

## KATALIS HETEROGEN ASAM $\text{SO}_4/\text{ZrO}_2$ DAN BASA $\text{Zr}/\text{CaO}$ PADA PEMBUATAN BIODIESEL DARI MINYAK GORENG KELAPA BEKAS

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### INTISARI

Sintesis katalis  $\text{SO}_4/\text{ZrO}_2$  dan  $\text{Zr}/\text{CaO}$  serta aplikasinya pada proses pembuatan biodiesel dari minyak goreng kelapa bekas telah dilakukan melalui reaksi esterifikasi dilanjutkan dengan transesterifikasi. Proses sulfatasi  $\text{ZrO}_2$  dioptimasi pada temperatur ruang dengan konsentrasi  $\text{H}_2\text{SO}_4$  0,3, 0,6, 0,9 M dan temperatur kalsinasi 400, 500, 600, dan 700 °C untuk memperoleh kasaman tertinggi. Katalis ZCa disintesis secara refluks menggunakan CaO dan prekursor  $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ . Optimasi dilakukan dengan konsentrasi berat Zr terhadap berat total Zr-CaO sebesar 1, 5, 10, 15% (b/b) dan temperatur kalsinasi 600, 700, 800, dan 900 °C untuk memperoleh kebasaaan tertinggi. Karakterisasi katalis dilakukan menggunakan FTIR, uji keasaman dengan metode gravimetri, uji kebasaaan dengan metode titrasi, XRD, SAA, dan SEM-EDX. Katalis SZ digunakan pada reaksi esterifikasi dengan variasi berat katalis 1, 3, dan 5%, rasio mol minyak:metanol 1:5, 1:10, dan 1:15, serta waktu reaksi 0,5, 1,0, dan 1,5 jam. Katalis ZCa dengan kebasaaan optimum digunakan pada reaksi transesterifikasi untuk memperoleh biodiesel. Karakterisasi biodiesel dilakukan dengan FTIR, GC-MS, dan  $^1\text{H-NMR}$ .

Hasil analisis menunjukkan bahwa SZ-500 memiliki keasaman tertinggi yaitu  $3,60 \text{ mmol g}^{-1}$  dan ZCa-800 memiliki kebasaaan tertinggi sebesar  $27,78 \text{ mmol g}^{-1}$ . Reaksi esterifikasi dengan variasi berat katalis 5%, rasio mol minyak:metanol 1:15, dan waktu reaksi 1,5 jam dapat menurunkan kadar FFA hingga 64,29% yakni dari 1,17% menjadi 0,42%. Analisis FTIR produk transesterifikasi menunjukkan adanya puncak pada bilangan gelombang  $1033,85 \text{ cm}^{-1}$  yang merujuk pada vibrasi ulur simetri C–O–C khas dari biodiesel. Analisis menggunakan  $^1\text{H-NMR}$  menunjukkan bahwa minyak goreng kelapa bekas berhasil terkonversi menjadi biodiesel sebesar 55,35%. Analisis GC-MS menunjukkan bahwa biodiesel yang diperoleh adalah metil ester dengan komponen yang terdiri dari metil laurat (48,41%), metil miristat (18,55%), metil palmitat (10,82%), metil oktanoat (7,19%), metil oleat (5,71%), metil dekanat (5,56%), metil stearat (3,28%), dan metil palmilinoat (0,47%).

Kata kunci: katalis  $\text{SO}_4/\text{ZrO}_2$ , katalis  $\text{Zr}/\text{CaO}$ , biodiesel, esterifikasi, transesterifikasi

***THE HETEROGENEOUS CATALYST OF ACID  $\text{SO}_4/\text{ZrO}_2$  AND BASE  $\text{Zr}/\text{CaO}$  FOR BIODIESEL PRODUCTION FROM USED COCONUT COOKING OIL***

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***ABSTRACT***

Synthesis of  $\text{SO}_4/\text{ZrO}_2$  and  $\text{Zr}/\text{CaO}$  catalysts and their applications in the process of making biodiesel from used coconut cooking oil have been carried out through the esterification followed by transesterification reaction. The sulfatation process of  $\text{ZrO}_2$  was optimized at room temperature with concentration of  $\text{H}_2\text{SO}_4$  0.3, 0.6, 0.9 M and calcination temperatures of 400, 500, 600 and 700 °C to obtain the highest acidity. The ZCa catalyst is synthesized by reflux method using CaO and  $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$  precursor. Optimization is done by weight % Zr to total weight of Zr-CaO of 1, 5, 10, 15% (w/w) and calcination temperatures of 600, 700, 800, and 900 °C to obtain the highest basicity. Catalyst characterization was completed using FTIR, acidity test by gravimetric method, basicity test by titration method, XRD, SAA, and SEM-EDX. The SZ catalyst then was used in the esterification reaction with variations in weight of catalysts that is 1, 3, and 5%, molar ratio of 1: 5, 1:10, and 1:15, and reaction time of 0.5, 1.0, and 1.5 hr. While the ZCa catalyst with optimum basicity was used in the transesterification reaction to obtain biodiesel. Biodiesel characterization was performed by FTIR, GC-MS, and  $^1\text{H-NMR}$ .

The analysis showed that 0.9 SZ-500 had the highest acidity of 3.60 mmol  $\text{g}^{-1}$  and ZCa-800 had the highest basicity of 27.78 mmol  $\text{g}^{-1}$ . Esterification reaction with catalyst of 5% (w/w), molar ratio of 1:15, and reaction time of 1.5 h could reduce FFA level up to 64.29% from 1.17% to 0.42%. While the FTIR analysis on the results of transesterification shows the absorption of typical biodiesel bands at wave number 1033.85  $\text{cm}^{-1}$ , which is a vibration of C–O–C. Analysis using  $^1\text{H-NMR}$  confirmed that used coconut cooking oil was successfully converted into biodiesel by 55.35%. GC-MS analysis suggested that the obtained biodiesel was methyl ester with components of methyl laurate (48.41%), methyl myristate (18.55%), methyl palmitate (10.82%), methyl octanoate (7.19%), methyl oleate (5.71%), methyl decanoate (5.56%), methyl stearate (3.28%), and methyl palmilinoleate (0.47%).

Keywords:  $\text{SO}_4/\text{ZrO}_2$  catalyst,  $\text{Zr}/\text{CaO}$  catalyst, biodiesel, esterification, transesterification