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SYNTHESIS AND CHARACTERIZATION OF MOLECULARLY IMPRINTED POLYMERS (MIPs) AS ADSORBENTS FOR METHYLENE BLUE DYE

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ABSTRACT

Molecular imprinting technology (MIT) utilizes molecularly imprinted polymers (MIPs) to create highly selective adsorbents with tailored binding sites. This study focuses on developing methylene blue (MB) adsorbents using MIPs synthesized via photopolymerization. The optimal adsorption capacity was achieved with a monomer-to-crosslinker ratio of methacrylic acid (MAA) to ethylene glycol dimethacrylate (EGDMA) at 1:20 mol. The MIPs demonstrated efficient adsorption with extraction and re-extraction times of 40 and 60 minutes, respectively, and an optimal pH of 4.0, resulting in an adsorption capacity of 787.74 mg/g. The MIPs offer advantages for MB adsorbents, such as simplicity, stability, and reusability, distinguishing them from traditional adsorbents, such as activated carbon, due to their superior selectivity.

Keywords: Molecularly Imprinted Polymers (MIPs), Methylene Blue (MB), Adsorption, Photopolymerization, adsorption capacity.

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INTRODUCTION

Methylene blue (MB) is used in various industrial processes such sectors as textiles, paper, and leather,[1]chemical, diagnostics, surgery, aquaculture, medical, pharmaceutical, microbiology, and as a sensitizer in the photo-oxidation of pollutants.[2] MB is a heterocyclic compound with the chemical formula $C_{16}H_{18}N_3SCl$.[1,3,4] MB has the potential to cause increased heart rate, vomiting, shock, and tissue damage,[5] eye burns, difficulty breathing, jaundice, vomiting, excessive sweating, mental confusion, methemoglobinemia, allergic dermatitis, skin irritation,[6] cancer, convulsions, blindness, cyanosis, mutagenicity.[7] Therefore, the disposal of MB from wastewater is an important environmental issue.[6] In dye wastewater treatment, the adsorption method is more popular because of its good treatment effect, low treatment cost, simple operation, and no harmful substances.[8,9,10] Various materials have been developed as MB absorbing materials, such as Chitosan-polyvinyl alcohol-diatomite hydrogel,[11] mesoporous grass-based activated carbon,[12] cellulose-based adsorbents from sugarcane bagasse,[5] hydroxyl groups materials,[10] mesoporous zeolite-A/reduced graphene oxide nanocomposite.[13] In addition, MIPs have also been developed as MB absorbent materials, such as sodium alginate/polyethylene oxide nanofibrous membranes,[14] Hybrid amino-functionalized TiO2/sodium lignosulfonate surface MIPs,[15] bioinspired sorbent,[16] hybridized membrane cellulose and polysulfone,[17] Hybrid magnetic molecularly imprinted polymers.[18] MIPs have several advantages, such as selectivity, excellent stability, ease of synthesis, strong bonds, and resistance to temperature and pressure.[19,20] Although MIPs have several advantages and have

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been widely developed to absorb MB, they have weaknesses because the synthesis method is complicated and requires a longer time. A more efficient and rapid method for synthesizing molecularly imprinted polymers (MIPs) is essential to expedite the treatment of Methylene Blue (MB) waste. Therefore, this study synthesized MIPs through photopolymerization to adsorb MB selectively. The process utilized Methylene Blue as the template, methacrylic acid (MAA) as the monomer, ethylene glycol dimethacrylate (EGDMA) as the crosslinking agent, and 2,2-dimethoxy-2-phenylacetophenone (DMPP) as the photoinitiator, with acetonitrile serving as the porogenic solvent. Compared to traditional polymerization techniques, photopolymerization offers several advantages, including being solvent-free, energy-efficient, cost-effective,[21] and environmentally sustainable.[22] Additionally, it significantly reduces synthesis time, making it an attractive alternative for large-scale applications.

EXPERIMENTAL

Materials and Instruments

The chemicals used in this research were Methylene Blue (MB), Methacrylic Acid (MAA), Ethylene glycol dimethylacrylate (EGDMA), 2-2-Dimethoxy-2-Phenylacetophenone (DMPP), and Acetonitrile (C2H3N) were supplied by Sigma- Aldrich. At the same time, Sodium Hydroxide (NaOH) 0.1 M and hydrochloric Acid (HCl) 0.1 M were obtained from Aldrich). Meanwhile, the instrument used is photopolymerization with ultraviolet radiation of a wavelength of approximately 250–350 nm.

