

**REGENERATION AND CHARACTERIZATION OF SPENT BLEACHING
CLAY**

By

NUR SULIHATIMARSYILA BT. ABD. WAFTI

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Master of Science**

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**Abstract of thesis presented to the Senate of Universiti Putra Malaysia in
fulfilment of the requirement for the degree of Master of Science**

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Chairman: Associate Professor Thomas Choong Shean Yaw, PhD

Faculty: Engineering

The spent bleaching clay (SBC), a solid waste generated from palm oil refinery, may be recycled rather than simply disposed off in landfill. The aim of this study is to investigate the heat regeneration of SBC and to evaluate the performance of the heat-treated SBC on the bleaching of crude oil. The quality of oil is first studied. The quality of the oil extracted from spent bleaching clay from palm oil refinery (SBC-PO) and spent bleaching clay from palm kernel oil refinery (SBC-PKO) much inferior compared to that of crude oil. De-oiling efficiency for both SBC increases as the solid to solvent ratio is decreased.

Two type of SBC are studied such as acid-activated clay (XMP, WAC) and natural clay (AH). Two types of regeneration processes are performed, such as (a) solvent extraction followed by heat treatment and (b) regeneration carried out by direct heat treatment. Heat treatment is conducted in a box furnace at temperatures ranging from 400 to 1000°C. Red color indices of oils are used to determine the regeneration efficiency. Spent bleaching clay produced by direct

heat treatment (HRSBC), yields a higher regeneration efficiency than the deoil-heat-regenerated spent bleaching clay (DHSBC), produced by solvent extraction and heat treatment. The specific surface area, total pore volume and average pore size of SBC are measured using nitrogen adsorption-desorption method. The proximate analysis is also performed to clarify the mechanism of regeneration process. The data shows that the HRSBC at 500°C possess the highest specific surface area and total pore volume and give better bleaching efficiency than that of HRSBC at 400 and 800°C. All the regenerated SBC samples are mesoporous material.

Adsorption isotherm is useful for determining the efficiency of the HRSBC in removing pigments. The Henry's Law equation is found to be more applicable than the Langmuir and Freundlich isotherms in the adsorption of pigments from degummed palm oil on HRSBC. The adsorptive efficiency of the HRSBC is optimum at regeneration temperature of 500°C.

**Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master Sains**

PEMULIHAN SEMULA DAN SIFAT TANAH PELUNTUR TERGUNA

Oleh

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Tanah peluntur terguna (TPT) adalah sisa pepejal terhasil daripada kilang pemprosesan minyak sawit, boleh dikitar semula selain daripada dibuang ke tempat pelupusan. Tujuan penyelidikan ini adalah untuk menyiasat proses pemulihan menggunakan pemanasan haba terhadap TPT serta menilai kecekapan TPT haba-terpulihan terhadap proses pelunturan minyak mentah. Kualiti minyak tersimpan dikaji terlebih dahulu. Kualiti minyak yang diekstrak daripada TPT dari kilang minyak sawit dan minyak isirung kelapa sawit adalah lebih rendah berbanding minyak mentah. Kecekapan membuang minyak tersimpan untuk kedua-dua TPT meningkat apabila nisbah pepejal kepada larutan hexane menurun.

Dua jenis TPT telah digunakan iaitu peluntur tanah asid teraktif (WAC, XMP) dan peluntur tanah semulajadi (AH). Dua jenis proses pemulihan telah digunakan iaitu (a) pengekstrakan larutan diikuti dengan pemulihan pemanasan haba dan (b) pemulihan oleh pemanasan haba terus. Pemulihan secara pemanasan telah dijalankan di dalam relau berkotak pada suhu di antara 400 hingga 1000°C. Warna merah minyak sawit mentah dan minyak sawit yang diproses digunakan untuk mengira kecekapan pemulihan. TPT yang

dihasilkan oleh pemanasan haba terus memperoleh kecekapan pemulihan yang lebih tinggi berbanding TPT yang dihasilkan daripada pengekstrakan larutan dan pemanasan haba. Luas permukaan spesifik, jumlah isipadu ruang dan pembahagian saiz ruang dianalisa, diperolehi daripada kaedah penjerapan-nyahpenjerapan nitrogen. Penentuan analisa yang paling hampir ditentukan dengan tujuan untuk menerangkan mekanisma proses pemulihan. Data menunjukkan bahawa TPT dari pemanasan haba terus pada suhu 500°C menghasilkan luas permukaan spesifik dan jumlah isipadu ruang paling tinggi serta memberi kecekapan pelunturan lebih baik daripada TPT terpulih pada suhu 400 dan 800°C. Semua sampel TPT menunjukkan sifat bahan yang mempunyai ruang *meso*.

Isoterma penjerapan berfaedah untuk menentukan kecekapan TPT terpulih dalam menyingkirkan pikmen. Hukum *Henry* didapati lebih sesuai digunakan berbanding isoterma *Langmuir* dan isoterma *Freundlich* dalam penjerapan pikmen daripada minyak sawit yang dinyahgam oleh TPT terpulih haba dari proses pemanasan terus. Kecekapan penjerapan adalah optima pada suhu pemulihan 500°C.

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I certify that an Examination Committee has met on 28th March 2006 to conduct the final examination of Nur Sulihatimarsyila Bt. Abd. Wafti on her Master of Science thesis entitled “Regeneration and Characterization of Spent Bleaching Clay” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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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 is has not been previously or concurrently submitted for any other degree at UPM or other institutions.

