FABRICATION AND CHARACTERIZATION OF ELECTROSPUN CELLULOSE ACETATE/GRAPHENE OXIDE/SODIUM DODECYL SULPHATE NANOFIBER MEMBRANE FOR PHOSPHATE ION REMOVAL

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DEDICATION

This thesis is dedicated to my husband, Muhammad Syafiq bin Mohd Najib who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my parents, for their endless love, support and encouragement.

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ABSTRACT

Excessive amounts of nutrient content such as phosphorus can pollute the river, harm the ecosystem, and if the issue persists, even contribute to eutrophication. It is essential to monitor and control the concentration of phosphorus elements (< 5mg/L) for sustaining good water quality andreducing water pollution of natural water. Hence, the main idea of this research is to fabricatean adsorbent based on cellulose named cellulose acetate/graphene oxide/sodium dodecylsulphate (CA/GO/SDS) through hydrogen bonding to adsorb phosphate ions from aqueous solution. A 13% (w/v) CA solution and a 13% (v/v) GO/SDS solution were prepared using a binary solvent system acetone:*N-N* dimetylacetamide (Ac:DMAc) with a ratio of 2:1. The mixture of the two solutions was electrospun in a fume hood to produce an electrospun CA/GO/SDS nanofiber membrane. Based on the characterization of electrospun CA/GO/SDS nanofiber membrane, it was proven that GO/SDS were successfully incorporated onto the CAas demonstrated by Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR), X-ray Diffraction (XRD) and X-ray Photoelectron Spectroscopy (XPS). In a Field Emission Scanning Electron Microscopy (FESEM), the electrospun CA nanofiber membrane is pure white, while the electrospun CA/GO/SDS nanofiber membrane is not as white as that of CA since the GO content darken the membrane. A study of optimization for the adsorption process was conducted by varying the pH, immersion time and mass of membrane. In our study, hydroxyl groups on the surface of electrospun CA/GO/SDS nanofibermembrane assisted the removal of phosphate from aqueous solution through ligand exchange mechanism between the hydroxyl group and negatively charged phosphate ions. The electrospun CA/GO/SDS nanofiber membrane exhibited excellent ion removal properties dueto the hydrophilic properties as well as their smooth surface membrane. The solution reached a maximum removal efficiency of 87.1% at pH 7 since the surface charge of the membrane was negative, promoting the specific adsorption of phosphate ions to occur where phosphate ions replaced hydroxyl groups on the membrane surface. A kinetic study revealed that 180 min of immersion time could adsorb about 87.2% of phosphate onto the membrane. The maximum phosphate removal on 0.05 g electrospun CA/GO/SDS nanofiber membrane was 175.44 mg-P/g. The data showed that adsorption kinetic followed the pseudo- second-order model whereas the adsorption mechanism followed the Langmuir model.

ABSTRAK

Jumlah kandungan nutrien yang berlebihan seperti unsur fosforus boleh menyebabkan pencemaran sungai dan membahayakan ekosistem, malah ia turut menyumbang kepada eutrofikasi sekiranya masalah ini berlarutan. Kepekatan unsur fosforus haruslah dipantau supaya tidak melebihi nilai 5 mg/L bagi mengekalkan kualiti air dan seterusnya dapat mengurangkan pencemaran air semula jadi. Oleh itu, penyelidikan ini bertujuan untuk memfabrikasi penjerap yang berasaskan selulosa iaitu selulosa asetat/grafen oksida/ sodium dodesil sulfat (CA/GO/SDS) melalui ikatan hidrogen untuk menyerap ion fosfat daripada larutan akueus. Larutan CA 13% (w/v) dan larutan GO/SDS 13% (v/v) telah disediakan dengan menggunakan sistem pelarut binari (aseton:*N-N* dimetilasetamida) (Ac:DMAc) dengan nisbah 2:1. Campuran kedua-dua larutan tersebut telah diputar secara elektro di dalam kebuk wasap untuk menghasilkan sehelai membran nanofiber elektroputaran CA/GO/SDS. Berdasarkan kepada pencirian membran nanofiber elektroputaran yang ditunjukkan oleh spektroskopi pemantulan jumlah yang dilemahkan (ATR-FTIR), pembelauan sinaran-X (XRD) dan fotoelektron sinaran-X (XPS) membuktikan bahawa GO/SDS berjaya disatukan bersama CA. Imej yang dipaparkan melalui mikroskopi electron di dalam mikroskopi elektron pengimbasan pelepasan medan (FESEM) menunjukkan bahawa membran nanofiber CA elektroputaran berwarna putih tulen, manakala membran nanofiber elektroputaran CA/GO/SDS kelihatan lebih gelap dengan kehadiran GO. Kajian optimum untuk proses penjerapan telah dijalankan dengan kepelbagaian pH, masa rendaman dan jisim membran. Dalam penyelidikan ini, kumpulan hidroksil yang terdapat pada permukaan membran nanofiber elektroputaran CA/GO/SDS membantu menyerap ion fosfat daripada larutan akueus melalui mekanisma pertukaran ligan antara kumpulan hidroksil dan ion fosfat di mana kedua- duanya bercas negatif. Membran nanofiber elektroputaran CA/GO/SDS mempunyai keupayaan penyingkiran ion fosfat yang sangat baik kerana sifat hidrofilik dan permukaannya yang licin. Penyerapan ion fosfat yang maksimum iaitu 87.1% berlaku pada pH 7 kerana permukaan membran bercas negatif yang dapat menggalakkan penjerapan ion fosfat, di mana ion fosfat lebih cenderung untuk menggantikan kumpulan hidroksil yang terdapat di atas permukaan membran nanofiber elektroputaran CA/GO/SDS. Kajian kinetik menunjukkan bahawa 180 minit merupakan masa rendaman yang paling optimum untuk menjerap 87.2% ion fosfat daripada larutan akueus. Penjerapan ion fosfat yang maksimum (175.44 mg-P/g) diperoleh apabila menggunakan membran nanofiber elektroputaran CA/GO/SDS yang berjisim 0.05 g. Data yang diperoleh menunjukkan bahawa kinetik penjerapan mengikuti model pseudo-tertib kedua manakala mekanisma penjerapan mengikuti model Langmuir.

