

SINTESIS DAN KARAKTERISASI KATALIS SO_4/ZrO_2 DAN Zr/CaO SERTA APLIKASINYA DALAM PEMBUATAN BIODIESEL DARI MINYAK KELAPA BEKAS

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INTISARI

Katalis SO_4/ZrO_2 dan Zr/CaO telah berhasil disintesis dan diaplikasikan untuk mengkonversi minyak kelapa bekas menjadi biodiesel melalui dua tahap, yakni esterifikasi dan transesterifikasi. Katalis asam SO_4/ZrO_2 disintesis dengan metode impregnasi basah dan dioptimasi dengan variasi konsentrasi H_2SO_4 sebesar 0,5; 0,7; dan 0,9 M serta temperatur kalsinasi 400, 500, 600, dan 700 °C untuk memperoleh katalis dengan keasaman total tertinggi. Katalis basa Zr/CaO disintesis dengan metode refluks menggunakan pemanasan *microwave* dan dioptimasi dengan variasi persen berat Zr terhadap Zr/CaO yakni 1%, 5% dan 10% serta temperatur kalsinasi 600, 700, 800, dan 900 °C untuk memperoleh katalis dengan kebasaaan total tertinggi. Karakterisasi katalis meliputi FTIR, XRD, uji keasaman dengan metode gravimetri, uji kebasaaan dengan metode titrasi, SEM yang dikombinasikan dengan EDX, dan *surface area analyzer*. Hasil transesterifikasi dikarakterisasi menggunakan FTIR, GC-MS, dan $^1\text{H-NMR}$.

Hasil karakterisasi menunjukkan katalis SO_4/ZrO_2 dengan konsentrasi 0,9 M H_2SO_4 dan temperatur kalsinasi 500 °C memiliki keasaman total tertinggi, yakni sebesar 1,9796 mmol g^{-1} . Katalis Zr/CaO memiliki kebasaaan total tertinggi sebesar 27,78 mmol g^{-1} pada konsentrasi 1% Zr terhadap Zr/CaO dan temperatur kalsinasi 900 °C. Berat molekul minyak kelapa bekas sebesar 787,40 g mol^{-1} . Pada tahap esterifikasi, katalis 0,9 M SO_4/ZrO_2 mampu menurunkan *Free Fatty Acid* (FFA) minyak goreng bekas dari 1,18% menjadi 0,42% pada kondisi 5% berat katalis, rasio mol minyak:metanol 1:15, temperatur reaksi 65 °C selama 3 jam. Katalis Zr/CaO yang digunakan pada tahap transesterifikasi berhasil mengkonversi minyak goreng bekas menjadi biodiesel sebesar 62,25%. Terbentuknya biodiesel dikonfirmasi melalui adanya puncak metil laurat (50,48%), metil miristat (19,05%), metil stearat (11,05%), metil 11-oktadekanoat (6,09%), metil oktanoat (5,25%), metil dekanooat (5,05), dan metil oktadekanoat (3,03%).

Kata kunci: katalis SO_4/ZrO_2 , katalis Zr/CaO , biodiesel, minyak kelapa bekas

SYNTHESIS AND CHARACTERIZATION OF THE SO_4/ZrO_2 AND Zr/CaO CATALYSTS AND THEIR APPLICATION FOR BIODIESEL PRODUCTION FROM USED COCONUT OIL

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ABSTRACT

The SO_4/ZrO_2 and Zr/CaO catalysts had been synthesized and applied to convert used cooking oil become biodiesel throught two stage of reaction, esterification and transesterification. The SO_4/ZrO_2 acid catalysts was synthesized using wet impregnation method with variation of concentration 0.5; 0.7; and 0.9 M H_2SO_4 and calcination temperature of 400, 500, 600, and 700 °C to obtain the highest total acidity catalyst. The Zr/CaO base catalysts was synthesized by reflux method using microwave heating and was optimized with variation of weight percent of 1%, 5% and 10% Zr/CaO and calcination temperature of 600, 700, 800 and 900 °C to obtain the highest basicity catalyst. Characterization of catalyst included FTIR, XRD, gravimetric method to determine total acidity, titration method to determine total basicity, SEM combined with EDX, and surface area analyzer. Characterization of transesterification product using FTIR, GC-MS, and $^1\text{H-NMR}$.

The result of catalyst characterization showed that SO_4/ZrO_2 catalyst with a concentration of 0.9 M H_2SO_4 and calcination temperature of 500 °C had the highest total acidity of 1.9796 mmol g^{-1} . The Zr/CaO had the total basicity of 27.78 mmol g^{-1} at a concentratiof of weight percent 1% and calcination temperature 900 °C. Weight molecular of used coconut oil 787,40 g mol^{-1} . In esterification reaction, the 0.9 M SO_4/ZrO_2 catalyst could decrease the Free Fatty Acid (FFA) content of used cooking oil from 1.18% to 0.42% under condition of 5% catalyst weight, 1:15 mol ratio of used cooking oil:methanol, 65 °C reaction temperature for 3 hours of reaction. In transesterification, the 1% Zr/CaO -900 catalyst succeeded in converting used cooking oil became biodiesel by 62.25%. The formation of biodiesel was confirmed by the presence of methyl laurate (50.48%), methyl myristate (19.05%), methyl stearate (11.05%), methyl 11-octadecanoic (6.09%), methyl octanoate (5.25%), methyl deanate (5.05), and methyl octadecanoate (3.03%).

Keywords: SO_4/ZrO_2 catalyst, Zr/CaO catalyst, biodiesel, used coconut oil