ADSORPTION OF METHYL ORANGE AND METHYLENE BLUE DYES BY LEAD SULPHIDE MODIFIED CALIX[N]ARENES (N = 4, 6, 8)

By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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Various industries around the world such as textile, printing, and leather industries have contributed to major sources of coloured wastewater that can cause severe water pollution. As a result, the current study aims to determine the adsorption behaviour of methyl orange (MO) and methylene blue (MB) dyes based on calix[n]arene-modified lead sulphide (PbS) (n=4, 6, 8) nanoadsorbents under optimal conditions. The calix[n]arene-modified PbS was formed by mixing 3-glycidoxypropyltrimethoxy silane, *p-tert*-butylcalix[n]arene, and PbS in dry toluene with triethylamine as a catalyst. Characterization studies using fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), and energy dispersive X-ray spectroscopy (EDX) revealed a successful synthesis of calix[n]arene-modified PbS. FTIR and EDX showed the presence of O–H stretching vibration, stretching vibration of C-CH_3, C=C aromatic vibration, C-O stretching, carbon (C) and silicon (Si) which are evidence of successful immobilization of *p-tert*-butyl-calix[n]arene on the PbS surface. Morphological change from nanoparticles to flake-like structures is another indication the presence of *p-tert*-butyl calix[n]arene. According to the findings, the pH of point of zero charge (pH_{PZC}) of the calix[4]arene-modified PbS, calix[6]arene-modified PbS, and calix[8]arene-modified PbS were pH 6.80, pH 7.28, and pH 7.31, respectively. Response surface methodology (RSM) was executed to develop a response surface for the optimization of adsorption conditions. The evaluation was further conducted to investigate the interactive effect of various factors (initial concentration, adsorbent dosage, contact time, pH, and temperature) on the adsorption of dye using a central composite design (CCD). The percentage removal of dyes from the water system was used to assess adsorption performance. The results of batch adsorption tests are presented in the study, which include the adsorption capacities, kinetics, and isotherms of the MO and MB adsorption processes. The isotherm parameters were determined using the Langmuir and Freundlich equilibrium models. The isotherm study of MO adsorption by calix[4]arene-modified PbS were best fitted by the Freundlich, indicating adsorption on heterogenous surfaces. In contrast, the isotherm studies of MB adsorption by calix[4]arene-modified PbS, calix[6]arene-modified PbS, and calix[8]arene-modified PbS were best described by the Langmuir, suggesting monolayer adsorption onto

homogenously distributed adsorption sites of the adsorbent surfaces. The fitted model with the maximum adsorption capacity (q_{max}) of 3.268 mg/g, was discovered for the adsorption of MO by calix[4]arene-modified PbS. Meanwhile, the q_{max} for MB adsorption by calix[4]arene-modified PbS, calix[6]arene-modified PbS, and calix[8]arene-modified PbS were found to be 5.850 mg/g, 5.495 mg/g, and 11.90 mg/g, respectively. Pseudo-first order and pseudo-second order of kinetic studies were demonstrated for their quality to fit the data. The adsorption kinetics of MO adsorption by calix[4]arene-modified PbS, MB adsorption by calix[4]arene-modified PbS, calix[6]arene-modified PbS, and calix[8]arene-modified PbS were well described by the pseudo-second order model suggesting that the adsorption on the adsorbent's surfaces occurred via chemical adsorption. The reusability and leaching tests were performed to reduce the adsorbent disposal issues. The results showed that the adsorbents synthesized were stable, and there was a low risk of contaminant (Pb) released into bodies of water. Adsorption mechanisms such as electrostatic interaction, hydrogen bonding interaction, Yoshida hydrogen bonding interaction, and π-π interactions were proposed for dye adsorption on calix[n]arenes-modified PbS. Overall, the research presented novel adsorbents, calix[n]arenes-modified PbS, and a reusability strategy that could be used as alternative adsorbents in MO and MB dye removal and environmentally sustainable dye wastewater treatment.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENJERAPAN PEWARNA METIL JINGGA DAN METILENA BIRU OLEH PLUMBUM SULFIDA DIUBAHSUAI OLEH KALIKS[N]ARENA (N = 4, 6, 8)

