PREPARATION OF P-SULPHONATED CALIX[4]ARENE FUNCTIONALISED CELLULOSE ACETATE NANOFIBER FOR METHYLENE BLUE DYE REMOVAL FROM AQUEOUS SOLUTION

By

NUR AMILAH FADLINA BINTI BASRI

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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DEDICATION

I would like to dedicate this thesis to my beloved parents, Mr. Basri Bin Osman and Madam Che Zaiton Binti Yahaya, for their relentless supports, understanding and encouragement throughout my studies.

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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Chair : Mohd Haniff Wahid, PhD
Faculty : Science

Rapid growth in various industrial sectors, namely, dye related industries, has led to high levels of dye discharged into water resources thus posing threat to the environment and human health. Owing to high effectiveness and low cost, adsorption features as an ideal method for dye removal. Herein, adsorbent material in the form of nanofibers comprised of cellulose acetate based, *p*-sulphonated calix[4]arene (*p*-SOCX) functionalised graphene oxide (GO) were successfully prepared using the electrospinning technique. *p*-sulphonated calixarene and graphene oxide are promising extractants for benzene-based contaminants such as methylene blue due to the presence of electron-rich benzene rings through π-π interaction. Furthermore, the negatively charged sulfonate groups on the upper rim of *p*-sulphonated calixarene and oxygen rich functional groups on graphene oxide enable electrostatic and hydrogen bond interactions. Fourier Transform Infrared (FT-IR) spectroscopy and carbon-hydrogen-nitrogen-sulphur (CHNS) elemental analysis confirmed the presence of *p*-sulphonated calixarene within the fiber matrix. Scanning electron microscope (SEM) images of the prepared samples revealed the structural changes upon addition of graphene oxide and *p*-sulphonated calix[4]arene. The fiber's surface became rougher upon addition of graphene oxide and increased in fiber diameter was observed with increase in *p*-sulphonated calix[4]arene. Surface area analysis via Brunauer, Emmet and Teller (BET) method showed that the specific surface area of cellulose acetate/graphene oxide/*p*-sulphonated calix[4]arene (CA/GO/SOCX) nanofiber was 12.36 m^2/g. Thermal profiles of the nanofibers recorded via thermogravimetric analysis displayed slight enhancement in thermal stability after adding graphene oxide to cellulose acetate. However, for samples containing *p*-sulphonated calix[4]arene, the thermal stability decreased with increase in *p*-sulphonated calix[4]arene amount. Several reaction parameters namely, pH, adsorbent dosage, dye concentration, contact time, and temperature for methylene blue removal was investigated herein. Results show increased adsorption capacity of methylene blue was achieved in presence of *p*-sulphonated calix[4]arene compared to pure cellulose acetate nanofiber and cellulose acetate/graphene oxide nanofiber at an ideal pH 8. With an initial MB dye concentration of 10 mg/L, adsorbent dosage of 20 mg/10 mL, and contact time of 60 minutes at room temperature, *ca.* 298 K, the best removal efficiency was achieved, i.e., 88.84 %. The pseudo-second-order kinetic was considered to be more accurate to describe the adsorption behavior of MB ions compared to the pseudo-first-order model and intraparticle diffusion model. According to the fitting result of the Langmuir equation, maximum adsorption capacity of CA/GO/SOCX1% reached as high as 175.4 mg/g. The inclusion of *p*-sulphonated calix[4]arene at appropriate concentrations was observed to lead to a notable enhancement in both the adsorption capacities and the percentage removal of MB ions. The reusability study however shows considerable downfall in adsorption efficiency throughout the fifth cycle which is from 92% to 73%, 59%, 49% and 43% after five times of usage. It can be concluded that CA/GO/SOCX1% has potential to be used as an adsorbent for the methylene blue dye removal from aqueous solution.