Synthesis and Characterization of Molecularly Imprinted Polymer-Methylene Blue (MIPs-MB)

Polymer-Methylene Blue (MIPs-MB) was prepared according to the method previously reported by Djaha et al.[23] MB was weighed as much as 1.0 mg and DMPP as much as 0.03 gr was mixed with MAA (4 mol) and EGDMA (20 mol) at optimum composition. Then, the mixture was homogenized by sonication for 60 minutes. After the homogeneous mixture was polymerized with UV light under continuous nitrogen gas flow. As a control, a non-imprinted polymer (NIP) was prepared without using MB, serving as a comparison to the methylene blue-imprinted polymer (MIP-MB). The functional groups of the MIP-MB and the control NIP were analyzed using FTIR Spectroscopy to assess any differences in their chemical structures. The adsorption capacity of MIPs-MB, q (mg/g), was determined using the equation: $q=(C_0-Ct)/m$, where C_0 and C_t (mg/L) are the concentrations of MB in the solution before and after treatment, respectively, and mg is the amount of adsorbent in 1L of MB solution.[24] Meanwhile, C_t is calculated using the UV-Vis spectrophotometry method which uses the MB calibration curve.

Extraction and Re-extraction of Methylene Blue from Polymer Membrane Synthesis to Produce MIP

To extract MB from the MIP, 0.1 g of MIP-MB was soaked in 2 mL of acetonitrile for 1 to 60 minutes. During this period, the absorbance of the solution was determined using a UV-Vis spectrophotometer at regular intervals, from the first minute to the 60th minute. After determining the optimum extraction time, the MIP-MB was air-dried at room temperature, and the extraction results were analyzed using FTIR to confirm the removal of MB. For the re-extraction process, 0.1 g of the previously extracted MIP-MB was soaked in a 20ppm methylene blue solution for the same 1-60minute duration, with absorbance being monitored again using a UV-Vis spectrophotometer. After soaking for optimal re-extraction, the MIP-MB was dried at room temperature and analyzed via FTIR to assess its structural properties post-re-extraction. The reaction mechanism of MIP-MB synthesis is illustrated in Fig.-1.

Effect of pH and MB Amount on Adsorbent Capacity

The pH effect on the absorption capacity was tested by 0.1 g of MIP-MB, which was put into 3 mL of MB solution (50 ppm) with various pH (2.0, 4.0, 6.0, 8.0, and 10.0) for 60 minutes. Then, MIP-MB was removed from the MB solution, its absorbance was measured using a UV-Vis spectrophotometer, and its absorption capacity was calculated. Furthermore, the effect of MB concentration variations on the absorption capacity was tested by immersing 0.1 g of MIP-MB into 3 mL of MB solution with concentration variations of 10.0, 15.0, 20.0, 25.0, and 30.0 ppm at optimum pH for 60 minutes. Furthermore, MIP-MB was removed, the absorbance of the solution was determined using a UV-Vis spectrophotometer., and its absorption capacity was calculated.

