SYNTHESIS AND CATALYTIC EVALUATION OF NICKEL SUPPORTED ON FIBROUS ZEOLITE-Y FOR CO_2 REFORMING OF CH_4

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MASTER OF SCIENCE

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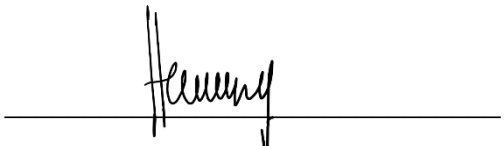

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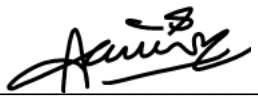

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ABSTRAK

Pembaharuan metana kering (CRM) telah dianggap sebagai pendekatan yang menjanjikan untuk mengurangkan pelepasan gas rumah hijau, CO_2 dan CH_4 sementara menghasilkan syngas berharga. Tindak balas CRM telah dikaji secara meluas menggunakan pelbagai pemangkin logam yang disokong, termasuk sokongan Ni/zeolite-Y (Ni/HY). Walaubagaimanapun, Ni/HY cepat dinyahaktif disebabkan pemendapan karbon dan pensinteran logam yang timbul dari interaksi sokongan logam yang lemah. Oleh itu, kajian ini memberi tumpuan kepada mengsintesis dan mencirikan pemangkin zeolite-Y berserabut yang diubah suai (FHY). Seterusnya, mengkaji aktiviti pemangkin Ni/FHY yang disintesis menggunakan CRM. Pemangkin Ni/FHY disediakan dengan zeolite-Y (HY) yang diubah suai menjadi zeolite-Y berserabut (FHY) menggunakan kaedah sistem mikroemulsi bersama penghabluran biji kristal HY, diikuti dengan impregnasi nikel kepada FHY dan HY. Pencirian Ni/FHY ditentukan menggunakan XRD, FTIR, BET, TEM, FESEM-EDX, CO_2-TPD dan H_2-TPR. Pada masa yang sama, penilaian CRM dijalankan menggunakan reaktor keluli tahan karat pada suhu 700 ^oC, halalaju ruang setiap jam 25,000 mL/g.h, dan nisbah CH_4: CO_2 = 1:1. Keputusan menunjukkan bahawa Ni/FHY ($X_{CH_4} = 90.0\%, X_{CO_2} = 85.9\%, H_2/CO = 0.85$) mempunyai prestasi yang lebih tinggi di suhu 700 ^oC berbanding Ni/HY ($X_{CH_4} = 85.9\%, X_{CO_2} = 84.9\%, H_2/CO = 0.73$) kerana ciri FHY yang menarik membenarkan taburan Ni yang lebih baik dengan saiz kristal Ni yang lebih kecil, sekali gus mengukuhkan interaksi sokongan logam. Kesan pemuatan Ni (1, 3, 5, dan 10 wt.%) menunjukkan bahawa 5Ni/FHY mempunyai prestasi pemangkin yang terbaik. Prestasi 5Ni/FHY yang baik adalah kerana interaksi kuat antara Si-O-Ni, saiz sederhana kristal NiO (9.35 nm), dan pengedaran sekata logam aktif, yang membawa kepada kesan sinergi yang kuat antara tapak aktif logam Ni dan FHY. Tambahan pula, keadaan operasi Ni/FHY terhadap CRM telah dioptimumkan menggunakan metodologi permukaan tindak balas (RSM) dibawah pembolehubah bebas yaitu suhu tindak balas (X_1, 700-900 ^oC), halaju ruang setiap jam gas (X_2, 15,000-35,000 mL/gh), dan nisbah CH_4/CO_2 (X_3, 1-3) dengan pembolehubah tindak balas penukaran CH_4 (Y_1), penukaran CO_2 (Y_2), dan nisbah H_2/CO (Y_3). Keadaan tindak balas optimum didapati pada X_1=833 ^oC, X_2= 24,968 mL/g.h, dan X_3=2.08 dengan Y_1 = 94.5%, Y_2 = 88.3%, dan Y_3 = 1.02. Kajian ini juga membuktikan bahawa mangkin Ni/FHY yang disintesis cukup stabil untuk 90 jam masa dalam aliran dengan prestasi $X_{CH_4} = 93.5\%, X_{CO_2} = 89.1\%$ dan boleh dijana semula oleh udara. Ringkasnya, Ni/FHY telah berjaya disintesis dan muncul sebagai pemangkin kemampanan tinggi untuk pengeluaran syngas yang cekap melalui CRM di bawah keadaan optimum yang dihasilkan daripada RSM. Pemangkin 5Ni/FHY menunjukkan prestasi terbaik dengan kestabilan tinggi di bawah keadaan optimum yang dihasilkan oleh RSM dan membuktikan keupayaannya untuk dijana semula melalui udara.

