SINTESIS KATALIS BASA CaO DARI LIMBAH CANGKANG KERANG DARA (*Anadara granosa*) TERAKTIVASI ASAM SITRAT UNTUK KONVERSI SATU LANGKAH (*ONE-POT*) MINYAK SAWIT BEKAS MENJADI BIODIESEL

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INTISARI

Penelitian ini bertujuan untuk menentukan aktivitas katalis bifungsional terbaik dari katalis basa CaO teraktivasi asam dalam reaksi satu langkah (esterifikasi-transesterifikasi), mempelajari pengaruh variasi massa katalis, rasio mol minyak:metanol, temperatur dan waktu reaksi, terhadap jumlah biodiesel yang dihasilkan menggunakan katalis terbaik, dan uji *reusability* katalis bifungsional terbaik pada kondisi reaksi optimum esterifikasi-transesterifikasi minyak sawit bekas. Katalis basa (CKS) disintesis melalui kalsinasi cangkang kerang dara pada temperatur 900 °C, kemudian diaktivasi dengan asam H_2SO_4, H_3PO_4, $C_6H_8O_7$ berturut-turut menghasilkan katalis bifungsional CKS–SO_4, CKS–PO_4, dan CKS–CA. Katalis yang telah disintesis diuji aktivitasnya melalui reaksi satu langkah minyak sawit bekas untuk memperoleh katalis bifungsional terbaik. Dilakukan variasi kondisi reaksi (massa katalis terhadap umpan, rasio mol minyak:metanol, temperatur, dan waktu reaksi) untuk menentukan kondisi optimum dan uji *reusability* untuk menentukan stabilitas katalis. Karakterisasi katalis dilakukan dengan XRF, FTIR, XRD, NH_3-TPD, CO_2-TPD, SAA, dan SEM-EDX. Produk cair dianalisis menggunakan GC-MS, FTIR, ^{1}H NMR, dan ^{13}C NMR.

Hasil penelitian menunjukkan terjadi peningkatan luas permukaan katalis CKS–CA (8,15 m^2/g) dibandingkan katalis CKS (2,89 m^2/g). Hasil SEM-EDX juga menunjukkan struktur CKS–CA membentuk agregat dan pori-pori yang lebih banyak. Katalis CKS–CA memiliki total keasaman dan kebasaan tertinggi, yaitu 11,62 dan 3,38 g/mol. Katalis CKS–CA memperoleh persen hasil biodiesel optimum 80,10% dengan selektivitas 100% pada kondisi reaksi 10% massa katalis, temperatur reaksi 65 °C, rasio mol minyak:metanol 1:15, dan waktu reaksi 5 jam. Analisis GC-MS menunjukkan kandungan utama dari biodiesel adalah metil palmitat dan metil oleat. Analisis FTIR produk biodiesel menunjukkan puncak khas pada bilangan gelombang 1195 dan 1033 cm^{-1} sebagai vibrasi ulur gugus metoksi (–OCH_3) dan vibrasi asimetri (–OCH_2–C). Analisis ^{1}H NMR menunjukkan pergeseran kimia pada 3,65 ppm dan ^{13}C NMR pada 51,42 ppm yang merupakan karakteristik metil ester. Hal ini membuktikan bahwa biodiesel telah berhasil diproduksi. Uji *reusability* katalis CKS–CA setelah penggunaan empat kali menghasilkan biodiesel sebesar 42,74% dengan selektivitas 99,71%.

Kata kunci: biodiesel, cangkang kerang dara, minyak sawit bekas, katalis bifungsional, esterifikasi-transesterifikasi

SYNTHESIS OF CaO BASE CATALYST FROM BLOOD COCKLE SHELL WASTE (*Anadara Granosa*) ACTIVATED BY CITRIC ACID FOR ONE-POT CONVERSION OF WASTE PALM OIL INTO BIODIESEL

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ABSTRACT

This study aimed to determine the best bifunctional catalyst activity of acid-activated base catalyst (CaO) in a one-pot esterification–transesterification reaction, to investigate the effect of catalyst weight, oil-to-methanol molar ratio, reaction temperature, and reaction time on biodiesel yield using the best catalyst, and to evaluate the reusability of the best bifunctional catalyst under the optimum reaction conditions for esterification–transesterification of used palm oil. The base catalyst (CKS) was prepared by calcining blood clam shells at 900 °C and then activated with H_2SO_4, H_3PO_4, and $C_6H_8O_7$ to produce CKS–SO_4, CKS–PO_4, and CKS–CA. These catalysts were tested in a one-pot reaction using used palm oil to determine the best performer. Reaction parameters were varied to find optimum conditions, and reusability tests were conducted to assess catalyst stability. Catalysts were characterized by XRF, FTIR, XRD, NH_3-TPD, CO_2-TPD, SAA, and SEM-EDX. The biodiesel products were analyzed using GC-MS, FTIR, ^{1}H NMR, and ^{13}C NMR.

The results of the study showed an increase in the surface area of the CKS–CA catalyst (8.15 m^2/g) compared to the CKS catalyst (2.89 m^2/g). SEM-EDX analysis also revealed that the CKS–CA catalyst had a more aggregated structure and a higher number of pores. CKS–CA exhibited the highest total acidity and basicity, at 11.62 and 3.38 g/mol, respectively. The CKS–CA catalyst achieved an optimum biodiesel yield of 80.10% with 100% selectivity under the reaction conditions of 10% catalyst weight, reaction temperature of 65 °C, oil-to-methanol molar ratio of 1:15, and reaction time of 5 hours. GC-MS analysis indicated that the main components of the biodiesel were methyl palmitate and methyl oleate. FTIR analysis of the biodiesel product showed characteristic peaks at 1195 and 1033 cm^{-1} corresponding to the stretching vibrations of methoxy (–OCH_3) and asymmetric vibrations of (–OCH_2–C) groups. ^{1}H NMR analysis showed a chemical shift at 3.65 ppm, and ^{13}C NMR at 51.42 ppm, both characteristic of methyl esters. These results confirmed the successful production of biodiesel. The reusability test of the CKS–CA catalyst after four cycles showed a biodiesel yield of 42.74% with 99.71% selectivity.

Keyword: bifunctional catalyst, biodiesel, blood clam shell, esterification-transesterification, used palm oil