

Sintesis dan Karakterisasi Katalis Bimetalik NiFe/H-FDU-12 dan Optimasi Kondisi Reaksi Konversi Asam Levulinat Menjadi Gamma-Valerolactone dengan Metode Box-Behnken Design = Synthesis and Characterization of NiFe/H-FDU-12 Bimetallic Catalyst and Condition Optimization of Conversion of Levulinic Acid to Gamma-Valerolactone Reaction Using the Box-Behnken Design Method

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Abstrak

Turunan biomassa lignoselulosa dapat diubah menjadi bahan bakar dan berpotensi menjadi sumber bahan bakar alternatif. Asam levulinat (AL) telah diidentifikasi sebagai salah satu turunan biomassa yang bernilai tinggi karena sifatnya yang reaktif dan dapat dengan mudah dan ekonomis dihasilkan dari limbah lignoselulosa. AL dapat diubah menjadi gamma (-) valerolactone (GVL), salah satu bahan kimia yang penting dan prekursor untuk biofuel. Logam bimetalik NiFe diimpregnasi dengan persen loading logam sebesar 5% menggunakan metode impregnasi basah. Perbandingan berat Ni terhadap Fe yang diimpregnasi ditentukan dengan perhitungan kemometrik Box-Behnken Design (BBD) yaitu sebesar 1:4, 2.5:2.5 dan 4:1. Katalis NiFe/H-FDU-12 hasil sintesis dikarakterisasi menggunakan metode karakterisasi zat padat seperti FTIR, XRD, XRF, SAXS, SEM, dan BET SAA. Katalis kemudian diuji aktivitas katalitiknya dalam reaksi siklisasi hidrogenatif asam levulinat dengan metanol sebagai donor H⁺. Variasi perbandingan berat Ni:Fe, suhu, dan waktu reaksi dilakukan sesuai dengan desain eksperimen BBD untuk penentuan kondisi optimum. Hasil reaksi uji aktivitas katalitik untuk konversi asam levulinat (AL) menjadi gamma-valerolactone (GVL) dengan yield GVL tertinggi sebesar 87.52% dihasilkan menggunakan katalis Ni₁Fe₄/H-FDU-12 dengan suhu reaksi 180 C selama 2 jam. Metode Box-Behnken digunakan untuk melihat pengaruh variasi perbandingan berat Ni:Fe, suhu, dan waktu terhadap reaksi konversi AL menjadi GVL. Dengan model koefisien linear, ditentukanlah bahwa suhu memiliki pengaruh terbesar dibandingkan faktor lainnya. Dengan mengoptimalkan faktor dalam rentang masing-masing, konversi AL sekitar 98.83%, yield GVL 77.17% dan selektivitas 81.95% dicapai pada kondisi spesifik: ratio Ni:Fe 1:4, suhu reaksi 180 °C, waktu reaksi 3 jam.

.....Lignocellulosic biomass derivatives can be converted into fuel and have the potential to become alternative fuel sources. Levulinic acid (LA) has been identified as one of the high value biomass derivatives due to its reactive nature and can be easily and economically produced from lignocellulosic waste. LA can be converted into gamma (-) valerolactone (GVL), an important chemical and a precursor for biofuel. The bimetallic NiFe metal was impregnated with a metal loading percentage of 5% using wet impregnation method. The weight ratio of Ni to Fe impregnation was determined using Box-Behnken Design (BBD) chemometric calculations, resulting in ratios of 1:4, 2.5:2.5, and 4:1. The synthesized NiFe/HFDU- 12 catalysts were characterized using solid-state characterization methods such as FTIR, XRD, XRF, SAXS, SEM, and BET SAA. The catalyst was then tested for its catalytic activity in the hydrogenative cyclization reaction of levulinic acid with methanol as the H⁺ donor. Variations in the Ni:Fe weight ratio, temperature, and reaction time were conducted according to the BBD experimental design to determine the optimum condition. The results of the catalytic activity test showed that the highest yield of gamma-valerolactone (GVL), reaching 87.52%, was obtained using the Ni₁Fe₄/H-FDU-12 catalyst at a reaction temperature of

180 °C for 2 hours. The Box-Behnken method was used to assess the influence of variations in the Ni:Fe weight ratio, temperature, and reaction time on the conversion of LA to GVL. Through the linear coefficient model, it was determined that temperature had the greatest influence compared to other factors. By optimizing the factors within their respective ranges, a conversion of approximately 98.83% for LA, a GVL yield of 77.17%, and a selectivity of 81.95% were achieved under specific conditions: Ni:Fe ratio of 1:4, reaction temperature of 180°C, and reaction time of 3 hours.