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LIST OF ABBREVIATIONS

ATR-FTIR	-	Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy
EDX	-	Energy Dispersive X-ray Spectroscopy
FESEM	-	Field Emission Scanning Electron Microscopy
LOD	-	Limit of Detection
LOQ	-	Limit of Quantification
RSD	-	Relative Standard Deviation
UV-VIS	-	Ultraviolet Visible Spectrophotometry
XPS	-	X-ray Photoelectron Spectroscopy
XRD	-	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Research Background

Excessive discharge of phosphate in agricultural and industrial areas can cause possible land and water contamination. The Malaysia river monitoring assessment from DOE reveals clean and slightly polluted rivers in Malaysia classified as Water Quality Index Class II and Class III respectively [2]. In the agricultural sector, fertilizer particularly organophosphate and animal waste are main factors that cause pollutants to water course [3]. Wastewater from industrial sector, such as steel and chemical industry contains high amount of heavy metals and can lead to severe water pollution. Improper and ineffective wastewater treatment can contribute to river water pollution. Excessive phosphorus in water bodies may increase the growth of algae and aquatic plant in a pond that leads to eutrophication phenomena and these phenomena can harm the river water quality [4]. Malaysia has set a limit level of 5 mg/L phosphorus in an enclosed water of body and 10 mg/L of phosphorus released to rivers as an acceptable condition for effluent [5]. The Malaysian effluent standard has been permitted to release higher concentration of phosphorus into waters in comparison to the European Union (EU) and United State Environmental Protection Agency (USEPA) effluent standards. According to the EU, the limit values of phosphorus for effluent discharge in wastewater treatment plants are in a range of 1.0-2.0 mg/L [6]. The effluent limit of total phosphorus according to USEPA Clean Water Act is 0.5-0.8 mg/L [7].

Currently, the common methods of phosphate removal include chemical precipitation, biological method, membrane separation and adsorption [8]. The adsorption process is a favourable method for decreasing the level of phosphate ions in water due to its low cost, simplicity of operation and good efficiency [9]. Although many adsorbents have been utilized for phosphate removal, it is still important to develop an alternative low-cost adsorbents for phosphate removal in order to ensure sustainable environment. Polymeric adsorbents have been increasingly used for contaminants removal due to their wide variation in surface chemistry, regenerability on site, and porosity and they also can be produced by many techniques [10]. Of these, electrospinning is a particularly low cost and versatile method to produce adsorbent in terms of electrospun nanofiber membranes (ENMs) [11]. Nanofibers prepared by electrospinning method are good candidates for the adsorption of various contaminants from water due their high porosity and large specific area [12]. Many studies have been conducted using ENMs for phosphate removal and most of them have achieved their objectives [13]–[15]. Other studies using ENMs for water bacteria removal [16], arsenate removal and heavy metals removal [17].

