NH₂). Katalis bifungsional (Ni/SM – NH₂/SM dan Ni-NH₂/SM) digunakan untuk uji aktivitas katalis dalam reaksi satu langkah esterifikasi-transesterifikasi konversi minyak sawit bekas menjadi biodiesel. Penelitian ini bertujuan untuk mempelajari karakter katalis silika mesopori tercetak CTAB, katalis bifungsional Ni-NH₂/SM, Ni/SM - NH₂/SM, variasi kondisi reaksi meliputi konsentrasi katalis, waktu, suhu, rasio molar minyak:metanol dan uji *reusability* pada uji aktivitas katalis. Karakterisasi katalis dilakukan dengan FTIR, XRD, XRF, TEM, SAA, NH₃-TPD, dan CO₂-TPD. Produk cair terbaik hasil esterifikasi-transesterifikasi dianalisis menggunakan GC-MS, FTIR, ¹H NMR, ¹³C NMR.

Hasil penelitian menunjukkan pengembangan katalis silika mesopori memiliki luas permukaan spesifik 1.452,3 m² g⁻¹. Katalis Ni/SM, NH₂/SM, dan Ni-NH₂/SM memiliki luas permukaan, diameter pori dan volume pori berturut-turut sebesar 739,7 m² g⁻¹, 3,6 nm, 0,7 cm³ g⁻¹; 21,8 m² g⁻¹, 18,5 nm, 0,1 cm³ g⁻¹; dan 540,7 m² g⁻¹, 3,2 nm, 0,4 cm³ g⁻¹. Katalis Ni/SM - NH₂/SM menghasilkan rasio basa/asam tertinggi sebesar 1,9 mmol g⁻¹ dibandingkan katalis Ni-NH₂/SM sebesar 0,6 mmol g⁻¹. Katalis Ni-NH₂/SM menghasilkan *yield* 44,4%, sedangkan katalis Ni/SM - NH₂/SM memperoleh hasil optimum 78,7% pada kondisi reaksi 3% berat katalis, waktu reaksi 180 menit, rasio mol minyak:metanol 1:21, dan suhu reaksi 65 °C. Analisis GC-MS menunjukkan hasil biodiesel dengan kandungan utama berturut-turut yaitu metil oleat, metil palmitat, dan metil linoleat sebesar 40,5, 37,5, dan 13,6%. Analisis FTIR produk biodiesel menunjukkan puncak khas pada bilangan gelombang 1018 cm⁻¹ yang merujuk pada vibrasi ulur simetri C-O-C. Hasil analisis ¹H NMR menunjukkan pergeseran kimia khas biodiesel pada 3,6 ppm. Analisis ¹³C NMR menunjukkan adanya puncak karakteristik metil ester pada 51,4 ppm. Uji *reusability* katalis Ni/SM - NH₂/SM setelah penggunaan ketiga kali menghasilkan *yield* biodiesel sebesar 72%.

Kata kunci : lumpur Sidoarjo, silika mesopori, katalis bifungsional, minyak sawit bekas, biodiesel

ONE STEP CONVERSION WASTE PALM OIL INTO BIODIESEL USING BIFUNCTIONAL CATALYST Ni/MESOPOROUS SILICA - NH₂/MESOPOROUS SILICA, AND Ni-NH₂/MESOPOROUS SILICA FROM SIDOARJO MUD PRINTED CTAB

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ABSTRACT

Synthesis of mesoporous silica from Sidoarjo mud Templated with CTAB (MS) as catalyst support has been successfully carried out. MS was modified with nickel metal impregnation and/or grafting techniques by adding amine groups (NH₂). Bifunctional catalysts (Ni/MS – NH₂/MS and Ni-NH₂/MS) were used to test catalyst activity in a one-step esterification-transesterification reaction converting waste palm oil into biodiesel. This study aimed to investigate the characteristics of CTAB-templated mesoporous silica catalysts, bifunctional catalysts Ni-NH₂/MS, Ni/MS - NH₂/MS, and the variation of reaction conditions including catalyst concentration, time, temperature, methanol to oil ratio, and reusability tests on catalyst activity. Catalyst characterization was carried out using FTIR, XRD, XRF, TEM, SAA, NH₃-TPD, and CO₂-TPD. The best liquid product from the esterification-transesterification was analyzed using GC-MS, FTIR, ¹H NMR, and ¹³C NMR.

The results showed that the mesoporous silica catalyst support had a specific surface area of 1,452.3 m² g⁻¹. The Ni/MS, NH₂/MS, and Ni-NH₂/MS catalysts had surface areas, pore diameters, and pore volumes of 739.7 m² g⁻¹, 3.6 nm, 0.7 cm³ g⁻¹; 21.8 m² g⁻¹, 18.5 nm, 0.1 cm³ g⁻¹; and 540.7 m² g⁻¹, 3.2 nm, 0.4 cm³ g⁻¹, respectively. The Ni/MS - NH₂/MS catalyst produced the highest base/acid ratio of 1.9 mmol g⁻¹ compared to the Ni-NH₂/MS catalyst at 0.6 mmol g⁻¹. The Ni-NH₂/MS catalyst yielded 44.4%, while the Ni/MS - NH₂/MS catalyst achieved an optimum yield of 78.7% under reaction conditions of 3 wt% catalyst, 180 minutes reaction time, oil:methanol molar ratio of 1:21, and reaction temperature of 65 °C. GC-MS analysis showed biodiesel products with main components being methyl oleate, methyl palmitate, and methyl linoleate at 40.5%, 37.5%, and 13.6%, respectively. FTIR analysis of the biodiesel product showed a characteristic peak at a wavenumber of 1018 cm⁻¹ referring to the symmetric stretching vibration of C-O-C. ¹H NMR analysis showed a characteristic chemical shift for biodiesel at 3.6 ppm. ¹³C NMR analysis indicated the presence of a characteristic methyl ester peak at 51.4 ppm. The reusability test of the Ni/MS - NH₂/MS catalyst after the third use resulted in a biodiesel yield of 72%.

Keywords : Sidoarjo mud, mesoporous silica, bifunctional catalysts, waste palm oil, biodiesel