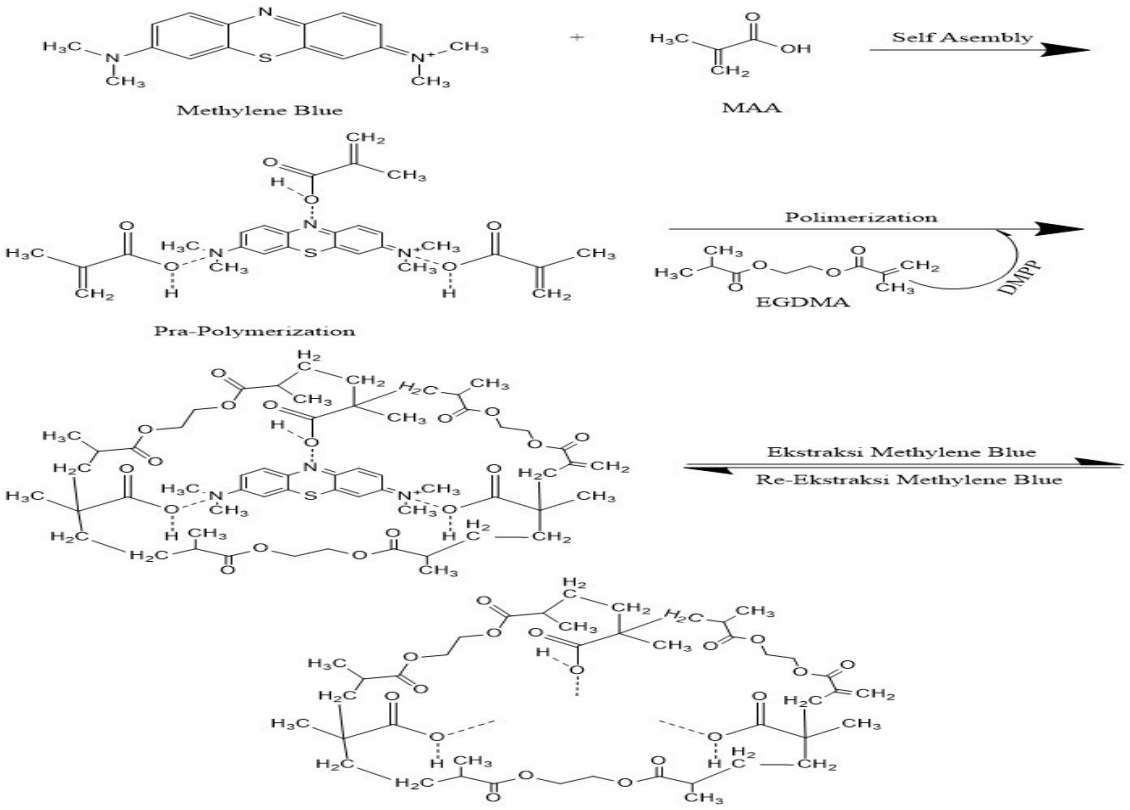


Fig.-1: A MIP-MB Synthesis Reaction Prediction

RESULTS AND DISCUSSION

Characterization of MIP-MB and MIP (control)

Functional group analysis of MB, MIP-MB, MIP (control), MIP-MB extraction, and MIP-MB re-extraction is seen in Fig.-2 below. In the spectrum of MB dye, there is a broad but low-intensity peak at wave number 3432 cm^{-1} caused by hydroxyl group stretching. At wave numbers 2702, 1948, and 1583 cm^{-1}, peaks indicate the presence of C=N and C=C bonds in the MB molecular. Asymmetric C-H stretching is observed at wave number 1382 cm^{-1}, and bending of C-H bonds outside the heterocyclic plane at wave number 1119 cm^{-1}. In addition, wave number 869 cm^{-1} shows asymmetric stretching of hydrogen bonds in MB.[25] The spectrum in the wave number range of 1320-1000 cm^{-1} shows the presence of C-O functional groups in MIP, MIP-MB, MIP-MB extraction, and MIP-MB re-extraction, namely Where the area describes the characteristics of ether, namely the EGDMA crosslinker and this C-O group is not present in MB dyes. Furthermore, the 1690-1760 cm-1 absorption area shows the presence of C=O and C=C functional groups in the absorption area of 1500-1700 cm^{-1} originating from MAA (methacrylic acid). In addition, in MB dyes, MIP-MB, and MIP-MB re-extraction, there are C=N functional groups in the absorption area of 1900-1970 cm^{-1} but not in the control MIP, indicating that the C=N functional group comes from the MB molecule. Although MB has been extracted from MIP-MB, there are still C=N groups; this shows that not all MB can be extracted from MIP-MB. The results of this FTIR study show that the MIP-MB membrane can bind analytes with the same characteristics and properties as the target molecules that have been synthesized, as previously reported,[26] which uses uric acid as a template.

Extraction and Re-extraction Time of Methylene Blue on MIP-MB

After the MIP-MB is made, it is necessary to wash it using the porogen solvent acetonitrile, where this washing aims to remove the MB as a template so that the MIP has pores that are adjusted to the shape of the MB.[27] Acetonitrile is a polar aprotic solvent that forms a complex bond of functional monomer-template prepolymer. In addition, acetonitrile was chosen because it has a high dielectric constant. Previous studies' results explained that using porogen solvents with high dielectric constants significantly increased the binding properties of MIP.[28] The variation of extraction time was determined to obtain the optimum

washing time until the template (MB) was removed from MIP-MB. Figure-3a shows the effect of extraction time on the amount of MB released from MIP-MB.

