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REMOVAL OF β-CAROTENE FROM PALM OIL MILL EFFLUENT BY MOLECULAR IMPRINTED POLYMER BASED ON CYCLODEXTRIN- CO-VINYLCARBAZOLE AND CYCLODEXTRIN-CO-HYDROXYETHYL METHACRYLATE

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REMOVAL OF β-CAROTENE FROM PALM OIL MILL EFFLUENT BY MOLECULAR IMPRINTED POLYMER BASED ON CYCLODEXTRIN-CO-VINYLCARBAZOLE AND CYCLODEXTRIN-CO-HYDROXYETHYL METHACRYLATE

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Palm oil mill effluent (POME) the most significant pollutant in the form of wastewater, if not managed properly, it could have negative effects on the environment include the emission of biogas and water pollution which comes from discharging the brownish tick POME to the water bodies. Discharge of dark brownish colored of POME into water bodies will prevent the growth of aqua organism by reducing the penetration of sunlight and affect the photosynthetic activity. Such concern provides incentive to remove color from POME. In recent years, molecular imprinted polymers (MIP) attracted wide attention in treatment of wastewater due to their capability for molecular recognition, easiness of preparation, stability and cost-effective production. In this research, MIPs were synthesized by bulk polymerization method using β-cyclodextrin (β-CD) as monomer, 9-vinylcarbazole (9VC) and 2-hydroxylethyl methacrylate (HEMA) as co monomers, Toluene2,4-diisocyanate (TDI) as cross-linker, N,N-Dimethylformamide (DMF) as porogen solvent and Benzoyl peroxide (BPO) as initiator. The temperature used during polymerization process was 70ºC. The obtained MIP was crushed, sieved and washed to remove the template. Increase the ratio of monomer to cross-linker lead to increase the yield weight of the MIPs. The optimal ratio of monomer to cross-linker was 1:5:58 and 1:17:58 for MIP-β-CD-9VC and MIP-β-CD-HEMA respectively. FTIR result showed that MIP (Molecular Imprinted Polymer) and NIP (Non-Imprinted Polymer) have similar characteristic peak with different peaks intensity, indicating the similarity in the backbone structure of polymerization. TGA result displayed high thermal stability with final decomposition at 320ºC and 307ºC for MIP-β-CD-9VC and MIP-β-CD-HEMA respectively. FESEM shows that MIP has rough surface while NIP possess densely packed particles with smooth surface. From BET result, the surface area for MIP-β-CD-9VC is higher than NIP-β-CD-9VC, while the surface area for MIP-β-CD-HEMA is smaller compared with NIP-β-CD-HEMA. The pH study shows that sorption of β-

carotene increased with decreasing the pH of POME and the maximum sorption capacities achieved at pH 2 were 10 μg/g and 8 μg/g for MIP-β-CD-9VC and MIP-β-CD-HEMA, respectively. Also, the sorption of β-carotene increased with increasing the dosage of MIP, and the maximum sorption achieved by using 500 mg of MIP. Both MIP-β-CD-9VC and MIP-β-CD-HEMA followed Freundlich isotherm with R^2=0.9688 and R^2=0.9817 respectively. Besides that, the sorption of β-carotene by MIPs is fast within few minutes and both MIP-β-CD-9VC and MIP-β-CD HEMA followed the pseudo second order with R^2=0.9999. The reusability study for MIP-β-CD-9VC and MIP-β-CD-HEMA were tested five times and no significant loss in sorption capacity. However, MIP-β-CD-HEMA showed higher reusability compared with MIP-β-CD-9VC.

A new type of molecular imprinted polymer as a sorbents for removal of β-carotene from palm oil mill effluent (POME) was successfully synthesized. The synthesized MIP-β-CD-9VC and MIP-β-CD-HEMA showed several characteristic such as high thermal stability, fast sorption kinetic and good reusability. Thus, MIP-β-CD-9VC and MIP-β-CD-HEMA can be potentially used as a sorbents for the removal of β-carotene from palm oil mill effluent (POME).