ABSTRACT

CO_2 reforming CH_4 (CRM) has been a promising approach to mitigate the CO_2 and CH_4 greenhouse gas emissions while evolving valuable syngas. The CRM reaction has been investigated extensively using a variety of supported metal catalysts, including Ni/zeolite-Y (Ni/HY). However, Ni/HY rapidly deactivated due to the carbon deposition and metal sintering, which arose from the weak metal-support interaction. Therefore, this study synthesizes and characterizes the modified fibrous zeolite-Y catalyst (FHY). Next, to examine the catalytic activity of synthesized FHY catalysts toward CRM. Ni/FHY catalysts were prepared by modifying HY into FHY using a microemulsion system coupled with the HY crystal-seed crystallization method, followed by the impregnation of Ni to the synthesized FHY and commercial HY. XRD, FTIR, BET, TEM, FESEM-EDX, CO_2-TPD, and H_2-TPR determined the properties of Ni/FHY. At the same time, CRM evaluation was conducted using a stainless steel fixed-bed reactor at a reaction temperature of 700 ^oC, gas hourly space velocity of 25,000 mL/g.h, and CH_4/CO_2 ratio of 1. The results showed that Ni/FHY ($X_{CH_4} = 90.0\%, X_{CO_2} = 85.9\%, H_2/CO = 0.85$) possessed higher performance at a temperature of 700 ^oC compared to Ni/HY ($X_{CH_4} = 85.9\%, X_{CO_2} = 84.9\%, H_2/CO = 0.73$) attributed to the fascinating characteristics of FHY that allowed a homogenous and better Ni distribution with a smaller Ni crystallite size, thus strengthening the metal-support interaction. The effect of Ni loadings (1, 3, 5, and 10 wt.%) showed that 5Ni/FHY had the best catalytic performance. The excellent performance of 5Ni/FHY because of the strong interaction between Si-O-Ni, the moderate size of the NiO crystallites (9.35 nm), and the even distribution of the active metals led to a strong synergistic effect between the Ni metal active sites and FHY. Moreover, the operating conditions of Ni/FHY over CRM were optimized using response surface methodology (RSM) under independent variables of reaction temperature (X_1, 700-900 ^oC), gas hourly space velocity (X_2, 15,000-35,000 mL/g.h), and CH_4/CO_2 ratio (X_3, 1-3), with response variables of CH_4 conversion (Y_1), CO_2 conversion (Y_2), and H_2/CO ratio (Y_3). The optimal reaction conditions were found at X_1 = 833 ^oC, X_2 = 24,968 mL/g.h, and X_3 = 2.08, with Y_1 = 94.5%, Y_2 = 88.3%, and Y_3 = 1.02. This study also proved that the synthesized Ni/FHY catalyst was appreciably stable for 90 h time-on-stream with the performance of $X_{CH_4} = 93.5\%, X_{CO_2} = 89.1\%$ and could be regenerated by air. In summary, Ni/FHY was successfully synthesized and emerged as a high sustainability catalyst for efficient syngas production via CRM under optimal conditions generated from RSM. The 5Ni/FHY catalyst performed the best with high stability under optimum conditions generated by RSM and proved its ability to be regenerated by air.

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