KATALIS ASAM SO_4/TiO_2 DAN BASA CaO/TiO_2: PREPARASI, KARAKTERISASI DAN APLIKASINYA DALAM KONVERSI MINYAK SAWIT BEKAS MENJADI BIODIESEL

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INTISARI

Preparasi katalis asam SO_4/TiO_2 dan basa CaO/TiO_2 telah berhasil dilakukan dalam konversi minyak sawit bekas menjadi biodiesel. Katalis asam dipreparasi dengan metode impregnasi basah TiO_2 dan larutan H_2SO_4 konsentrasi 0,7; 0,9; 1,1; 1,3 dan 1,5 M (v/v). Katalis basa dipreparasi dari TiO_2 dan CaO konsentrasi 1, 5, 10, 15 dan 20% (wt) dalam reaktor autoklaf. Pembuatan katalis asam dan basa dilakukan menggunakan $NaHCO_3$ sebagai agen pencetak pori dan kalsinasi temperatur 400, 500, 600, 700 dan 800 ˚C. Karakterisasi katalis dilakukan menggunakan FTIR, XRD, SEM-EDS, TEM, SAA dan TGA-DSC. Total keasaman katalis SO_4/TiO_2 ditentukan dengan metode adsorpsi amonia dan kebasaan katalis CaO/TiO_2 dengan metode titrasi asam basa. Katalis asam digunakan untuk mereduksi asam lemak bebas (FFA) dari minyak sawit bekas dan katalis basa untuk mengkonversi hasil esterifikasi menjadi biodiesel.

Hasil penelitian menunjukkan bahwa katalis SO_4/TiO_2 yang dipreparasi dengan H_2SO_4 1,5 M (v/v) dan kalsinasi temperatur 600 ˚C (ST-1,5-600) merupakan katalis yang memiliki keasaman tinggi (2,486 mmol NH_3 g^{-1}), fase kristalin anatase 100%, kandungan unsur sulfur 1,36%, ukuran partikel 50-250 nm, luas permukaan 6,559 m^2/g, distribusi pori optimum 3 nm dan mengalami kehilangan masa pada temperatur 250, 650 dan 800 ˚C. Katalis ST-1,5-600 mampu menurunkan kadar FFA minyak sawit bekas dari 1,5562 menjadi 0,5305% dengan persentase penurunan 66,231%. Katalis CaO/TiO_2 yang dipreparasi dengan CaO 20% (wt) dan kalsinasi temperatur 700 ˚C (CT-20-700) memiliki kebasaan tinggi (8,1270 mmol HCl g^{-1}), fase kristalin TiO_2 anatase, $Ca(OH)_2$, CaO dan $CaTiO_3$, kandungan unsur kalsium 9,96%, ukuran partikel 15-120 nm, luas permukaan 8,292 m^2/g, distribusi pori optimum 4 nm dan kestabilan termal hingga temperatur 600 ˚C. Berdasarkan analisis ^{1}H-NMR dan GC-MS, menjelaskan katalis CT-20-700 mampu menghasilkan konversi biodiesel sebesar 58,13% dengan senyawa metil ester utama yaitu metil oleat (58,23%).

Kata kunci: Katalis asam dan basa, esterifikasi, asam lemak bebas, metil oleat, transesterifikasi, biodiesel

SO_4/TiO_2 ACID AND CaO/TiO_2 BASE CATALYSTS: PREPARATION, CHARACTERIZATION AND THEIR APPLICATIONS IN CONVERTION OF WASTE PALM OIL INTO BIODIESEL

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ABSTRACT

SO_4/TiO_2 acid catalyst and CaO/TiO_2 base catalyst have been successfully prepared in conversion of waste palm oil into biodiesel. The acid catalyst was prepared through wet impregnation of TiO_2 with H_2SO_4 solution at concentrations of 0.7; 0.9; 1.1; 1.3 and 1.5 M (v/v). The base catalyst was prepared using TiO_2 and CaO at concentration of 1, 5, 10, 15 and 20 (wt%) in an autoclave reactor. The acid and base catalysts were treated with $NaHCO_3$ as pore-forming agent and then calcined at temperatures of 400, 500, 600, 700 and 800 °C. Characterizations of the catalysts were carried out by FTIR, XRD, SEM-EDS, TEM, SAA and TGA-DSC. Total acidity of the SO_4/TiO_2 catalyst was determined using the ammonia adsorption method and basicity of the CaO/TiO_2 catalyst was measured by acid-base titration method. The acid catalyst was used to reduce the free fatty acid (FFA) of waste palm oil and base catalyst to convert esterified yield into biodiesel, respectively.

The results showed that the SO_4/TiO_2 treated with 1.5 M H_2SO_4 (v/v) and calcined at 600 ˚C (ST-1.5-600) was the catalyst with high total acidity (2.486 mmol NH_3 g^{-1}), anatase crystalline phase, sulfur content of 1.36%, particle size of 50-250 nm, surface area of 6.559 m^2/g, optimum pore distribution of 3 nm and mass loss at temperature of 250, 650 and 800 ˚C. The ST-1.5-600 catalyst was reduced FFA level of waste palm oil from 1.556 to 0.5365% with a percentage reduction of 66,231%. The CaO/TiO_2 treated with CaO 20% (wt) and calcined at 700 ˚C (CT-20-700) was the catalyst with high basicity (8.127 mmol HCl g^{-1}), obtained anatase, $Ca(OH)_2$, CaO and $CaTiO_3$ crystalline phases, calcium content of 9.96%, particle size of 15-120 nm, surface area of 8.291 m^2/g, optimum distribution pore of 4 nm and thermal stability until 600 ˚C. Based on GC-MS and ^{1}H-NMR analysis, the CT-20-700 catalyst produced of 58.13% biodiesel with the main compound of methyl ester is methyl oleate (58.23%).

Keywords: Acid and base catalysts, esterification, free fatty acid, methyl oleate, transesterification, biodiesel