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Kepelbagaian industri di seluruh dunia seperti industri tekstil, percetakan, dan kulit, telah menjadi penyumbang utama kepada pelepasan sisa air berwarna yang menyebabkan pencemaran yang teruk terhadap alam sekitar. Justeru itu, kajian ini adalah bertujuan untuk menentukan sifat penjerapan bagi pewarna metil jingga (MO) dan metilena biru (MB) berasaskan nanopenjerap kaliks[n]arena diubah suai pada plumbum sulfida (PbS) (n = 4, 6, 8) dalam keadaan yang optimum. Kaliks[n]arena diubah suai PbS telah disediakan dengan mencampurkan 3-glikidosipropiltrimetoksi silana, *p-tert*-butilkaliks[n]arena, dan PbS dalam toluena kering dengan trietilamina sebagai pemangkin. Kajian pencirian menggunakan spektroskopi inframerah transfomasi fourier (FTIR), mikroskopi elektron pengimbasan pancaran medan (FESEM), dan spektroskopi sinar-X penyebaran tenaga (EDX) telah menunjukkan kejayaan sintesis kaliks[n]arena diubah suai PbS. FTIR dan EDX menunjukkan kehadiran getaran regangan O–H, getaran regangan C-CH_3, getaran aromatik C=C, regangan C-O, karbon (C) dan silikon (Si) yang kejayaan imobilisasi *p-tert*-butilkaliks[n]arena pada permukaan PbS. Perubahan morfologi daripada nanozarah kepada struktur seperti serpihan adalah satu lagi petunjuk kehadiran *p-tert*-butilkaliks[n]arena. Berdasarkan hasil kajian, pH titik cas sifar (pH_{PZC}) bagi kaliks[4]arena diubah suai PbS, kaliks[6]arena diubah suai PbS, dan kaliks[8]arena diubah suai PbS masing-masing ialah pH 6.80, pH 7.28 , dan pH 7.31. Kaedah respon permukaan (RSM) telah dijalankan untuk menghasilkan tindak balas permukaan bagi keadaan penjerapan yang optimum. Penilaian seterusnya dijalankan untuk mengkaji kesan interaktif pelbagai faktor (kepekatan awalan, dos penjerap, masa, pH, dan suhu) ke atas penjerap pewarna dengan menggunakan reka bentuk komposit berpusat (CCD). Peratusan penyingkiran pewarna daripada sistem air digunakan untuk menentukan prestasi penjerapan. Keputusan ujian penjerapan berkelompok dalam kajian ini, merangkumi kapasiti penjerapan, kinetik, dan isoterma proses penjerapan MO dan MB. Parameter isoterma ditentukan menggunakan model Langmuir dan Freundlich. Kajian isoterma penjerapan MO oleh kaliks[4]arena diubah suai PbS paling sesuai diwakili oleh Freundlich, menunjukkan penjerapan pada permukaan heterogen. Sebaliknya, kajian isoterma bagi penjerapan MB oleh kaliks[4]arena diubah suai PbS, kaliks[6]arena diubah suai PbS, dan kaliks[8]arena diubah suai PbS telah diterangkan dengan baik oleh Langmuir, menunjukkan penjerapan satu lapisan secara homogen pada tapak penjerapan permukaan penjerap. Model suaian dengan kapasiti penjerapan maksimum (q_{max}) 3.268 mg/g, diperolehi untuk penjerapan MO oleh kaliks[4]arena diubah suai PbS. Sementara itu, q_{max} bagi penjerapan MB oleh kaliks[4]arena diubah suai PbS, kaliks[6]arena diubah suai PbS, dan kaliks[8]arena diubah suai PbS masing-masing ialah 5.850 mg/g, 5.495 mg/g, dan 11.90 mg/g. Untuk kajian kinetik, kualiti data disuaikan dengan pseudo-tertib pertama dan pseudo-tertib kedua. Kinetik penjerapan MO oleh kaliks[4]arena diubah suai PbS, penjerapan MB oleh kaliks[4]arena diubah suai PbS, kaliks[6]arena diubah suai PbS, dan kaliks[8]arena diubah suai PbS telah diterangkan dengan baik oleh model pseudo-tertib kedua yang menunjukkan bahawa penjerapan pada permukaan penjerap berlaku melalui penjerapan kimia. Ujian kebolehgunaan semula dan ujian larut lesap telah dilakukan untuk mengurangkan isu pelupusan penjerap. Keputusan menunjukkan bahawa penjerap yang telah disintesis adalah stabil, dan berisiko rendah bagi pembebasan bahan pencemar (Pb) ke dalam air. Mekanisme penjerapan pewarna oleh kaliks[n]arena diubah suai PbS yang dicadangkan adalah seperti interaksi elektrostatik, interaksi ikatan hidrogen, interaksi ikatan hidrogen Yoshida, dan interaksi π-π. Secara keseluruhannya, kajian menghasilkan penjerap baru, kaliks[n]arena diubah suai PbS, yang boleh digunakan semula sebagai penjerap alternatif untuk menyingkirkan pewarna MO dan MB dan rawatan air sisa pewarna yang mampan terhadap alam sekitar.