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

PENYEDIAAN P-SULFONAT KALIKS[4]ARENA BERFUNGSIAN NANOSERAT SELULOSA ASETAT UNTUK PENYINGKIRAN PEWARNA METILENA BIRU DARI LARUTAN AKUAS

Oleh

NUR AMILAH FADLINA BINTI BASRI

Mei 2023

Pengerusi : Mohd Haniff Wahid, PhD
Fakulti : Sains

Pertumbuhan pesat dalam pelbagai sektor industri, khususnya, industri berkaitan pewarna, telah membawa kepada pembuangan pewarna pada tahap tinggi ke dalam sumber air, sehingga memberi ancaman kepada alam sekeliling dan kesihatan manusia. Oleh kerana keberkesanan yang tinggi dan kos yang rendah, penjerapan berperanan sebagai kaedah ideal untuk penyingkiran pewarna. Di dalam kajian ini, bahan penjerap dalam bentuk nanoserat berasaskan selulosa asetat dan grafen oksida dengan fungsian *p*-sulfonat kaliks[4]arena (*p*-SOCX) telah berjaya disediakan menggunakan teknik eletroputaran. *p*-SOCX dan grafen oksida adalah agen pengekstrak berkesan untuk pencemar berasaskan benzena seperti metilena biru kerana kandungan benzena yang kaya dengan elektron melalui interaksi π-π. Tambahan pula kumpulan sulfonat yang bercaj negatif pada pinggiran atas *p*-SOCX dan kumpulan berfungsi yang kaya dengan oksigen membenarkan interaksi elektrostatik dan ikatan hidrogenuntuk terjalin. Spektroskopi Inframerah Transformasi Fourier (FT-IR) dan analisis unsur karbon-hidrogen-nitrogen-sulfur (CHNS) mengesahkan kehadiran *p*-SOCX di dalam matriks nanoserat. Imej mikroskop pengimbasan elektron (SEM) bagi sampel yang disediakan mendedahkan perubahan struktur dengan penambahan grafen oksida dan peningkatan dalam diameter nanoserat diperhatikan dengan peningkatan kandungan *p*-SOCX. Analisis luas permukaan melalui kaedah Brunauer, Emmet, dan Teller (BET) menunjukkan bahawa luas permukaan spesifik nanofiber selulosa asetat/graphene oksida/*p*-sulfonat kalix[4]arena (CA/GO/SOCX) adalah 12.36 m^2/g. Profil termal nanoserat yang direkodkan melalui analisis termogravimetrik menunjukkan peningkatan sedikit dalam kestabilan termal selepas penambahan grafen oksida kepada selulosa asetat. Walau bagaimanapun, untuk sampel yang mengandungi *p*-SOCX, kestabilan terma berkurangan dengan peningkatan jumlah *p*-SOCX. Beberapa parameter tindak balas seperti pH, dos penjerap, kepekatan pewarna, masa sentuh, dan suhu untuk penyingkiran metilena biru telah disiasat di sini. Hasil menunjukkan peningkatan kapasiti penjerapan MB telah dicapai dengan kehadiran *p*-SOCX berbanding dengan nanoserat selulosa asetat tulen dan nanoserat selulosa asetat/grafen oksida pada pH ideal 8. Dengan kepekatan pewarna MB awal sebanyak 10 mg/L, dos penjerap sebanyak 20 mg/10 mL, dan masa sentuh selama 60 minit pada suhu bilik, iaitu sekitar 293K, kecekapan penyingkiran terbaik telah dicapai, iaitu 88.84%. Model kinetik pseudo-tertib-kedua dianggap lebih tepat untuk menggambarkan tingkah laku penjerapan ion MB berbanding model kinetik pseudo-tertib-pertama dan model difusi intrapartikel. Menurut keputusan penyesuaian dengan persamaan Langmuir, didapati kapasiti penjerapan maksimum CA/GO/SOCX1% mencapai 175.4 mg/g. Telah didapati bahawa penambahan *p*-SOCX pada kepekatan yang betul dapat meningkatkan secara signifikan kapasiti penjerapan dan peratusan penyingkiran ion MB. Walau bagaimanapun, kajian mengenai kebolehgunaan menunjukkan penurunan yang ketara sepanjang lima kitaran iaitu dari 92% hingga 73%, 59%, 49%, dan 43% selepas lima kali penggunaan. Dapat disimpulkan bahawa CA/GO/SOCX1% berpotensi digunakan sebagai penjerap untuk penyingkiran pewarna MB dari larutan akuatik.

