PREPARATION AND CHARACTERIZATION OF PALM KERNEL SHELL ACTIVATED CARBONS

By

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The physical properties of steam-activated palm kernel shell activated carbons (OPSA) were determined by nitrogen physisorption at 77 K. Adsorption models such as Dubinin-Raduschkevich (DR), Brunnauer-Emmet-Teller (BET), Barret-Joyner-Halenda (BJH) and Hovath-Kawazoe (HK) are used in complimentary to elucidate the extent of micro- and mesoporosity in the complex porous structures of OPSA. The characterization studies reveal interesting insights towards the degree of porosity, which vary according to the particle sizes of the commercial OPSA. Appreciable increase of mesoporosity from 15 to 39 % of the total porosity occurred as particles size decreases from granular OPSA1 of 1.7-2.38 mm to OPSA4 of less than (0.6 mm). Scanning electron microscopy studies reveal the pore channeling effect in granular OPSA where large openings appears as passageway to a whole network of pores in the interior of the particle.

The flexibility of commercial OPSA in altering the physical attributes is investigated by subjecting it to minimal treatment without requiring any chemical agents

simultaneously eliminating the generation of chemical wastes. The treatments employed are mechanical grinding and oxidation in air. A 2-fold increase in mesopore volume is experienced when granular OPSA1 (1.7-2.38 mm) and OPSA2 (0.6-1.7 mm) are ground to smaller particle sizes of less than 0.15 mm. Interestingly, the high fraction of micropores remains unchanged, hence, providing dual adsorption capabilities contributed by both micro- and mesoporosity. On the other hand, oxidized OPSA2 in air at optimum temperature of 450 ^{o}C for 3 hours resulted in a substantial increase of mesopore volume from 0.19 to 0.33 cm^3/g, whilst maintaining the micropore volume of the structures. This enables the preservation of high micropore volume while altering the mesoporosity properties.

Liquid phase adsorption at ambient temperature of methylene blue (MB) and iodine were conducted to support and verify the validity of the nitrogen physisorption analyses on the micro- and mesoporosity of the OPSA. The higher degree of mesoporosity in the OPSA structures provides higher adsorption capacity of MB. On the other hand, the adsorption of iodine molecules correlates to the degree of microporosity. High accessibility to the abundant micropores in the interior of the OPSA structure results in higher fraction of micropore coverage, thus, enabling maximum exploitation of the internal porosity. The sorption kinetics of MB conforms to the pseudo-second order, where rapid equilibrium phase of dye-OPSA systems were achieved. Shorter equilibrium time was achieved in OPSA with high mesopore volume.

OPSA of higher mesoporosity were utilized in the refining of palm oil in order to justify the importance of mesoporosity in this application. Feasible results were achieved as performance of OPSA proven to be competitive towards other commonly used bleaching adsorbents in the industries such as bleaching earth and synthetic silica. OPSA exhibit high affinity towards the removal of chlorophyll in the oils. OPSA has also shown good performance in prohibiting the formation of oxidation products such as peroxides, conjugated dienes and trienes. This study simultaneously highlights the effect of acid-treated activated carbons on the final quality of the oil. This study also incorporates the compatibility of OPSA with adsorbents such as attapulgite and silica. Overall, this study provides the physical criteria of the OPSA to be utilized as an effective bleaching adsorbent and may serve as a useful guide in the synthesis of better performing adsorbent from palm kernel shell activated carbons. On the other hand, OPSA exhibit good performances in the purification of glycerin. Higher mesoporosity in OPSA provides higher economical viability where high mesoporosity would require lesser dosage of activated carbons than OPSA of low mesopore volume.

In newer applications, selected OPSA are used as catalyst supports. This study provides information on the implications of the method of preparation and impregnation solution onto the physical properties of the supports. Vast differences occur in the preparation of platinum on the supports of different particle sizes due to the different porosity characteristics provided to the particular catalytic system. In granular OPSA, development of mesopore occurs due to the structural collapse of the support when the impregnation solution is introduced. On the contrary, the support of smaller particle size does not emulate similar porosity behaviour, where reduction in micro-, mesopore and total pore volume occurred.