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LIST OF SYMBOLS AND ABBREVIATIONS

AB-1	Acid black 1
AC	Activated carbon
ANOVA	Analysis of variance
AO-7	Acid orange 7
AR-18	Acid red 18
BBD	Box-Behnken design
CaIP	Calix[4]arene-based polymer
CCD	Central composite design
C_f	Final concentration
C_i	Initial concentration
CIP	Ciprofloxacin
COD	Chemical oxygen demand
COVID-19	Coronavirus disease 2019
CV	Coefficient of variation
DB-38	Direct black
DIS resin	*p*- diethanolaminomethylcalix[4]arene on silica surfaces
DO	Dissolved oxygen
DOE	Design of experiment
DOF	Degree of freedom
EDX	Energy dispersive X-ray
FESEM	Field emission scanning electron microscopy
FSNCS/SDBS	Magnetic nickel cobalt sulphide/ sodium dodecyl benzene sulfonate
FTIR	Fourier transform infrared
ICP-MS	Inductively coupled plasma mass spectrometry

LOF	Lack of fit
MB	Methylene blue
MFS	Modified flax shives
MGO	Malachite green oxalate
MLBC	Mango leaves biochar
MO	Methyl orange
MTCFs	Magnetic properties of tubular carbon nanofibers
MWCNTs	Multi-walled carbon nanotubes
n	Freundlich constant
NCs	Nanocrystals
PRESS	Prediction error sum of squares
p-SC8SR	*p*-sulphonatocalix[8]arene-based silica resin
PZC	Point of zero charge
q_{max}	Maximum adsorption capacities
R^2	Coefficient of determination
R^2_{adj}	Adjusted coefficient of determination
RhB	Rhodamine B
R_L	Separation factor
RSE	Residual standard error
RSM	Response surface methodology
SDGs	Sustainable Development Goals
Std. Dev.	Standard deviation
TSS	Total suspended solids
USEPA	United States Environmental Protection Agency
UV-vis	Ultraviolet-vis

CHAPTER 1

INTRODUCTION

1.1 Research background

Water is classified as one of the most important elements for all human beings and living organisms on earth to sustain life. In these modern times, the demand for clean fresh water consumption has increased tremendously in many sectors, including industrial, agricultural, and domestic. However, due to the rapid pace of industrialization, tremendous growth population, and expansions of civilization, have caused water stress conditions around the world (Miklos et al., 2018; Xiang et al., 2020).

According to one of the 17 Sustainable Development Goals (SDGs) established by the United Nations in 2015, water quality must be improved by 2023 by reducing pollution, eliminating dumping, reducing the release of hazardous chemicals and materials, controlling the discharge of untreated wastewater, and significantly increasing recycling and safe reuse globally (Giannetti et al., 2020). Based on statistics from several international organizations also stated that 70% of industrial waste in developing countries is discharged into heavily polluting industries without proper treatment, and 90% of harmful pollutants in these countries end up in municipal systems or directly into natural water bodies (Jayaswal et al., 2018; Singh et al., 2020).

Industries such as wet fabric industries are widespread as major contributors of industrial dye waste due to high consumption of water during the fabric production processes, such as washing, dyeing, bleaching, printing, and finishing. The processes produce a highly polluted effluents, as well as increasing water scarcity and pollution on the global water environment. One of the most popular fabric industries in Southeast Asia is the batik industry, which is one of the largest cottage industries and has become a significant contributor to water pollution; it's extremely high-water consumption, discharges wastewater rich in colour, and high pollution potential (Ramakreshnan et al., 2020). More seriously, the majority of dye pollutants are toxic and high chemical oxygen demand (COD) which can degrade environmental quality and endanger human health (Liang et al., 2018). As a result, pollutant removal is critical for fabric industrial manufacturers as a tool for evaluating and controlling the pollution control performance of cleaner production technologies.