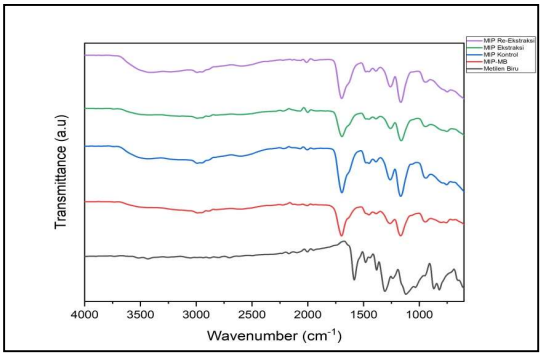
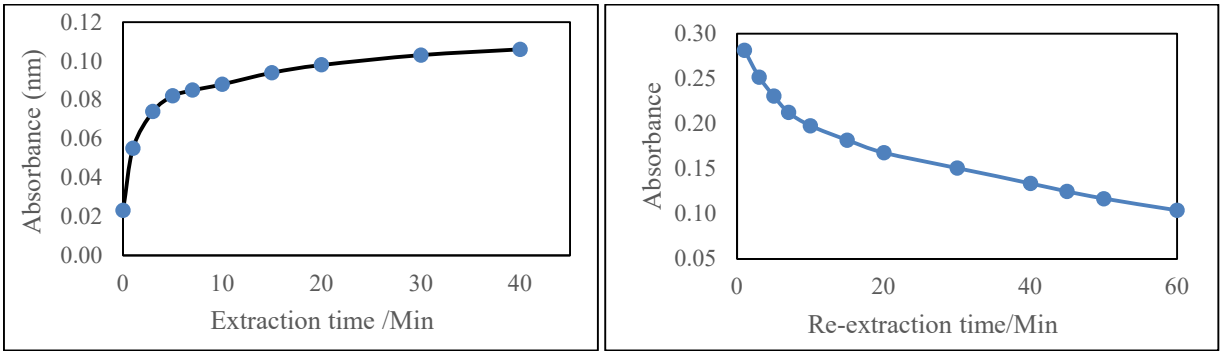


Fig.-2: FTIR Spectrum of Methylene Blue (a), MIP-MB (b), Non-Imprinted Polymers (NIPs) (c), MIP-MB that Has Been Extracted with MB (d), MIP-MB that has Been Re-Extracted with MB Solution (e)

The absorbance value of the extract solution (acetonitrile) increased along with the length of the extraction time; this indicates that the amount of MB released from MIP-MB is increasing. The analysis showed that the extraction time of 60 minutes produced the highest absorbance value of (0.108). This theory states that the absorbance value is directly proportional to the dye concentration, so the higher the absorbance of the MB solution, the higher the concentration of the MB solution that comes out of MIP-MB. Template extraction from MIP-MB can occur because the hydrogen bond between the template (MB) and MAA can be broken using a solvent with high polarity, which produces a significant difference in electronegativity so that the bond between the solvent-template becomes stronger than the template-monomer bond. Figure-3b shows the effect of contact time on the amount of MB adsorbed in the MB re-extraction process on MIP-MB. The decrease in the absorbance of the MB solution from 0.282 to 0.104, along with the increase in the re-extraction time, indicates an increase in the amount of MB absorbed by MIP-MB. The decrease in the absorbance value indicates a decrease in the concentration of the MB solution after interacting with MIP-MB; this also proves that MIP-MB can absorb and recognize MB as its target molecule. At the beginning of MIP-MB being contacted with the MB solution, there were still many active sites of MIP that had not yet bound to MB, so the tendency for MB to be absorbed by MIP-MB was higher. The effect of contact time on absorption has also been reported previously[29,30] the longer the contact time, the greater the amount of MB absorbed by the MIP.



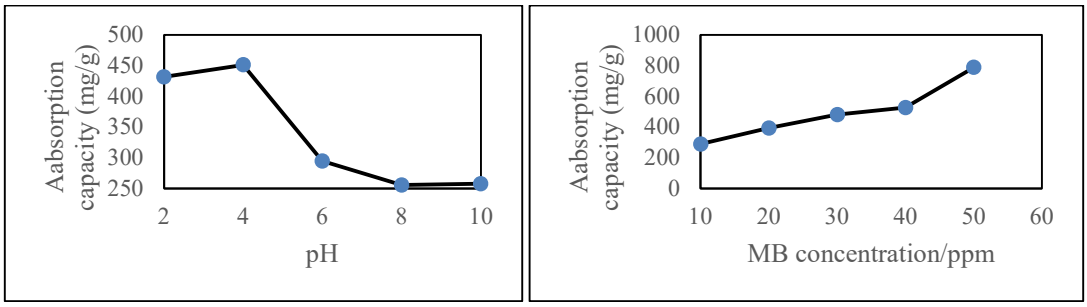
(a) (b)