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

PENYINGKIRAN BETA-KAROTEN DARI EFLUEN KILANG MINYAK KELAPA SAWIT OLEH POLIMER TERCETAK MOLEKUL BERDASARKAN CYCLODEXTRIN-CO-VINYLCARBAZOLE DAN CYCLODEXTRIN-CO-HYDROXYETHYL METHACRYLATE

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Efluen kilang minyak kelapa sawit (POME) adalah bahan pencemar yang paling signifikan dalam bentuk air sisa, dan jika ia tidak diuruskan dengan baik, ia boleh memberi kesan negatif terhadap alam sekitar, termasuk pelepasan biogas dan pencemaran air yang terhasil oleh pengeluaran POME tebal berwarna coklat ke dalam air. Kebimbangan sebegitu memberikan insentif kepada usaha untuk menjalankan rawatan terhadap POME. Pelepasan warna gelap POME ke dalam badan airakan menghalang pertumbuhan organisma akuatik dengan megurangkan penembusan cahaya matahari dan menjejaskan aktiviti fotosintesis. Dalam tahun-tahun kebelakangan ini, polimer tercetak molekul (MIP) telah menarik perhatian luas dalam rawatan air sisa disebabkan oleh keupayaannya untuk mengenal pasti molekul, penyediaannya yang mudah, kestabilannya dan keberkesanan kos pengeluarannya. Dalam kajian ini, MIP disintesis dengan kaedah pempolimeran pukal dengan menggunakan β-cyclodextrin (β-CD) sebagai monomer, 9-vinylcarbazole (9VC) dan 2-hydroxylethyl methacrylate (HEMA) sebagai monomer bersama, Toluene 2,4-diisocyanate (TDI) sebagai penghubung silang, N,N-Dimethylformamide (DMF) sebagai pelarut porogen dan Benzoyl peroksida (BPO) sebagai pemula. Suhu setinggi 70ºC digunakan semasa proses pempolimeran. MIP yang diperolehi dihancurkan, disaring dan dibasuh untuk mengeluarkan templat tersebut. Peningkatan nisbah monomer kepada penghubung silang telah menyebabkan kenaikan dalam berat hasil MIP. Nisbah optimum monomer kepada penghubung silang adalah 1:5:58 dan 1:17:58 untuk MIP-β-CD-9VC dan MIP-β-CD-HEMA masing-masing. Keputusan FTIR menunjukkan bahawa oleh kerana MIP (polimer tercetak molekul) dan NIP (polimer tidak dicetak) mempunyai ciri puncak yang sama walaupun keamatan puncak mereka berbeza, maka ini menunjukkan bahawa struktur tulang belakang pempolimeran mereka adalah sama. Keputusan TGA menunjukkan kestabilan haba yang tinggi dengan penguraian akhir pada 320ºC dan 307ºC untuk MIP-β-CD-9VC dan MIP-β-

CD-HEMA masing-masing. FESEM menunjukkan bahawa MIP mempunyai permukaan yang kasar manakala NIP mempunyai zarah-zarah yang disusun padat dengan permukaan yang licin. Hasil BET menunjukkan bahawa MIP-β-CD-9VC mempunyai luas permukaan yang lebih tinggi daripada NIP-β-CD-9VC, sementara luas permukaan MIP-β-CD-HEMA adalah lebih kecil dibandingkan dengan NIP-β-CD-HEMA. Kapasiti penyerapan optimum semasa kajian pH telah dicapai pada pH 2 untuk kedua-dua MIP-β-CD-9VC dan MIP-β-CD-HEMA. Juga, penyerapan β-karoten telah meningkat dengan kenaikan dos MIP, dan penyerapan maksimum yang dicapai dengan menggunakan 500 mg MIP. Kedua-dua MIP-β-CD-9VC dan MIP-β-CD-HEMA telah mengikut isotherm Freundlich dengan R^2 =0.9688 dan R^2= 0.9817 masing-masing. Selain itu, penyerapan β-karoten oleh MIP adalah cepat dan hanya memakan masa beberapa minit sahaja dan kedua-dua MIP-β-CD-9VC dan MIP-β-CD HEMA telah mengikut urutan kedua pseudokod dengan R^2=0.9999. Kajian kebolehgunaan semula untuk MIP-β-CD-9VC dan MIP-β-CD-HEMA telah diuji sebanyak lima kali dan tiada kehilangan kapasiti penyerapan yang ketara berlaku. Walau bagaimanapun, MIP-β-CD-HEMA menunjukkan kebolehgunaan semula yang lebih tinggi berbanding dengan MIP-β-CD-9VC.

