

SYNTHESIS OF GRAPHENE SAND  
COMPOSITE BY USING *ARENGA* PALM  
AND TABLE SUGAR TO REMOVE  
METHYLENE BLUE AND NICKEL  
FROM AQUEOUS SOLUTION

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DOCTOR OF PHILOSOPHY

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## **SUPERVISOR'S DECLARATION**

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy in Environmental Technology.

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## **STUDENT'S DECLARATION**

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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NORUL WAHIDA BINTI KAMARUZAMAN

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## ABSTRAK

Pencemaran sumber air telah meningkat di beberapa kawasan di dunia disebabkan oleh pertumbuhan industri dan pelepasan efluen yang tidak dirawat, peningkatan gaya hidup urban dan tenaga intensif, kekurangan perlindungan hutan dan kesedaran alam sekitar, dan kelemahan pelaksanaan dan penguatkuasaan peraturan alam sekitar. Objektif tesis ini adalah untuk mensintesis komposit grafin dengan menggunakan gula kabung dan gula biasa sebagai sumber karbon untuk menyerap metilena biru dan nikel dari larutan akueus. Gula kabung dipilih sebagai sumber karbon kerana persamaan sifat dengan gula biasa dan tiada kajian mendalam dibuat ke atas bahan tersebut setakat ini. Proses sintesis dilakukan dengan merawat pasir melalui ayakan. Larutan gula disediakan dan dipanaskan sehingga 95 °C selama 2 jam dengan pengacauan berterusan bersama-sama dengan pasir sebelum dipanaskan di dalam relau dengan persekitaran nitrogen bagi menghasilkan 'graphene sand composite', GSC<sub>aps</sub> dan GSC<sub>ts</sub>. Komposit kemudiannya diaktifkan dengan menggunakan asid sulfurik pekat untuk memaksimumkan kapasiti penyerapan. Analisis karakter GSC<sub>aps</sub> dan GSC<sub>ts</sub> dilakukan melalui mikroskop pengimbasan pelepasan elektron (FESEM), x-ray unsur serakan (EDX), pemetaan unsur (EM) dan spektroskopi raman. Analisis pengimbasan pelepasan elektron (FESEM) menunjukkan proses salutan gula dilakukan secara seragam dengan lapisan di luar permukaan pasir. Unsur-unsur yang terbesar dalam komposit pasir grafin adalah karbon, oksigen dan silika. Terdapat juga puncak pembelauan terletak pada 26.66 ° dan 26.80 ° yang menunjukkan pembentukan hablur di permukaan GSC<sub>aps</sub> dan GSC<sub>ts</sub> yang memberi kesan kilauan kepada komposit melalui refleksi cahaya. Ujian spektroskopi raman mengesan dua puncak penting iaitu band G dan D pada 1547 cm<sup>-1</sup> dan 1292 cm<sup>-1</sup>. Ujian penyerapan menunjukkan bahawa 100 % daripada 1 ppm metilena biru dan nikel telah berjaya diserap dengan menggunakan 1 g GSC dengan penyerapan 50 % dan 48 % pada jam pertama. Keseimbangan penyerapan metilena biru dan nikel dicapai pada kira-kira 10-12 jam. Persamaan pseudo kedua adalah sesuai menggambarkan kinetik penyerapan metilena biru dan nikel dengan  $R^2 = 0.9993$  dan  $R^2 = 0.9972$  dengan menggunakan GSC<sub>aps</sub> manakala  $R^2 = 0.9981$  dan  $R^2 = 0.9948$  dengan menggunakan GSC<sub>ts</sub> secara berasingan. Data isoterma bagi penyingkiran metilena biru dan nikel adalah sesuai dengan model Langmuir dengan  $R^2 = 0.9999$  dan  $R^2 = 0.9975$  dengan menggunakan GSC<sub>aps</sub> manakala  $R^2 = 0.9945$  dan  $R^2 = 0.9962$  dengan menggunakan GSC<sub>ts</sub>. Penggunaan semula GSC<sub>aps</sub> dan GSC<sub>ts</sub> mencatatkan 87 % dan 88 % kehilangan upaya sebelum proses generasi dan berjaya mengembalikan 76 % upaya dalam menyerap metilena biru dan nikel. Kesimpulannya, ia menunjukkan bahawa kedua-dua GSC mempunyai potensi sebagai penyerap yang baik dengan 7 % peningkatan penyerapan berbanding dengan kaedah konvensional dengan menggunakan set parameter yang sama.

