PRODUCTION OF HYDROGEN BY PHOTOREFORMING OF FORMALDEHYDE CONTAINING WASTEWATER OVER CdO–CuO@exfoliated g–C_3N_4 NANOHETEROJUNCTION

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DOCTOR OF PHILOSOPHY

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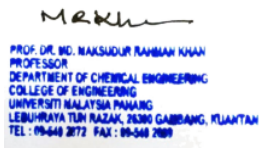

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ABSTRAK

Pada masa ini, pertambahan penduduk dan pembangunan perindustrian menimbulkan dilema besar selaras dengan peningkatan permintaan tenaga global dan isu–isu alam sekitar. Untuk mengatasi cabaran semasa pelupusan bahan bakar fosil dan pencemaran air yang terus meningkat, usaha intensif telah dilakukan untuk meneroka jalan lestari bagi mengatasi masalah ini. Dalam konteks ini, fotokatalisis sangat menjanjikan kerana ia mengunakan sumber tenaga suria yang boleh diperbaharui secara meluas dan menghasilkan hidrogen. Oleh hal yang demikian, pembaharuan fotokatalitik atau pembaharuan fotoreformasi air sisa petrokimia menawarkan faedah yang lebih ganda untuk pengeluaran tenaga hijau dan pemulihan air sisa. Penyelidikan semasa bertujuan untuk mensintesis fotokatalis berasaskan polimerik karbon nitrida grafit (g–C_3N_4) sebagai pemangkin dan diselaraskan dengan pengelupasan dan didoping dengan oksida logam seperti CdO dan CuO. Kemudian, novel CdO–CuO@exfoliated nanoheterojunction telah disintesis melalui teknik impregnasi yang mudah. Daripada hasil XRD, pengubahan g–C_3N_4 (27.90°) ke puncak yang lebih rendah dalam sampel exfoliated g–C_3N_4 (27.15°) mewujudkan lebih banyak ruang antara lapisan planar dengan itu mendedahkan pengubahan bentuk planar triazin dan berjaya menghasilkan lembaran nano. Lebih–lebih lagi, hasil BET menunjukkan peningkatan besar luas permukaan exfoliated g–C_3N_4 (104.4 m^2/g) dan CdO–CuO@exfoliated g–C_3N_4 (388.4 m^2/g) masing–masing sebanyak 3 dan 11 kali berbanding dengan g–C_3N_4 (34.5 m^2/g). Selepas itu, fotokatalis yang telah disintesis diuji untuk aktiviti fotoreformasi hidrogen dalam medium formaldehid dan disinari di bawah cahaya LED. Sebagai hasilnya, trend aktiviti hidrogen berada dalam urutan g–C_3N_4<exfoliated g–C_3N_4<CdO@exfoliated g–C_3N_4< CuO@exfoliated g–C_3N_4<CdO–CuO@exfoliated g–C_3N_4. Aktiviti nanoheterojunction yang sangat baik disumbangkan oleh kawasan permukaan yang besar menyebabkan banyak tapak aktif tersedia untuk reaksi redoks. Lebih–lebih lagi, pengurangan intensiti puncak secara besar–besaran dalam analisis PL menghalang kadar pengumpulan semula pasangan elektron–hole, mendorong pemisahan dan migrasi cas mudah alih, menyebabkan peningkatan pengeluaran hidrogen. Selepas itu, CdO–CuO @ exfoliated g–C_3N_4 diperiksa lebih lanjut untuk mengoptimumkan parameter dan mengkaji korelasi antara faktor yang berbeza. Berdasarkan ANOVA, model ini sesuai dengan nilai–F (54.97) dan nilai–p (<0.0001) yang kurang dari 0.05. Selain itu, interaksi antara parameter yang berbeza disiasat dan ditunjukkan melalui kontur dan plot tiga dimensi. Hidrogen maksimum yang dihasilkan adalah 9025 µmol/g untuk keadaan berikut: masa tindak balas 8 jam, dos pemangkin 0.73 g/L, kepekatan awal FA–573 ppm dan intensiti cahaya 152 W/m^2. Pemangkin heterojunction n–n–p yang disintesis dalam kajian ini menunjukkan hasil hidrogen yang lebih tinggi berbanding sistem pemangkin sedia ada. Dengan merujuk kepada analisis Mott–Schottky dan mekanisme pemindahan caj yang dicadangkan, perumusan struktur unik n–n–p CdO–CuO@exfoliated g–C_3N_4 nanoheterojunction memenuhi potensi reduksi–oxidasi fotoreformasi, seterusnya meningkatkan pemisahan dan migrasi cas, sehingga membawa kepada prestasi pengeluaran hidrogen yang cekap. Dari hasil LSV dan Tafel, CdO–CuO@exfoliated g–C_3N_4 mempamerkan aktiviti HER terbaik dengan nilai cerun Tafel terkecil 53 mV/dec berbanding elektrod lain yang telah diuji. Selain daripada itu, model kinetik autokatalitik dicadangkan dan menunjukkan kesesuaian yang sangat baik untuk reaksi fotoreformasi seperti yang dibuktikan dari plot kadar eksperimen dan teori serta nilai R^2 yang tinggi untuk semua kepekatan formaldehid. Oleh itu, penyelidikan ini memberikan pandangan