Sejenis polimer yang dicetak dengan molekul baru sebagai penyerap untuk penyingkiran β-karotene daripada efluen kilang miyak sawit (POME) telah berjaya disintesis. MIP-β-CD-9VC yang disintesis dan MIP-β-CD-HEMA menunjukan beberapa ciri-ciri seperti kestabilan haba yang tinggi, kinetic penjerapan pantas dan boleh digunkan semula. Oleh itu, MIP-β-CD-9VC dan MIP-β-CD-HEMA berpotensi digunakan sebagai sorben untuk penyingkiran β-karotene daripada efluen kilang miyak sawit (POME).

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

9VC	9-Vinylcarbazole
ATR	Attenuated Total Reflectance
BET	Brunaue Emmett Teller
BPO	Benzol peroxide
DMF	Dimethyl formamide
DTG	Differential Thermogravimetric Analysis
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared
HEMA	2-Hydroxyethyl Methacrylate
MIP	Molecular Imprinted Polymer
MIP-β-CD	Molecular Imprinted Polymer-β-Cyclodextrin
MIP-β-CD-9VC	Molecular Imprinted Polymer-β-Cyclodextrin-9-Vinylcarbazole
MIP-β-CD-HEMA	Molecular Imprinted Polymer- β-Cyclodextrin-2-Hydroxyethyl Methacrylate
NIP	Non- Imprinted Polymer
NIP-β-CD	Non-Imprinted Polymer- β-Cyclodextrin
NIP-β-CD-9VC	Non-Imprinted Polymer- β-Cyclodextrin-9-Vinylcarbazole
NIP-β-CD-HEMA	Non-Imprinted Polymer-β-Cyclodextrin-2-Hydroxyethyle Methacrylate
POME	Palm Oil Mill Effluent
TDI	Tolylene-2,4-diisocyanate
TGA	Thermal Gravimetric Analysis
β-CD	β-Cyclodextrin

CHAPTER 1

INTRODUCTION

1.1 Background of the research

The Malaysian palm oil industry is growing rapidly and has become a very important agriculture-based industry. The country is currently one of the largest producers and exporters of palm oil. However, the wet process of palm oil milling consumes a large amount of process water. The production of one tonne of crude palm oil requires approximately 5–7.5 tonnes of water, and more than 50% of the water will end up as palm oil mill effluent (POME) Wu et al. (2007).

POME is acidic and has a pH of between 4 and 5, Chemical Oxygen Demand (COD) of 40,000 -100,000 mg/L, and Biological Oxygen Demand (BOD) of 25,000-60,000 mg/L. The color of the effluent is due to the presence of organic matters such as lignin, phenolics and carotenes (Bello et al. 2013; Ahmed et al. 2015). Palm oil mill effluent (POME) with very dark color will not meet the discharge standard since it has been identified as a major contributor to the pollution load in ground water.

The brownish color of the effluent has raised the public concern regarding its impact on water body. The color pigment present in the effluent has caused colorization of the pipelines in the industry; this reduces possibility of reclamation. As the world continues to find ways to deal with the worsening global water crisis, reclamation of wastewater so that it can be reused in industrial processes would undoubtedly provide an alternative water resource in dealing with problems of water shortage Tan et al. (2017).

Among the technologies which can be used to remove color from POME are advanced oxidation process, membrane filtration, ozonation, ion exchange, and adsorption Zahrim et al. (2014). Among these methods, adsorption is by far the most versatile and frequently used due to its low initial cost, simplicity of design, ease of operation, and insensitivity to toxic. There are number of studies have been conducted to investigate the ability of several adsorbents to remove color from POME, such as activated carbon silica particles and agricultural waste Neoh et al. (2014). However, the ability of these adsorbents to achieve the intended objectives is limited by their selectivity and specificity properties. Furthermore, there has been no report on the reusability of the adsorbents. In the recent past, the usage of molecular imprinted polymers to treat wastewater has been attracting a lot of attention.

Molecularly imprinted polymers (MIPs) are cross-linked polymeric materials that exhibits high selectivity and great affinity for a particular target molecule (template) present during the synthesis process (Asman et al. 2016; Dai et al. 2011). Additionally, MIPs have higher physical strength, robustness, resistance to high pressure and temperature, and inertness against various chemicals (organic solvents, acids, bases, and metal ions). The cost for producing MIPs is low and their life span can be as long as several years at room temperature Cheong et al. (2013). MIPs can be synthesized from a wide range of substances Poma et al. (2010)

The imprinting process occurs in the interaction between a functional monomer and a selected template in a specific solvent which acts as the porogen in the presence of a cross-linker. The template molecule are removed subsequent to polymerization through extensive washing steps to disrupt the interactions between the template and the monomers, thus leaving cavities of binding sites that are complementary to the template in size, shape and position of the functional group. The cavities thus formed can function as selective binding sites for the template molecule (Figueiredo et al. 2016). The resulting MIP can be used in growing number of applications, such as chromatographic separations, solid-phase extraction, and catalysis Xu et al. (2015).