## ABSTRACT

Pollution of water sources has increased in a number of regions of the world due to the growth of industries and its untreated effluent discharge, increasing urbanization and energy intensive life styles, loss of forest cover, lack of environmental awareness and lack of implementation of environmental rules and regulations. The aim of the thesis is to synthesis green graphene sand composite (GSC) by using arenga palm sugar and table sugar as carbon sources to remove methylene blue and nickel from aqueous solution through adsorption process. The arenga palm sugar was chosen as carbon source due to the similarity on properties with ordinary table sugar and no research has been conducted so far on this particular material. The synthesis was conducted by removing deleterious materials on sand's surfaces before sieved. The solutions of sugar were prepared and heated to 95 oC for 2 hours with constant stirring together with sand before being put in furnace with nitrogen environment to produce graphene sand composite, GSCaps and GSCts. The composites then were activated by using concentrated sulphuric acid to maximise the capacity of absorbency. The analyses on the characteristic of both GSC were conducted through field emission scanning electron microscope (FESEM), elemental dispersive x-ray (EDX), elemental mapping (EM) and raman spectroscopy. Field Emission Scanning Electron Microscope (FESEM) analyses exhibited that the sugar coating process was done uniformly as there were layers of coating sheets formation outside the sand particles' surface. It was found that the biggest elements in the composite were carbon, oxygen and silica in scattered distribution. There was also a strong diffraction peak located at 26.66 ° and 26.80 ° which indicates the formation of the crystalline of the GSCaps and GSCts respectively which give glowing effect to the composites due to the light reflection. The test on raman spectroscopy detected two important peaks namely G and D band at 1547 cm<sup>-1</sup> and 1292 cm<sup>-1</sup> respectively. Adsorption test showed that 100 % of 1 ppm methylene blue and nickel were successfully adsorbed by using 1 g of GSC with 50 % and 48% adsorption in the first hour of contact respectively. Complete adsorption of methylene blue and nickel was successfully achieved at 10 – 12 hours of contact. The analysis exhibited pseudo second order equation is more appropriate in describing kinetic of the methylene blue and nickel adsorption with R<sup>2</sup>=0.9993 and R<sup>2</sup>=0.9972 by using GSCaps whereas R<sup>2</sup>=0.9981 and R<sup>2</sup>=0.9948 by using GSCts respectively. The isotherm data of methylene blue and nickel removal were fitted Langmuir model best with R<sup>2</sup>=0.9999 and R<sup>2</sup>=0.9975 by using GSCaps whereas R<sup>2</sup>=0.9945 and R<sup>2</sup>=0.9962 by using GSCts respectively. The reusability of GSCaps and GSCts recorded 87 % and 88 % adsorbency capacity loss before regeneration and able to restore approximately 76 % capacity in adsorbing methylene blue and nickel. In conclusion, both GSCs have shown potentials to be acted as good adsorbent with a total of 7 % adsorption increment comparing to conventional method, by using similar parameters setting in the experiment.

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## LIST OF SYMBOLS

$\text{Al}_2\text{O}_3$	Aluminium Oxide
C	Carbon
$C_e$	Equilibrium concentration
$C_i$	Initial concentration
$C_f$	Final concentration
Co (II)	Cobalt (II)
Cr (VI)	Chromium (IV)
Cu (II)	Copper (II)
$\text{Fe}_2\text{O}_3$	Ferric (III) Oxide
$\text{Fe}_3\text{O}_4$	Ferroferric Oxide
g	Gram
H	Hydrogen
HCl	Hydrochloric Acid
$k_1$	Pseudo first order constant
$k_2$	Pseudo second order constant
$K_F$	Freundlich constant
$K_L$	Langmuir constant
L	Litre
mg	Miligram
$\text{MnO}_2$	Manganese Dioxide
$\text{MnFe}_2\text{O}_4$	Manganese Ferrite
n	Adsorption intensity
NaOH	Sodium hydroxide
Ni (II)	Nickel (II)
Pb (II)	Plumbum (II)
ppm	Parts per million
$q_e$	Equilibrium adsorption
$q_m$	Maximum adsorption
$q_t$	Adsorption as a factor of time
$R^2$	Correlation
$R_L$	Separation factor
t	Time

TiO <sub>2</sub>	Titanium Dioxide
V	Volume
W	Weight
Zn (II)	Zinc (II)
°C	Degree Celsius
%	Percentage



## LIST OF ABBREVIATIONS

ASTM	American Society of Testing Materials
BS	British Standard
EDX	Elemental Dispersive X-Ray
FESEM	Field Emission Scanning Electron Microscopic
GSC	Graphene Sand Composite
GSC <sub>aps</sub>	Graphene Sand Composite using <i>Arenga</i> Palm Sugar
GSC <sub>ts</sub>	Graphene Sand Composite using Table Sugar
IWMI	International of Water Management Institute
POFA	Palm Oil Fuel Ash
XRD	X-Ray Diffraction

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