
**UNIVERSITI PUTRA MALAYSIA**

# *EXTRACTION AND CHARACTERIZATION OF NANOCRYSTALLINE CELLULOSE FROM OIL PALM EMPTY FRUIT BUNCH AND ITS POTENTIAL AS ADSORBENT FOR DYE REMOVAL*

**BAWAANII SHANMUGARAJAH**

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By

**BAWAANII SHANMUGARAJAH**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment 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 fulfillment of the requirement for the Degree of Master of Science

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**BAWAANII SHANMUGARAJAH**

**May 2017**

**Chairman : Professor Ir Thomas Choong Shean Yaw,PhD**
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Over the past few decades, enormous interest has been manifested in using biomass as a source of renewable energy and materials. Cellulose, in the form of Nanocrystalline Cellulose (NCC), gains prominence as a new nanostructured material. In this study, NCC was extracted from oil palm empty fruit bunch (EFB) via acid hydrolysis method. NCC was successfully extracted from EFB using 64 wt% sulfuric acid at 45°C for 45 min. The products obtained from each stage were characterized to confirm the removal of non-cellulosic components. In addition, X-ray Diffraction (XRD) results disclosed that nanocrystals exhibited crystallinity index of 60%. On the other hand, Transmission Electron Microscope (TEM) showed NCC exhibited network like structure. It also showed that the native cellulose crystalline network region is approximately 20 nm diameter, while Dynamic Light Scattering (DLS) suggested NCC to have an average particle size of 291.4 nm. Following that, NCC was employed for Methylene Blue (MB) dye removal. Batch adsorption studies were carried out to analyze the effect of contact time, initial dye concentration, adsorbent dosage and agitation speed at a temperature of 30 ± 2°C. The obtained experimental data fitted well to Langmuir isotherm with monolayer adsorption capacity of 144.93 mg/g. Pseudo-second-order kinetic model provided a better correlation of the experimental data than others such as Lagergrens pseudo-first-order model. It was also found that intraparticle diffusion was not the only rate controlling step, instead, boundary layer diffusion was also contributing to the adsorption activity.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

**KAJIAN PENCIRIAN KRISTAL SELULOSA NANO DIEKSTRAK DARIPADA TEMPURUNG KELAPA SAWIT DAN POTENSINYA SEBAGAI PENYERAP UNTUK PENYINGKIRAN PEWARNA**

Oleh

**BAWAANII SHANMUGARAJAH**

**Mei 2017**

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Sejak beberapa dekad yang lalu, kepentingan yang sangat besar telah dimanifestasikan dalam menggunakan biojisim sebagai sumber tenaga dan bahan yang boleh diperbaharui. Selulosa, dalam bentuk selulosa kristal nano (NCC), mendapat kemasyhuran sebagai bahan baru yang bersize nano. Dalam kajian ini, NCC telah diasingkan daripada tempurung kelapa sawit (EFB) melalui proses asid hidrolisis. NCC telah berjaya diekstrak daripada EFB dengan menggunakan 64 wt% asid sulfurik pada suhu 45°C selama 45 min. Produk yang diperolehi dari setiap peringkat diuji menggunakan untuk mengesahkan penyingkiran komponen bukan selulosa. Selain itu, keputusan *X-ray Diffraction* (XRD) menunjukkan bahawa kristal nano mempamerkan indeks penghabluran sebanyak 60%. *Transmission Electron Microscope* (TEM) menunjukkan pendedahan kawasan rangkaian kristal nano asli (kira-kira 20 nm diameter), manakala *Dynamic Light Scattering* (DLS) mencadangkan NCC membawa saiz zarah purata 291.4 nm. Bahan tersebut kemudiannya digunakan untuk penyingkiran pewarna Metil Biru (MB). Kajian penjerapan kumpulan telah dijalankan untuk menganalisis kesan masa sentuhan, kepekatan awal pewarna, dos bahan penjerap dan kelajuan agitasi pada suhu 30 ± 2 ° C. Data eksperimen yang diperolehi melengkapi *Langmuir isotherm* dengan kapasiti penjerapan monolayer pada 144.93 mg/g. Model kinetik *pseudo-second-order* memberi korelasi yang lebih baik bagi data eksperimen berbanding model lain seperti model kinetik *Lagergrens pseudo-first-order*. Didapati juga *intraparticle diffusion* bukan satu-satunya langkah kadar kawalan, sebaliknya, *boundary layer diffusion* juga menyumbang kepada aktiviti penjerapan.