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Date: 28 May 2006

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LIST OF NOTATIONS/SYMBOLS/ABBREVIATIONS

a_s	Adsorbance of sample	
a_b	Cuvette error	
α_L	Energy of adsorption	L/mg
C_o	Initial concentration of solution	mg/L
C_e	Liquid phase concentration at equilibrium	mg/L
$E_{233c}^{1\%}$	Specific extinction in UV-light 233 nm	
$E_{269c}^{1\%}$	Specific extinction in UV-light 269 nm	
K_F	Freundlich adsorption capacity	mg/g
K_H	Henry's Law constant	L/g
K_L	Langmuir constant	L/g
n	Surface heterogeneity	
P	Actual gas pressure	mmHg
P_o	Vapor pressure	mmHg
P/P_o	Relative pressure	
q_e	Solid phase concentration	mg/g
r^2	Square correlation coefficient	
R_o	Red color index of unbleached oil	
R_r	Red color index of bleached oil by regenerated clay	
R_a	Red color index of bleached oil by fresh clay	
S_{BET}	BET specific surface area	m²/g
V_{total}	Total pore volume determine at 0.99 P/Po	cm³/g
V	Volume of oil	L
ASAP	Accelerated surface area and porosimetry	
ASTM	American Society for Testing and Materials	
B	Blue Lovibond unit	
BDDT	Brunaller, Deming, Deming and Teller	
BET	Brunnauer-Emmet-Teller	
BJH	Barret-Joyner-Halenda	
CPO	Crude palm oil	

CPKO	Crude palm kernel oil
DHRSBC	Deoil-heat-regenerated spent bleaching clay
DOBI	Deterioration of bleachability index
DSBC	Deoiled spent bleaching clay
	Oil extracted from spent bleaching clay at palm oil refinery
EPO	
	Oil extracted from spent bleaching clay at palm kernel oil refinery
EPKO	
FFA	Free fatty acid
g	gram
HRSBC	Heat-regenerated spent bleaching clay
hrs	hours
IUPAC	International Union of Pure and Applied Chemistry
IV	Iodine value
MPOB	Malaysian Palm Oil Board
mins	minutes
mmHg	Milimetre mercury
nm	nanometre
N	Neutral Lovibond unit
PORIM	Palm Oil Research Institute Malaysia
R	Red Lovibond unit
RBD	Refined, bleached and deodorized oil
SBC	Spent bleaching clay
SCOPA	Seed Crushers and Oil Produce's Association
UV	Ultraviolet
wt	Weight
Y	Yellow Lovibond unit

CODE OF SAMPLES

AH	Attapulgite Hudson
DHRSBC-WAC400	DHRSBC of WAC at regeneration temperature of 400°C
DHRSBC-WAC500	DHRSBC of WAC at regeneration temperature of 500°C

DHRSBC-WAC800	DHRSBC of WAC at regeneration temperature of 800°C
DHRSBC-AH400	DHRSBC of AH at regeneration temperature of 400°C
DHRSBC-AH500	DHRSBC of AH at regeneration temperature of 500°C
DHRSBC-AH800	DHRSBC of AH at regeneration temperature of 800°C
DHRSBC-PKO400	DHRSBC of PKO at regeneration temperature of 400°C
DHRSBC-PKO500	DHRSBC of PKO at regeneration temperature of 500°C
DHRSBC-PKO600	DHRSBC of PKO at regeneration temperature of 600°C
DHRSBC-PKO800	DHRSBC of PKO at regeneration temperature of 800°C
DHRSBC-PKO1000	DHRSBC of PKO at regeneration temperature of 1000°C
DHRSBC-PO400	DHRSBC of PO at regeneration temperature of 400°C
DHRSBC-PO500	DHRSBC of PO at regeneration temperature of 500°C
DHRSBC-PO600	DHRSBC of PO at regeneration temperature of 600°C
DHRSBC-PO800	DHRSBC of PO at regeneration temperature of 800°C
DHRSBC-PO1000	DHRSBC of PO at regeneration temperature of 1000°C
HRIBC-PKO400	HRIBC of PKO at regeneration temperature of 400°C
HRIBC-PKO500	HRIBC of PKO at regeneration temperature of 500°C
HRIBC-PKO600	HRIBC of PKO at regeneration temperature of 600°C
HRIBC-PKO800	HRIBC of PKO at regeneration temperature of 800°C
HRIBC-PKO1000	HRIBC of PKO at regeneration temperature of 1000°C
HRIBC-PO400	HRIBC of PO at regeneration temperature of 400°C
HRIBC-PO500	HRIBC of PO at regeneration temperature of 500°C
HRIBC-PO600	HRIBC of PO at regeneration temperature of 600°C
HRIBC-PO800	HRIBC of PO at regeneration temperature of 800°C
HRIBC-PO1000	HRIBC of PO at regeneration temperature of 1000°C
HRIBC-AH400	HRIBC of AH at regeneration temperature of 400°C
HRIBC-AH500	HRIBC of AH at regeneration temperature of 500°C
HRIBC-AH800	HRIBC of AH at regeneration temperature of 800°C
HRIBC-WAC400	HRIBC of WAC at regeneration temperature of 400°C
HRIBC-WAC500	HRIBC of WAC at regeneration temperature of 500°C
HRIBC-WAC800	HRIBC of WAC at regeneration temperature of 800°C
SBC-PKO	Spent bleaching clay from palm kernel oil refinery
SBC-PO	Spent bleaching clay from palm oil refinery

WAC
XMP

WAC Classic
WAC XMP