Fig.-3: Effect of Extraction Time (a) and Re-Extraction (b) of MB on MIP-MB

Effect of pH and MB Concentration on Absorption Capacity

The acidity level of the solution greatly influences the adsorption process, thus affecting the absorption capacity. The presence of hydroxyl ions (OH^-) and hydrogen ions (H^+) in the solution affects the interaction between the adsorbent and the adsorbate molecules due to the interaction between (OH^-) and hydrogen ions (H^+) with MB as an anion molecule.[30] In addition, the isoelectric point greatly influences the adsorbent's absorption capacity, where the pH at the isoelectric point is where the maximum absorption capacity occurs.[31] In addition, the high adsorption under high acid conditions can increase the electrostatic attraction

between the charged adsorbent and the MB adsorbate.[32] Figure-4a shows the effect of pH on the adsorption capacity of MIP-MB on MB. The optimum adsorption capacity is 450.96 mg/g at pH 4.0, while the minimum adsorption capacity occurs at pH 8.0, 255.48 mg/g. The adsorption capacity at pH 2 to 4 increases because of a strong electrostatic attraction between the adsorbent and the cationic MB adsorbate at low pH. At high pH, there is a decrease in the absorption capacity caused by the interaction of MB with the OH group. While, the variations in concentration were carried out, namely 10, 20, 30, 40, and 50 ppm, to obtain information on the concentration needed to achieve optimum absorption of MB dye by MIP. The results of MB adsorption with concentration variations are seen in Fig.-4b. The adsorption capacity increases with increasing MB concentration. The adsorption capacity also increases when the MB concentration is increased from 10 ppm to 50 ppm. This increase in adsorption capacity indicates that at an MB concentration of 50 ppm, active sites are still available to adsorb MB with an adsorption capacity of 787.74 mg/g. Similar findings have also been reported by Fazal *et al*., who used starch phosphate grafted polyvinyl imidazole as an MB adsorbent,[30] Zakria *et al.,* which uses mangrove-based activated carbon for MB dye absorption,[33] Similar results have also been reported by Li et.al. who used starch-based composite hydrogel microspheres for MB removal.[34]



(a) (b)

Fig.-4: Effect of pH (a) and MB Concentration (b) on Adsorption Capacity

Adsorption Capacity and Mechanism Study of MIP-MB

The Langmuir and Freundlich curves are used to describe the isothermal sorption model.[35,36] This study was conducted on MB isotherm adsorption with variations in the concentration of MB absorbed. This isotherm model was tested using the experimental results to determine the adsorption equilibrium model. The Langmuir and Freundlich isotherm equations were converted into linear equilibrium curves to determine the equilibrium model, which can be seen from the value of the determinant coefficient (R^2) approaching or equal to 1.0. Because the adsorption isotherm results with a value of $R^2 \approx 1$ are Freundlich isotherms. (Fig.-5).

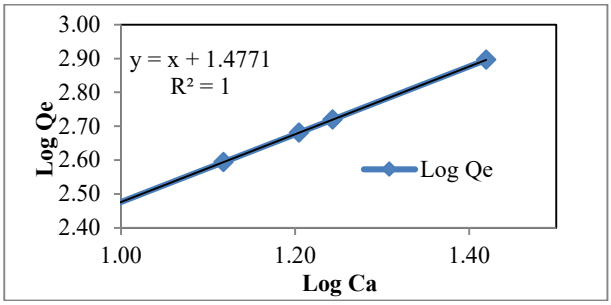


Fig.-5: Freundlich Isothermal Graph

The linear equation of the Freundlich isotherm model is obtained by connecting the values of log Ca and log Qe. Ca is the concentration of MB absorbed, and Qe is the adsorption capacity. Based on the graph above, the adsorption of MB by MIP adsorbent tends to follow the Freundlich isotherm. The value of the Freundlich isotherm's determinant coefficient (R2) is higher, 1, compared to the Langmuir isotherm, which is less than one. This states that the physically adsorbed MB forms a multilayer layer, with the surface of the active side of the MIP adsorbent being heterogeneous. The same thing was previously reported by Xiaoya *et al.*[37]

CONCLUSION

This study successfully synthesized MIP-MB using methylene blue as a template through photopolymerization. The MIPs demonstrated notable selectivity, stability, and reusability, significantly improving over traditional adsorbents like activated carbon. Key findings indicated that the adsorption capacity of the MIP-MB was influenced by both the pH level and the concentration of methylene blue, with optimal adsorption achieved at pH 4. The study also established that the MIPs follow a multilayer adsorption mechanism per the Freundlich isotherm model, further validating their efficiency for industrial applications in dye removal from wastewater.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All authors have contributed significantly to this manuscript. They have participated in writing, reviewing/editing, and approving the final draft of the article for publication. The author's research profile can be verified via their ORCID ids, given below:

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