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I certify that a Thesis Examination Committee has met on 31 May 2017 to conduct the final examination of Bawaanii a/p Shanmugarajah on her thesis entitled "Extraction and Characterization of Nanocrystalline Cellulose from Oil Palm Empty Fruit Bunch and its Potential as Adsorbent for Dye Removal" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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| | |
|---|---|
| API | American Petroleum Institute |
| ASTM | American Society for Testing and Materials |
| BET | Brunauer-Emmett-Teller |
| BJH | Barrett-Joyner-Halenda |
| DLS | Dynamic Light Scattering |
| DTA | Differential Thermal Analysis |
| EFB | Empty fruit bunch |
| FESEM | Field Emission Scanning Electron Microscope |
| FTIR | Fourier Transform Infrared Spectrometer |
| GDP | Gross domestic product |
| HCl | Hydrochloric acid |
| ICr | Crystallinity Index |
| MB | Methylene blue |
| MF | Mesocarp fruit fibers |
| NaOH | Sodium hydroxide |
| NCC | Nanocrystalline cellulose |
| OPF | Oil palm fronds |
| OPT | Oil palm trunks |
| PCS | Photon Correlation Spectroscopy |
| PdI | Polydispersity Index |
| PKS | Palm kernel shell |
| POME | Palm oil mill effluent |
| PPF | Palm pressed fibers |

| | |
|---|---|
| RT | Room temperature |
| TGA | Thermogravimetric analysis |
| TEM | Transmission Electron Microscope |
| XRD | X-ray diffraction |
| Z-Avg | Z-Average |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background

The growth of oil palm industry in Malaysia is phenomenal. Starting off as an ornamental plant, the crop has been developed into a multibillion ringgit industry. Basically, products from oil palm are being used widely in food industries, cosmetics, personal care, and pharmaceutical. However, with the increase in the demand globally, total wastage upon extraction also rises remarkably. Hence, a technique or ways need to be implemented in order to discard this wastage without affecting the environment (Mondragon et al., 2014).

Besides, environmental pollution has certainly been regarded as one of the most severe problem in developed and developing countries. One of the industries which produce the most high-polluting contaminants is the textile industry. Concerning their complex aromatic structure, dye poses significant threat to human health and aquatic life. Therefore, in relation to both the problems, effort to search for a more cost effective and environmental friendly solution need to be deliberated. Thus, attention has been shifted towards nanomaterials. Today, nanotechnology has a great potential to profoundly improve standard of living. The recent leaps in nanostructured materials encourage scientists and engineers to deliberately look for bio-based resources to make materials at nanoscale, leading a radical new approach in emerging green nanoparticle for quality improvement (Nanogov, 2016).

Cellulose in the form of nanocrystalline cellulose (NCC) gains notable attention among researchers. Not only the research activities had been increased remarkably in the past 10 years, the first pilot plant of nanocellulose owned by Innentia was launched in 2011, which has a capacity of 100 kg/day. Following that, CelluForce launched the world largest commercial-scale plant, with capacity to produce 300 tonnes of NCC per year (CelluForce, 2016). Engineers and researchers had continuously search for more cost-effective technology to obtain NCC from biomass. For instance, American Process Inc (API) and Blue Goose Biorefineries Inc. (Blue Goose) introduced BioPlus® Nanocellulose Fibrils & Crystals and R3$^{TM}$ technology, respectively, to preclude costly process from obtaining high quality NCC (American process, 2016 and Blue Goose Biorefineries Inc, 2016).

## 1.2 Problem Statement

Palm oil industry has contributed significantly to Malaysia economy. However, lignocellulosic biomass specifically empty fruit bunch, which contributes 22% of total wastage from the palm oil extractions, has created a major disposal problem (Abdullah and Sulaiman, 2013). Therefore, this study attempts to convert empty fruit

bunch into a more valuable material which is in the form of nanocrystalline cellulose (NCC). Apart from that, methylene blue is one of the most common dyes used in adsorption related research. It is harmful to human, causing irritation to the gastrointestinal tract and eye burns. Adsorption has been proven to be an excellent method for removing dyes from aqueous solutions because of its significant advantages. Activated carbon is perhaps the most widely used adsorbent but it is prohibitively expensive. Therefore, enormous interest has been manifested in finding other suitable alternatives that is more economical and effective. This study aims to narrow this gap by isolating more environmentally friendly nano bio-material from agricultural solid wastes and a more effective alternative to treat dye from aqueous solution.

## 1.3 Research objectives

The objectives of the study are:

- To extract and characterize nanocrystalline cellulose from palm oil empty fruit bunch.
- To evaluate the adsorption performance of nanocrystalline cellulose for the removal of methylene blue from aqueous solution

## 1.4 Scope of research

The main objective of this study was to isolate nanostructured material in the form of NCC from agricultural solid wastes and to compare its removal efficiency of methylene blue from aqueous solution with other adsorbents. The NCC extracted from oil palm empty fruit bunch was characterized using FTIR, XRD, TGA, TEM and DLS. Only batch adsorption study was attempted. Ultraviolet-visible spectrophotometer was used to determine the removal rate of methylene blue. Several important parameters, i.e. pH, contact time and agitation speed were also investigated. The adsorption isotherms, i.e. Freundlich, and Langmuir were used to describe the interaction between adsorbent and adsorbate; and the adsorption kinetics was evaluated using pseudo-first order, pseudo-second order and intraparticle diffusion model.

## 1.5 Thesis outline

The dissertation begins with an introduction which comprised of background, aim, problem statement, research objectives and scope of the study **(Chapter 1)**. Following that **(Chapter 2)**, a short literature review was conducted on isolation of NCC and its potential in methylene blue removal. **Chapter 3** consists of a more comprehensive discussion on NCC isolation, as well as its characterization. **Chapter 4** discussed how NCC could be utilized to remove methylene blue from aqueous solution and short comparison study by using other adsorbents as a reference. The last, a short conclusion was drawn in **Chapter 5**, as well as future recommendation was included.

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