The properties, physical appearance, morphology and the performance of the MIP are determined by reaction conditions such as formulation of MIP's reaction mixture, including choice of cross-linker, functional monomer, porogenic solvent, reaction temperature, and time Cheong et al. (2013).

MIPs can be classified based on whether they are fabricated using the covalent, non-covalent, or semi-covalent method. In the non-covalent imprinting method, which is the most frequently used method; a template is mixed with an appropriate functional monomer, a suitable porogenic solvent, a cross-linker agent and a polymerization initiator. Specific binding sites are formed via self- assembly of the template and the functional monomer, which is capable of forming a fairly stable complex via dipole interaction, hydrophilic interaction, van der Waals forces, hydrogen bonding, ion pair, etc. Following polymerization the template is removed by extracting the polymer with appropriate solvent Kyzas et al. (2014).

1.2 Problem Statement

Palm oil industries have been making significant contribution to the economic growth of South East Asian countries, hence increasing the standard of living of the population in these countries. Unfortunately, it has also been identified as the major sector discharging the largest pollution load in the form of Palm Oil Mills Effluent (POME) Tabassum et al. (2015). POME is considered as one of the most important source of environmental pollution in Malaysia due to the large volume of POME produced by the palm oil mills, along with high concentration of chemical oxygen demand (COD) (65,000 mg/L) and biochemical oxygen demand (BOD) (25,000 mg/L) (Parthasarathy et al. 2016). Among the concerns related to the increasing production of palm oil in Southeast Asia are water pollution, emission of greenhouse gases, and land conversion Ibrahim et al. (2010).

Color remains as one of the major constituents of the discharged effluent, and should be removed for environmental and esthetic reasons. The color of POME not only diminishes the esthetic value of the water bodies, it also interferes with the penetration of light into water, hence causing disturbances to the aquatic ecosystem Zahrim et al. (2017). The discharge of brownish wastewater into water bodies could have adverse effect on aquatic lives since the color filters the light which passes through the water. This will result in reduced photosynthesis activity and less dissolved oxygen. To prevent this impact, color should be removed from POME prior to being discharged into the environment Ratpukdi et al. (2012).

Various methods are available in industries for removal of color from POME such as ozonation, membrane filtration and adsorption using different types of adsorbates. However, there are several disadvantages of these methods for examples ozonation is considered as expensive technique for treatment of POME. Other than that, the major drawback of membrane filtration is fouling. So far, adsorption is an essential process for treatment of POME using commercial activated carbon or waste materials. However, most of these adsorbents are high cost, low reuse, tedious post-processing. Besides, all of these absorbents are applied for non-specific absorption, exhibiting poor selectivity. For these reasons, adsorbents with lower cost, higher reusability and selectivity still essential to be further investigates.

The aim of this research is to prepare molecular imprinted polymer which can be used for selective removal of β-carotene from Palm Oil Mill Effluent (POME). In this research, the MIPs are synthesized using the bulk polymerization method. The parameters studied are pH, dosage studies, sorption kinetics, sorption isotherms and reusability. Due to the versatility and high level of selectivity, and the recognition which can be achieved, the potential of using MIPs as sorbents to remove color (β-carotene) from POME is very promising.

1.3 Objectives of the research

The goal of this research is to remove β-carotene from Palm Oil Mill Effluent using Molecular Imprinted Polymer (MIP). The following specific objectives were designed to achieve this goal;

1- To synthesize molecular imprinted polymer (MIP) by bulk polymerization method using β-cyclodextrin (β-CD) as functional monomer, 9-Vinylcarbazole (9VC) and 2-Hydroxyethyle Methacrylate (HEMA) as co monomers and Tolylene-2, 4-diisocyanate (TDI) as cross linker.

2- To characterize the synthesized MIP by using Fourier-transform infrared spectroscopy (FTIR), Field emission scanning electron microscopy (FESEM), thermal gravimetric analysis (TGA), and Brunaue Emmett Teller (BET).

3- To study the binding capacity of β-carotene toward MIP based on pH, dosage, isotherm, kinetic and reusability.

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PUBLICATION

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