The electrospinning method produces a jet flow of a non-Newtonian fluid under a high-voltage electrostatic field. The jet deposits on the receiving plate after the volatilization and solidification of solvents, and nanofibers are thus obtained [18]. The method is easy to operate and widely used in the fields of catalysis, sensing, and adsorption. When the diameter of the membrane is reduced to nanoscale range, it gains various intriguing qualities such as high surface area to volume ratio, nano-sized pores, and outstanding mechanical properties have been imparted to the matrix [19]. These characteristics make ENMs a good choice for adsorption process, which is an excellent methods for removing undesirable species from polluted water or preventing dangerous toxins from entering the environment and human body.

In addition, the adsorption capacity of adsorbents can be improved by modification process such as introducing various functional groups (carboxyl, tetrazine, sulfonic, amino, and hydroxyl groups) on the ENMs surface with selective adsorption affinity to targeted adsorbates [20]. Modifications to the ENMs can improve the characteristics of nanofibers such as the availability of functional groups on the membrane surface. The oxidation process, plasma treatment, solvent vapour treatment, surface coating and solution blending are some of the additional surface modification techniques [21][22]. Surface coating is now the favoured approach for preventing fouling of reverse osmosis and nanofiltration membranes, and the coatings are durable enough to be called "permanent" [23]. The coating solution may not only covered the membrane surface, but also infiltrated into the open structures of the membrane, resulting in an increase in the number of active sites for adsorption process. The blending solution is a promising method to modify scaffolds containing polymers which lack suitable surface properties since it can enhance the biocompatibility, hydrophilicity and toughness while maintaining mechanical strength of polymer [24].

In this study, we have developed a novel flexible electrospun CA/GO/SDS nanofiber membrane for a highly effective phosphate ions removal via electrospinning technique. The modification processes have been done by incorporating GO/SDS solution into CA solution by solution coating technique and solution blending technique. Specially, the GO/SDS were incorporated into CA to promote more hydroxyl groups on the surface of the membrane and trigger the phosphate ions adsorption via ligand exchange mechanism. Characterization on the resulting electrospun CA/GO/SDS nanofiber membranes were then tested to confirm the hypothesis that the hydroxyl groups on the surface of the membrane had a significant impact on the phosphate ions removal. This present work studies the capability of electrospun CA/GO/SDS nanofiber membrane for the adsorption of phosphate ions in a batch system under different adsorption variables such as pH, immersion time and mass of membrane. The kinetic and equilibrium data of phosphate ions adsorption by the electrospun CA/GO/SDS nanofiber membrane were analyzed by the pseudo-first order and pseudo-second order kinetic Lagergren model and Langmuir and Freundlich, isotherm models, respectively.

1.2 Problem Statement

Phosphorus (P) is a major nutrient contaminant in water. It enters water bodies through mining, industrial and agricultural activities, and sewage discharges. Excessive concentrations of P in water cause eutrophication [25],which is defined as the enrichment of water bodies by nutrients and the consequent deterioration of quality due to the luxuriant growth of plants such as algae and its repercussions on the ecological balance of the waters affected [26]. Although both nitrogen (N) and P are considered to be the limiting nutrients for eutrophication, some algae are efficient in the fixation of atmospheric N and hence P often becomes the potentially limiting nutrient in freshwaters [27]. In advanced stages of eutrophication, dissolved oxygen can become depleted to dangerously low levels causing fish death when algae decay [28]. The large algae biomass produced by eutrophication can also affect water treatment by blocking filters or passing through them causing bad odour and taste in treated water [29]. Blue-green algae can produce compounds that are toxic to fish and other aquatic life [30]. These conditions are also potentially risky to human health, resulting from consumption of shellfish contaminated with algal toxins or direct exposure to waterborne toxins [31]. Therefore, it is of importance and urgency to control the discharge and reduce the total amount of the phosphate due to its harmful organism origin [32].

Different techniques have been developed and used for phosphate removal in the world such as chemical, biological and physical [33][34]. Among these, chemical and biological treatments have many drawbacks such as side reactions, side products, pretreatment, and many more [35], while treatment through adsorption is considered one of the more economical and effective procedures [36]. Adsorption can effectively reduce the phosphate level at low cost as compared with other methods [37], [38]. The adsorption process is generally considered to be an effective water treatment option because of convenience, ease of operation, and simplicity of design and economics [38].

Nanofibrous membranes are substantially more effective for surface adsorption of pollutants from contaminated water than conventional porous affinity membranes because of their large surface-to-volume ratio and tunable functionality[39]. Electrospinning produces a high specific surface area with great adsorption capacity. Electrospinning is a quick and easy way to make continuous fibres with diameters ranging from micrometres to few nanometers [40]. In particular, electrospun nanofibers with membrane structures might be employed as an efficient water treatment material due to their high porosity, interconnected pore structure, simplicity of incorporation of unique functionality, and safety [41].