To address those challenges, various physical, chemical, and biological approaches have been highlighted for the removal of dye pollutants, including adsorption, flocculation-coagulation, photocatalytic degradation, bio-treatment, and membrane filtration (Hube et al., 2020; Zhao et al., 2021). However, some of these techniques suffer by high cost, generation of voluminous sludge, and lack of low concentration removal, selectivity and sensitivity. Among the techniques, adsorption is considered one of the most effective treatment process due to its simple-implemented, cost-effectiveness, low-energy investment, highly-efficient technique and the high potential of a wide range of adsorbents for wastewater treatment (Burakov et al., 2018). Aside from that, adsorption is also known as a suitable technique for the removal of a various pollutants, including soluble and insoluble organic, inorganic, and biological.

The selection of suitable precursors for the adsorption of waste pollutants from water is an important aspect. Recently, nanomaterial adsorbents have received widespread attention from researchers due to their effectiveness in removing waste pollutants. Several types of nanomaterials, such as carbon materials, metal-organic frameworks, and minerals or metal oxides, have been widely studied and extensively reviewed (Wong et al., 2018). Other than that, metal sulphide loaded nanomaterials are also gaining popularity as adsorbents due to their efficiency in wastewater treatment (Yan Yang et al., 2017). However, several studies on metal sulphide adsorbents have been published, and they need to be studied in greater detail.

1.2 Problem statement

Water pollution caused by industrial and domestic dye wastes creates long term adverse effects to ecological and human health. If there is no serious action taken for water recovery, it is anticipated that only one third of the world population will have access of freshwater by 2025, where available freshwater sources fail to meet water demand due to poor quality (Ungureanu et al., 2020). Azo dyes, such as methyl orange and (MO) methylene blue (MB), contribute to serious environmental water pollution due to their harmful properties, such as high toxicity, difficult degradation, heat and light stability, and thus difficulty to decolorize (Baig et al., 2020; Chung, 2016). Several studies have found that MO dye can cause harm to the human body, including the hepatic system, skin, and central nervous system, as well as cerebral damage, renal disruption, and digestive tract dysfunction (Carolin et al., 2021; Yagub et al., 2014). Meanwhile, high doses of MB (>7.0 mg/kg) exposure can pose a serious threat to human health, causing symptoms such as high blood pressure, nausea, abdominal pain, methemoglobinemia, genetic disorders, digestive disease, and mental disorders (Sharma et al., 2018). More concerning, the majority of the dyes are mutagenic, carcinogenic, and teratogenic to humans, the environment, and marine life (Bilal et al., 2018; Ismail et al., 2019). For this reason, removing organic chemicals and dyestuffs from water streams is necessary in reducing their impact on the environment and human health.

Nanomaterial adsorbents have recently received a lot of attention due to their high efficiency in removing waste effluents, cost-effectiveness and good applicability in residual waste disposal technology, and efficient adsorption capacity. Various nanomaterial adsorbents were used based on their adsorption capacities. Metal sulphide adsorbents, on the other hand, have yet to be used to remove azo dyes from wastewater. Evidently, all previous investigations focused on metal sulphide as a heavy metal ion removal agent but there are no published reports on the use of lead sulphide (PbS) for the removal of azo dyes. Considering calixarene's extraordinary ability as the most promising for host-guest recognition, exploring strategies using different cavity dimensions, calix[4]arene, calix[6]arene, and calix[8]arene with PbS materials with efficient dye removal while maintaining high recyclability remains a challenge.

One of the major challenges in utilization of various techniques for dye removal is the proper selection of experimental conditions. In general, the traditional one-parameter-at-a-time approach is employed to optimize the experimental conditions. The main disadvantage of the traditional approach is that only one parameter is varied at a time, with no consideration given to the interactive effects of variables. Furthermore, this method necessitates a large number of experiments, which is both time-consuming and costly. To overcome this problem, the design of experiments (DOE) is used in order to evaluate the relationships between factors affecting a process and the maximum adsorption of water effluents, aiding in both cost reduction and the consumption of valuable resources such as energy and materials.

1.3 Research objectives

The main objective of this study is to develop novel nano-adsorbent, calix[n]arene-modified lead sulphide (PbS) (n=4, 6, 8) nanoparticles for the removal of dyes. In order to achieve the main aim, there are four specific objectives have been designed as follows:

i. To prepare and characterize PbS nanoparticles with different ring sizes of calix[4]arene, calix[6]arene, and calix[8]arene.
ii. To determine the adsorption process under optimized conditions using response surface methodology (RSM) approach.
iii. To evaluate the adsorption studies, desorption and reusability of adsorbent in succeeding cycles for the removal of dyes.
iv. To determine the applicability of selected adsorbent for adsorption of different dyes.

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