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Teknik penjerapan gas nitrogen pada suhu 77 K digunakan untuk pencirian sifat fizikal karbon teraktif daripada tempurung sawit (OPSA). Model-model penjerapan yang digunakan ialah Dubinin-Raduskevich (DR), Brunnauer-Emmet-Teller (BET), Barret-Joyner-Halenda (BJH) dan Hovath Kawazoe (HK) untuk mencirikan keliangan mikro dan meso secara terperinci di dalam struktur kompleks karbon teraktif. Hasil

kajian menunjukkan bahawa darjah keliangan berubah dengan saiz partikel karbon teraktif. Apabila saiz partikel berkurang dari 1.7-2.38 mm (OPSA1) ke kurang dari 0.6 mm (OPSA4), keliangan meso bertambah dari 15 hingga 39 %. Teknik mikroskopi electron (SEM) turut mendedah pencirian unik akan struktur karbon teraktif tempurung sawit. Kajian menunjukkan bahawa struktur OPSA mengandungi rangkaian keliangan sehingga ke struktur dalaman karbon.

Kajian turut mendedahkan keupayaan untuk mengubah ciri-ciri fizikal OPSA menerusi kaedah mekanikal dan kaedah pengoksidaan di dalam aliran udara. Pertambahan sebanyak dua kali di amati apabila OPSA1 di haluskan kepada saiz partikel kurang daripada 0.15 mm. Namun begitu, darjah keliangan mikro tidak mengalami sebarang perubahan yang ketara. Justeru itu, memberikan OPSA sifat dwipenjerapan disebabkan cirri-ciri keliangan mikro dan meso. Pengoksidaan OPSA pada suhu optimum 450 ^{o}C selama 3 jam di dalam aliran udara telah menghasilkan karbon teraktif yang mengandungi darjah keliangan meso yang lebih tinggi, iaitu pertambahan dari 0.19 ke 0.33 cm^3/g.

Penjerapan fasa cecair pada suhu persekitaraan di kaji dengan menggunakan ‘methylene blue’ (MB) dan iodine. Kajian dijalankan khas untuk menunjukkan sifat jerapan karbon teraktif serta membuktikan kesahihan teknik penjerapan gas nitrogen. Darjah keliangan meso yang lebih tinggi di dalam struktur OPSA membolehkan darjah penjerapan yang lebih tinggi. Di dalam penjerapan iodin pula, didapati kemudahan untuk memanipulasi keliangan mikro yang tinggi di pedalaman struktur

OPSA memberangsangkan penjerapan iodine. Kinetic jerapan MB mematuhi 'pseudo-second order' di mana fasa keseimbangan MB-OPSA adalah lebih singkat.

OPSA dengan darjah keliangan meso yang tinggi digunakan sebagai bahan penjerap di dalam aplikasi pemprosesan minyak sawit. Hasil kajian menunjukkan bahawa OPSA mempunyai kelebihan untuk menghasilkan minyak yang berkualiti memuaskan. Kajian ini turut menunjukkan keupayaan OPSA untuk bersaing dengan bahan penjerap yang biasa digunakan di dalam industri pemprosesan minyak sawit. OPSA turut mempamerkan keberkesanan dalam penjerapan klorofil. OPSA turut menghasilkan minyak yang mempunyai produk teroksida yang minimum seperti peroksida, aldehid, ketone dan polyene terkonjugat. Kajian ini turut mendedahkan kesan rawatan asid ke atas karbon teraktif serta pergabungan dengan bahan penjerap yang lain. Di dalam aplikasi oleokimia, OPSA turut menunjukkan pencapaian yang memberangsangkan di dalam pelunturan gliserin dengan warna kurang daripada 6 AHPA.

OPSA yang terpilih digunakan di dalam aplikasi pemangkinan. Kajian ini menunjukkan kesan larutan asid heksakloroplatinik dalam penyediaan mangkin dengan menggunakan OPSA2 dan OPSA2α. Perbezaan ciri keliangan yang ketara di dalam mangkin yang disediakan turut dipengaruhi oleh sifat keliangan penyokong OPSA2 dan OPSA2α yang mempunyai darjah keliangan meso yang berlainan. Untuk sistem pemangkin Pt-OPSA2, perkembangan keliangan meso dikatakan berpunca daripada pemecahan struktur pejal penyokong. Akan tetapi, untuk sistem pemnagkin

Pt-OPSA2α, perkembangan keliangan yang berbeza berlaku di mana keliangan mikro , meso dan keliangan keseluruhan berkurangan apabila peratus platinum di atas OPSA2α bertambah dari 1 hingga 5 %.

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DECLARATION

I hereby declare that the 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 UPM or other institutions.

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This thesis submitted to the Senate of Universiti Putra Malaysia and have been accepted as fulfilment of the requirements for the degree of Master of Science. The members of the Supervisory Committee are as follows:

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