yang bermakna untuk pembinaan fotokatalisis novel dengan menggabungkan persimpangan yang berbeza dan aplikasi untuk penjanaan tenaga hijau serentak dengan rawatan air sisa.

ABSTRACT

Today, population growth and development of industrializations creates huge dilemma by virtue of increasing global energy demand and environmental issues. To overcome the current challenges of fossil fuel depletion and ever–growing water pollution, intensive efforts have been taken towards exploring sustainable pathway to tackle the present problems. In this context, photocatalysis holds great promise as it harvests the widely available renewable source, solar energy to produce hydrogen. Accordingly, photocatalytic reforming or photoreforming of petrochemical wastewaters offers a sustainable routes for dual benefits of green energy production and wastewater remediation. The current research was intended to synthesize highly promising polymeric–based photocatalyst, graphitic carbon nitride (g–C_3N_4) as the support and further tuned by exfoliating the material and doped with metal oxides such as CdO and CuO. Subsequently, a novel CdO–CuO@exfoliated nanoheterojunction was constructed through a facile wet impregnation technique. From XRD results, the blue–shifting of the bulk g–C_3N_4 (27.90°) to lower peak of exfoliated g–C_3N_4 (27.15°), creating more spaces between the inter–planar layers thereby revealing successful distortion of the triazine planar and formation of nanosheets. Moreover, BET results showed large improvement of the specific surface area of exfoliated g–C_3N_4 (104.4 m^2/g) and CdO–CuO@exfoliated g–C_3N_4 (388.4 m^2/g) by 3 and 11 times respectively compared to bulk g–C_3N_4 (34.5 m^2/g). Subsequently, the as–synthesized photocatalysts were tested for the photoreforming activity of hydrogen production in aqueous formaldehyde medium and irradiated under LED light. As an outcome, the activity trend for the hydrogen was in an order of bulk g–C_3N_4 < exfoliated g–C_3N_4 < CdO@exfoliated g–C_3N_4 < CuO@exfoliated g–C_3N_4< CdO–CuO@exfoliated g–C_3N_4. An excellent activity of the nanoheterojunction was highly contributed by large surface area caused more active sites available for the redox reactions. Moreover, the tremendous reduction of peak intensity in PL analysis greatly hinders the recombination rate of electron–hole pairs, thereby promoting facile charge separations and migrations, fostering the hydrogen production. Thereafter, CdO–CuO@exfoliated g–C_3N_4 was further examined to optimize the parameters and study the correlation between different factors. Based on the ANOVA, the model was well–fitted with F–value and p–value of 54.97 and <0.0001 (less than 0.05) respectively. Besides, the interactions between the diverse parameters were investigated and demonstrated via the contour and three–dimensional plots. The maximum hydrogen produced was 9025 μmol/g for following conditions: reaction time of 8 h, catalyst dosage of 0.73 g/L, initial FA–concentration of 573 ppm and light intensity of 152 W/m^2. The developed n–n–p heterojunction catalyst demonstrated higher hydrogen yield compared to the existing catalytic systems. By referring to Mott–Schottky analysis and proposed charge transfer mechanism, the formulation of unique band structure of n–n–p CdO–CuO@exfoliated g–C_3N_4 nanoheterojunction fulfilled the photoreforming reduction and oxidation potentials, subsequently enhancing the charge separation and migrations, thus leading to an efficient hydrogen production performance. From the LSV results and Tafel slopes, CdO–CuO@exfoliated g–C_3N_4 exhibits best HER activity with smallest Tafel slope value of 53 mV/dec among all the prepared electrodes. Apart from that, an autocatalytic kinetic model was proposed and exhibits excellent fitting for the photoreforming reactions as evidenced from the plot of experimental and theoretical rates as well as high R^2 values for all the initial formaldehyde concentrations. Hence, the present research provides meaningful insights for the development of novel photocatalyst by combining different

junctions and the applications for simultaneous generation of green energy as well as valorization of wastewaters.

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