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Mohd Haniff bin Wahid, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Zulkarnain bin Zainal, PhD
Professor, Chm
Faculty of Science
Universiti Putra Malaysia
(Member)

Shahrul Ainliah binti Alang Ahmad, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

Mohd Rashidi bin Abdull Manap, PhD.
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

____
ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 14 December 2023

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LIST OF ABBREVIATIONS

^1H NMR	Proton Nuclear Magnetic Resonance
CA	Cellulose acetate
GO	Graphene oxide
p-SOCX	*p*-sulphonated calix[4]arene
MB	Methylene blue
FTIR	Fourier Transform Infrared
SEM	Scanning Electron Microscopy
BET	Brunauer-Emmett-Teller
TGA	Thermogravimetric analysis
PZC	Point of zero charge
PFO	Pseudo-first-order
PSO	Pseudo-second-order

CHAPTER 1

INTRODUCTION

1.1 Water pollution

There is a major global concern towards the declination of water quality mainly caused by industrialization, and the expansion of global population. Industrial effluents, such as, organic and inorganic contaminants, are among the reasons of worldwide shortage access to safe and uncontaminated water (Rajendran et al., 2022). According to the WHO, almost 1.9 billion people are in danger because of lack of access to clean water supply. Industries dealing with paper, plastics, textiles, food, cosmetics, and leather, have caused serious contamination problems due to the usage of dyes. Dye discharge from textile industry results in biological and chemical changes of water, which may bring harm to marine life and other living organisms (El-Barbary et al., 2021). There are two primary categories of chemical contaminants that could be present in drinking water. The initial category encompasses chemical dyes like amaranth, methyl orange, methylene blue, and tartrazine, while the second category encompasses heavy metals, which include Cu(II), Pb(II), Fe(II), Cd(II), Se(IV), and Cr(VI).

1.2 Methylene blue

Dyes are colored organic compounds, comprising auxochromes and chromophores as their functional groups. Chromophores and auxochromes make up the bulk of the dye molecular structure. Dye color is determined by chromophores (such as acetylene, ethylene, esters, carbonyls, acids, nitrile groups, etc.), while the dye intensity is determined by auxochromes (such as -OR, NHR, OH, $-NR_2$ - and $-NH_2$,). Dyeing is the addition of a colored organic compound to fibers, through chemical and/or physical processes, which allow the substance to aggressively absorb light in the visible spectrum (Soudagar et al., 2021). Dyes can be divided into three primary categories, namely, cationic, anionic, and non-ionic. Methylene blue (MB) is among the dyes that has many applications beyond the realm of chemistry and biology (Figure. 1.1). MB is a nitrogen and sulphur-containing polyaromatic ring molecule with a wide range of applications. MB has the formula $C_{16}H_{18}N_3SCl$, and it turns water into a brilliant shade of blue when dissolved (Mahmoud and Abdelwahab, 2019, Soudagar et al., 2021). Other names for MB include methylthioninium chloride, methylene blue chloride, urolene blue, Swiss blue, methylene blue N, Bleu de methylene and Basic blue -9. MB is extremely soluble in water, and its hydrated state consists of three water molecules for every methylene blue molecule (Soudagar et al., 2021).

