LANTHANUM OXIDE-PROMOTED COBALT CATALYST SUPPORTED ON MESOPOROUS ALUMINA FOR SYNGAS PRODUCTION VIA METHANE DRY REFORMING

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DOCTOR OF PHILOSOPHY

UNIVERSITI MALAYSIA PAHANG

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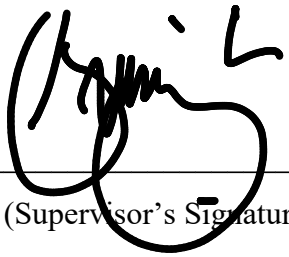
We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.



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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Tindak balas pembaharuan kering metana (MDR) baru-baru ini muncul sebagai antara pendekatan pelbagai guna yang terbaik untuk menukar dua gas rumah hijau, karbon dioksida (CO_2) dan metana (CH_4), kepada bahan mentah yang berharga untuk proses hiliran petrokimia. Pada masa ini, masih terdapat cabaran dalam membangunkan pemangkin yang sangat stabil dan aktif untuk tindak balas MDR di samping rintangan yang lebih baik terhadap pemendapan karbon. Baru0baru ini, pemangkin berasaskan kobalt yang disokong mesopori alumina muncul sebagai pemangkin yang berpotensi. Walau bagaimanapun, bahan-bahan konvensional yang digunakan untuk menyediakan sokongan pemangkin mesopori ini ialah prekursor organik dan etanol yang agak mahal dan berbahaya kepada alam sekitar. Oleh itu, dalam kajian ini, penggunaan mesopori alumina (Al_2O_3), yang direka menggunakan prekursor aluminium bukan organik yang murah dan tersedia dalam pelarut binari etanol-air, telah dikaji sebagai sokongan untuk pemangkin kobalt. Penyiasatan ini bertujuan untuk mereka bentuk sistem pemangkin berasaskan kobalt yang berkesan untuk tindak balas MDR, yang mengatasi halangan penyahaktifan yang disebabkan oleh karbon. Kesan promosi La_2O_3 pada ciri fizikokimia pemangkin kobalt yang disokong Al_2O_3 dan prestasi pemangkinnya juga telah dijelaskan. Penilaian mangkin dalam tindak balas MDR telah dijalankan untuk mangkin 10%Co/Al_2O_3 dan 10%Co/Al_2O_3 yang digalakkan La_2O_3 (pemuatan La adalah dalam 1% – 8%) dalam reaktor katil tetap pada julat suhu 923 – 1073 K dan tekanan separa bagi bahan tindak balas dari 10 hingga 40 kPa. Sokongan Al_2O_3 mempunyai luas permukaan BET 173.4 m^2 g^{-1} dan nanopartikel kobalt tersebar dengan halus diatas sokongan dengan saiz kristal yang dikehendaki berjulat dari 5.2 - 9.2 nm. Interaksi kuat antara CoO dan Al_2O_3 telah disahkan dengan kehadiran spinel kobalt-aluminat dan struktur tekstur pemangkin adalah stabil terhadap suhu tindak balas. Tingkah laku promosi La_2O_3 memudahkan pengurangan H_2 dengan menyediakan ketumpatan elektron yang lebih tinggi dan meningkatkan kekosongan oksigen dalam 10%Co/Al_2O_3. Penambahan La_2O_3 boleh mengurangkan tenaga pengaktifan ketara bagi penggunaan CH_4; lalu, meningkatkan penukaran CH_4 sehingga 93.7% pada 1073 K. Pembentukan lanthanum dioksikarbonat secara terus semasa MDR bertanggungjawab dalam pengurangan karbon termendap melalui kitaran redoks sebanyak 17-30% bergantung pada suhu tindak balas. Selain itu, tahap kekosongan oksigen meningkat kepada 73.3% dengan promosi La_2O_3. Pemuatan 5%La ialah kandungan penggalak yang optimum untuk penukaran bahan tindak balas serta penghasilan H_2 dan CO. 5%La-10%Co/Al_2O_3 juga mempamerkan rintangan tertinggi terhadap pemendapan karbon kerana sifat asas, ciri redoks penggalak La_2O_3. Tindak balas MDR ke atas pemangkin 5%La-10%Co/Al_2O_3 telah diyakini mengikuti mod penjerapan bersekutu CH_4 dan CO_2 pada dwi tapak zarah aktif atau berbeza dan pemangkin menunjukkan kestabilan yang baik semasa tindak balas 48 jam pada 1023 K. Nisbah H_2/CO 0.84-0.98 yang terhasil adalah sesuai untuk tindak balas Fischer-Tropsch di hiliran untuk menjana bahan api hidrokarbon cecair. Akibatnya, penggunaan sokongan mesopori alumina dan penggalakk La_2O_3 meningkatkan aktiviti Co dengan efektif dalam tindak balas MDR disamping menahan pemendapan karbon pada permukaan pemangkin.