The current flat sheet polyethersulfone (PES) membrane, which was employed in a prior research study, is not capable of removing tiny particle ions with nano-scale diameter [42], [43]. Therefore, electrospun CA nanofiber membrane must be modified to improve the effectiveness in removing phosphate ions from aqueous solution. This modification strategy tends to improve membrane phosphate removal effectiveness, and it will be more significant when hydroxyl groups on the electrospun membrane become more accessible [43].

1.3 Research Objectives

The objectives of the research are :

(a) To fabricate and to characterize electrospun cellulose acetate (CA) nanofiber membrane modified with graphene oxide/sodium dodecyl sulphate (GO/SDS).

(b) To perform adsorption study of electrospun CA/GO/SDS nanofiber membrane for phosphate ions removal.

(c) To validate the ability of electrospun CA/GO/SDS nanofiber membranes for phosphate ions removal from aqueous solution.

1.4 Scope of Study

The study began with the preparation of CA solution for fabricating the electrospun CA nanofiber membrane which was done by using electrospinning technique. The operating parameters on electrospun fiber diameters are dictated by varying the parameter values in an electrospinning hypothetical model, such as, solution parameter, processing parameter and ambient parameter. The binary solvents used for solution parameter to dissolve CA powder were acetone:*N-N,* dimetylacetamide (Ac:DMAc) and acetone:dimethyl sulfoxide (Ac:DMSO) mixture. Both binary solvents were set at 2:1 ratio. For processing parameter, the electrospinning was conducted at 20 kV, 150 cm of working distance, and 2mL/h solution flow rate, respectively. Humidity and temperature have been considered for the ambient parameter when producing fine fibers from CA solution. After that, the CA was blended and coated with GO/SDS for modification to boost surface charge in the electrospun nanofiber membrane.

The second main step in this study was the characterization of electrospun CA/GO/SDS composite nanofiber membrane. The structures and properties of the electrospun nanofiber membrane have been characterized by various methods. The Attenuated Total Reflectance – Fourier Transform Infrared Spectroscopy (ATR-FTIR) spectra were obtained acquired by putting the sample on the crystal and collecting the data from software in ATR-FTIR mode, in the range 650-4000 cm^{-1} with a resolution of 8 cm^{-1} and at 120 scans. The elemental composition (carbon, oxygen and phosphorus) within the upper 1-10 nm of the surface was determined by X-ray Photoelectron Spectroscopy (XPS) and the structural changes in the samples was identified by X-ray Diffraction (XRD) analysis. The surface morphology of the membrane, before and after phosphate loading have been examined by Field Emission Scanning Electron Microscopy (FESEM). For FESEM imaging, the membrane samples were coated with gold prior to FESEM examination. The FESEM used for the characterization was equipped by EDX spectroscopy system that was used to measure the composition of the elements constituting the membranes. The phosphate concentration has been determined by using spectrophotometer NANOCOLOR VIS II. Finally, contact angle measurement has been done to measure the hydrophilicity of surface of the membrane.

To study the ability of electrospun CA/GO/SDS nanofiber membrane in phosphate ions removal, the kinetic and sorption isotherms have been investigated by immersing the nanofiber membrane in phosphate solutions at desired concentration of 25mg/L. A study of optimization for the adsorption process was conducted by varying the pH, immersion time and mass of membrane. The phosphate concentration has been measured by a simple and sensitive spectrophotometric method based on a blue color of phosphate solution using orthophosphate NANOCOLOR® standard tests. The difference between the initial and final concentration of phosphates in aqueous phase will be used to compute the quantity adsorbed phosphate at equilibrium (q_e). The expected mechanism that will be involved in the process of phosphate ions uptake is the ligand exchange between the membrane functional groups. The validation methods were carried out by considering the wavelength accuracy check, limit of detection and quantification (LOD and LOQ) and the repeatability of measurement throughout the study.

1.5 Significance of Study

The study on the fabrication of electropsun CA/GO/SDS nanofiber membrane is very important as it can contribute to the application in the removal of excessive phosphorus by adsorption process for controlling water eutrophication. This study has a huge impact to increase awareness among society on environmental pollution issue and health problems by creating a useful novel methodology that could be used to efficiently decontaminate phosphates with minimal environmental impact. Finally, this study is in line with Sustainable Development Goals 6 (SDG 6) to improve water quality by reducing pollution before 2030.

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