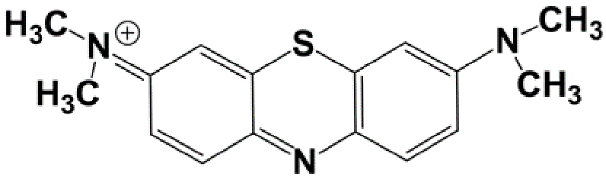

Figure 1.1: Chemical structure of methylene blue (MB)

1.3 Wastewater treatment

Variety of techniques, which can be categorized into biological, physical and chemical processes have been applied as initiatives to get rid of these dyes in aqueous systems (Goswami and Das, 2019). Biological approaches, for example, include dye bioremediation through microorganisms like bacteria or fungi, or by using isolated enzymes in both aerobic and anaerobic processes (Li et al., 2022). On the other hand, oxidation processes, and electrochemical destruction are examples of chemical methods. On many occasions, these techniques are more effective than biological ones; however, they have the drawback of requiring specialised hardware and chemical reagents, and they also produce harmful byproducts. The most well-known chemical wastewater treatment technologies include ion exchange, precipitation, reverse osmosis, adsorption, flocculation, and filtration (Li et al., 2022b).

Since many of the alternatives to adsorption have huge running costs and produce solid waste, of which presents a challenge, it is clear that adsorption is the preferred option for the waste treatment (Stephen et al., 2011). To ensure the success of the adsorption method, the adsorptive interface between the polluted solution and the adsorbents must exist. The adsorptive interactions between the adsorbent and the adsorbed species require a sufficiently large area at the interface between the two phases (Chung et al., 2022). In addition, adsorption includes physisorption and chemisorption, and ideally, adsorbents should remove dye efficiently and be easily regenerated (Li et al., 2022b). Dye adsorption studies have focused on a wide variety of adsorbents, such as zeolites, silica-based materials, metal-organic frameworks, polymeric materials, clay-organic composites, and carbons (graphitic carbon, activated carbon, carbon nanoparticles, biochar, carbon xerogels,).

Over the past few decades, natural polymers such as cellulose, starch, chitin, chitosan, and pullulan have garnered significant interest as adsorbent materials for dye removal. This is attributed to their biodegradability, cost-effectiveness, compatibility with living systems, and lack of environmental harm. Among these natural polymers, polysaccharides have gained prominence due to their diverse range of applications. Within this group of polysaccharides, cellulose and its derivatives hold a significant position (Shaheen et al., 2022). Cellulose acetate (CA) has been utilised in the realm of wastewater treatment, encompassing tasks such as the removal of heavy metals, degradation of dyes, and isolation of diverse organic compounds. Furthermore, the efficacy of CA can be elevated through the amalgamation with alternate polymers and/or the integration of inorganic constituents (Karamipour et al., 2020). On the other hand, graphene oxide (GO) offers advantages such as large surface area and has garnered significant attention among researchers (Liu et al., 2019). GO possesses amphiphilic properties which enables it to interact with a wide range of substances. Heavy metal ions removal utilising CA/GO nanofibers have been reported previously (Omer et al., 2022a). To further enrich the CA/GO composite with adsorptive sites, in this study functionalisation of GO with *p*-sulphonated calix[4]arene was proposed.

This study introduces the use of CA/GO coupled with *p*-sulphonated calix[4]arene (*p*-SOCX) in the form of nanofibers, as an adsorbent to remove MB dye. This study delved into optimising the adsorption process by studying key factors such as adsorbent dosage, contact time, initial concentration of the dye, temperature and pH. Through mathematical models and surface plots, the interactions between these factors were analysed. Ultimately, the adsorption of MB by CA/GO with *p*-SOCX was explored under optimal conditions. To comprehend the capabilities of CA/GO/SOCX in MB adsorption, kinetics and isotherms were examined.