ABSTRACT

Methane dry reforming reaction (MDR) has recently emerged as a promising multipurpose approach for converting two greenhouse gasses, included carbon dioxide (CO_2) and methane (CH_4), into valuable feedstock for downstream petrochemical processes. At present, there is still a challenge in developing the highly stable and active catalysts for MDR reaction as well as better resistance to carbon deposition. Though the mesoporous alumina supported Co-based catalysts have recently appeared to be the potential catalysts. However, the common starting materials for preparing these well-ordered mesoporous catalyst supports are organic precursors and anhydrous ethanol which are quite expensive and harmful to the environment. Therefore, in this study, mesoporous alumina (Al_2O_3), fabricated using a cheap and available inorganic aluminium precursor in binary water-ethanol solvent, was implemented as support for cobalt catalyst. This investigation aimed to design an effective cobalt-based catalyst system for MDR reaction, which overcomes coke-related deactivation barriers. The promotional effect of La_2O_3 on the physicochemical features of Al_2O_3 supported cobalt catalyst and its catalytic performance were also elucidated. The catalyst evaluations in MDR reaction were conducted for 10%Co/Al_2O_3 and La_2O_3-promoted 10%Co/Al_2O_3 catalysts (La loading was in 1% – 8%) in a fixed-bed reactor at temperature range of 923 – 1073 K and partial pressure of individual reactant from 10 to 40 kPa. The Al_2O_3 support has BET surface area of 173.4 m^2 g^{-1} and cobalt nanoparticles were finely dispersed on the support with desired crystallite size ranged from 5.2 - 9.2 nm. The strong interaction of CoO and Al_2O_3 phases was confirmed by the presence of cobalt-aluminate spinel and the textural structure of catalysts was stable with reaction temperature. The promotion behavior of La_2O_3 facilitated H_2-reduction by providing higher electron density and enhanced oxygen vacancy in 10%Co/Al_2O_3. The addition of La_2O_3 could reduce the apparent activation energy of CH_4 consumption; hence, increasing CH_4 conversion up to 93.7% at 1073 K. Lanthanum dioxycarbonate transitional phase formed *in situ* during MDR was accountable for mitigating deposited carbon via redox cycle for 17-30% relying on reaction temperature. Additionally, the oxygen vacancy degree increased to 73.3% with La_2O_3 promotion. 5%La loading was an optimal promoter content for reactant conversions as well as yield of H_2 and CO. 5%La-10%Co/Al_2O_3 also exhibited the highest resistance to carbon deposition owing to the basic nature, redox feature of La_2O_3 dopant. The MDR reaction over 5%La-10%Co/Al_2O_3 catalyst was convinced to follow an associative adsorption mode of CH_4 and CO_2 on dual or different sites of active particles and the catalyst exhibited a good stability during 48 h reaction at 1023 K. The resulting H_2/CO ratios of 0.84-0.98 are suitable for Fischer-Tropsch reaction in downstream to generate liquid hydrocarbon fuels. As a result, the employment of mesoporous alumina support and La_2O_3 promoter efficiently boosted the Co activity in MDR reaction along with suppressing the carbon deposition on the catalyst surface.

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