1.4 Problem statement

Facile, ease of operation and effective dye removal technique is in demand to address issues concerning MB removal form wastewater. Individuals who have been exposed to MB dyes were reported to experience symptoms of headaches, anxiety, somnolence, confusion, vertigo, visual abnormalities, and depression as it exceeds the licit limits of MB in water (> 1 mg L^{-1}) (Mahmoud and Abdelwahab, 2019, Chen et al., 2020, Heidari et al., 2023). At present, the textile sector uses 7,105 tonnes of dye annually, using more than 10,000 distinct types, and their eventual release is into waste systems. Such problematic issue must be solved and thus, numerous initiatives have been launched on a global scale to address the issue of toxic dyes in aquatic environments.

1.5 Significance of the study

The possibility to mix composites into a mixture and through application of potential difference to produce nanofiber is the beauty of the electrospinning technique. One can plan to combine different properties of materials in a simple way through this approach. Furthermore, electrospinning offers fast, cheap, and simple way to produce nanofibers with precise controls over the fiber diameter, compared to alternative approaches. Also, the formation of continuous fibers is simple, and no involvement of catalyst that will leave behind residuals. Taking this advantage, herein, CA was combined with GO and *p*-sulphonated calix[4]arene to produce a novel composite nanofiber for the effective removal of MB from wastewater. CA are commonly prepared due to their high ejection efficiency for nanofiber formation, low cost, simple preparation method, and possess antifouling behavior (Mahmoud and Abdelwahab, 2019). These molecules are interconnected through β (1 → 4)–glycosidic linkages and contain three OH groups within each hydroglucose unit. These OH groups serve as distinct sorption sites that are active, thereby augmenting the capacity for efficient dye removal (Kausar et al., 2023). The active functional groups present in CA make it amenable to modifications with other functional groups, leading to improved adsorption characteristics. Consequently, many studies have been dedicated to exploring modification of CA, together with GO through grafting and composite formation (Basha et al., 2022). Meanwhile, to further increase adsorptive sites, calixarene possessing sulphonate groups was added into the mixture namely, *p*-SOCX. Calixarenes are one type of supramolecules that possess a unique three-dimensional (3D) structure with the potential in the host/guest systems for various applications. Different strategies have been used to boost their attraction towards guest species, and one of them is by fixing the calixarenes on the polymeric supports. This method of designing compounds has several advantages, including; the ability to control and predict the number and the structure of active sites (Mazinani et al., 2022).The combined CA/GO/SOCX material offers plenty of potential adsorptive sites such as hydroxyl groups where MB dye contains $N(CH_3)_2$ groups, allowing lone pair electrons on N to form hydrogen bonds with *p*-SOCX hydroxyl groups. In water, the sulphonate group in *p*-SOCX binds to cationic N^+ of MB, a strong electrostatic interaction where SO_3 group of *p*-SOCX coordinates with MB cationic groups. This robust electrostatic interaction contributes to MB dye extraction. Due to the flat structure of MB, it easily adsorbs onto *p*-SOCX through π-π interactions (Kamboh et al., 2019). Thanks to their unique guest-host structures, these materials can effectively and selectively coordinate with MB ions through cation-π, ion-dipole, and other interactions (Wang and Zhuang, 2020). Since the calixarene cavity is capable of molecular recognition in solutions, and since water-soluble calixarenes contain sulphonate groups, as reported by Sasaki et al., (2006), they are of significant interests for applications in the remediation of contaminated groundwater and industrial effluents.

1.6 Objective of the study

1.6.1 General objective

The objective of this research was to produce cellulose acetate nanofibers, functionalised with graphene oxide and *p*-sulphonated calix [4] arene, as the adsorbent, for the MB removal from aqueous solutions.

1.6.2 Specific objectives

The objectives of this work are as follow**:**

1. To prepare and characterise the cellulose acetate/graphene oxide/*p*-sulphonated calix[4]arene composite nanofiber using the electrospinning technique.

2. To optimise the reaction conditions for Methylene Blue dye removal from aqueous solutions.

3. To investigate the dye adsorption kinetics and isotherm removal characteristics.

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