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RESEARCH ARTICLE

Adsorption and Kinetic Study of Toxic Dye (Methylene Blue) Removal from Simulated Industrial Wastewater Using Sustainable Material

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ABSTRACT

Water pollution is a global issue that affects both human health and the environment. The increasing use of the methylene blue (Mb) dye in various industrial applications poses significant risks. The study aimed to use hydrochloric acid-modified *Ziziphus Spina Christi* powdered leaves (ZSC) as an effective and low-cost adsorbent for removing Mb dye from effluents. Batch experiments were conducted with contact times of 10–180 minutes, initial dye concentrations of 10–60 mg/L, temperatures of 25–60°C, pH values of 5–9, and sorbent dosages of 0.25-2 g per 40 ml of dye solution. The results showed that the highest removal efficiency of Mb dye was 97.51% using an initial concentration of 10 mg/L, 1.5 g of adsorbent, pH 8, at 50 °C, a contact time of 60 minutes, a particle size of 0.075 μm, and a stirring speed of 150 rpm. The ZSC was characterized using SEM and FTIR techniques, and the adsorption kinetics were found to follow the pseudo-first-order model, with a correlation coefficient of 0.969. In general, the modified ZSC is an auspicious, friendly, and cost-effective adsorbent for removing Mb dye from aqueous solutions.

Keywords: Health concerns, Methylene blue (Mb), Sustainability, Water pollution, Ziziphus spina-christi (ZSC) leaves

Introduction

Water is an essential resource for humans and all living organisms. Rising consumption, population growth, and climate change have made water scarcity one of the most urgent global challenges. Although water is our largest natural resource, it is finite and irreplaceable; therefore, water conservation and sustainability are essential for surviva.[1] Even in small quantities, synthetic dyes in environmental settings, especially aquatic ones, can pose a threat to human health and the ecosystem over time due to their toxic metallic content, resistance to biodegradation, aromatic nature, and bioaccumulation.[2,3]

Dyes have been linked to various health effects, including cancer, genetic mutations, organ toxicity, and cardiovascular problems such as arrhythmia, when released into the environment.[4,5] Methylene Blue (Mb) dye “3, 7-bis (dimethylamino)phenothiazine chloride tetramethyl thionine chloride” is a commonly used cationic dye with high toxicity and ecological stability. It is used in many industries, like coloring paper, leather, textiles, cosmetics, and pharmaceuticals, as well as a fungicide and biocide in aquaculture.[6] Excessive usage in many sectors and untreated discharge into the environment have produced many health concerns, like cancer, mutations, pulmonary toxicity, and chromosomal breakage; it may lead to lethal serotonin poisoning in humans if concentrations surpass 5 μg/kg.[7] Methylene blue affects the growth of aquatic plants and algae, as it has been found to inhibit growth and reduce pigment content and total protein of microalgae (*Spirulina*

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platensis and *Chlorella vulgare),* causing the ecosystem to be unstable.[8,9] Due to its harmful consequences for the ecosystem, it must be properly removed before being disposed of in water. Several processes, like ozonation, ion exchange, filtration, coagulation, and photocatalysis and adsorption, are utilized for removing pollutants from wastewater.[10,11] These technologies have several drawbacks in terms of effectiveness, selectivity, overall cost, and secondary waste generation.[1,12] Adsorption is a separation process that involves increasing the concentration of molecules on the surface of a solid. It has proven its ability to remove a wide range of pollutants from wastewater and is characterized by its availability, cost, ease of use, high efficiency, and simple design and uncomplicated .[13,14]

Ziziphus spina-christi (ZSC) belongs to the Rhamnaceae family and grows well in warm climates and subtropical locations. These trees are tall and may reach a height of about 20 meters and a diameter of 60 cm, and they are characterized by shiny, oval, lanceolate leaves with prominent lateral veins on their upper surface, and sharp or semi-obtuse edges.[15] Numerous studies have demonstrated the ability of the plant *Zizyphus spina*-Christi (ZSC) to remove a variety of colors from aqueous solutions. For instance, a study by Bashanaini et al.[16] found that activated carbon made from *Zizyphus* spina-Christi seed (ZSCS) effectively eliminates the dye malachite green. Alsuhybani et al.[13] reported that *Zizyphus Spina-Christi* (ZSCS) seeds were highly effective in eliminating several industrial dyes like methylene blue (Mb) and methyl violet (MV) from water. In a comparative investigation, Abdulhameed et al.[17] employed a biocomposite of chitosan and modified *Ziziphus spina-christi* leaves to remove methylene blue dye and discovered an impressive dye adsorption capability.

Therefore, the primary objective of this study is to assess the efficiency of powdered *Ziziphus spina-christi* leaves (ZSC) in removing Methylene Blue dye from aqueous solutions under various laboratory conditions, and to examine their morphological characteristics.

Material and method

Experimental

Materials and devices utilize

Methylene blue (Mb) is a cationic dye. Its chemical formula is $C_{16}H_{18}ClN_3S_1$, with a molecular weight of 319.851 g/mol, and its structural formula as shown in Fig. 1. A UV-Vis spectrometer (Shimadzu, UV-1900i) was utilized to measure the concentration of methylene dye in the filtered samples at 665 nm. The experiment included three replicates for each parameter.[18] The main materials used in this study were *Ziziphus spina-christi* leaves, MB dye, deionized water, ethanol, and hydrochloric acid. Ethanol was applied after drying the plant leaves to remove the green pigment (chlorophyll) prior to the adsorption experiments, preventing interference with dye adsorption. Key equipment included a magnetic stirrer, centrifuge, oven, and digital balance.

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Fig. 1. Illustrates the molecular structure of Methylene Blue (Mb) dye.

Preparation of adsorbents (ZSC)

To prepare the powdered acid-modified ZSC leaves, the leaves were gathered, repeatedly cleaned with distilled water to get rid of dust and other impurities, allowed to dry at ambient temperature for 15 days, and then oven-dried for 24 hours at 70°C. The leaves were ground in an electric grinder (Model SC-EG150, Silver, Crest, Germany) and passed through a 0.075 μm mesh. After centrifuging them to get rid of any last traces of color, these leaves were dried at 60°C for 24 hours. Then, 50 grams of the powdered leaves were placed in 500 ml of hydrochloric acid (0.1 N) solution for 24 hours at ambient temperature. The filtered powder was then washed with deionized water until the filtered solution achieved neutral pH (7.0); the powder was then dried at 70°C, and the dried samples were put in a tightly sealed vial for later use.[19] Fig. 2.

Batch biosorption experiment

Mb dye solution (500 mg/L) was prepared by dissolving 0.5 g of dye (from Sigma-Aldrich) in 1000 mL of deionized distilled water and shaking it in an orbital shaker (Edmund Buhler SM25, Germany) to achieve optimal solubility. The remaining experimental concentrations were prepared by serial dilutions. Batch tests were conducted at room temperature to independently determine the optimal parameters for the Mb dye adsorption process, including contact time (10–180 min), ZSC adsorption dosage (0.25–2 g), Mb concentration (10–60 ppm), temperature (25–60 °C), and pH (5–9), which was adjusted with NaOH,

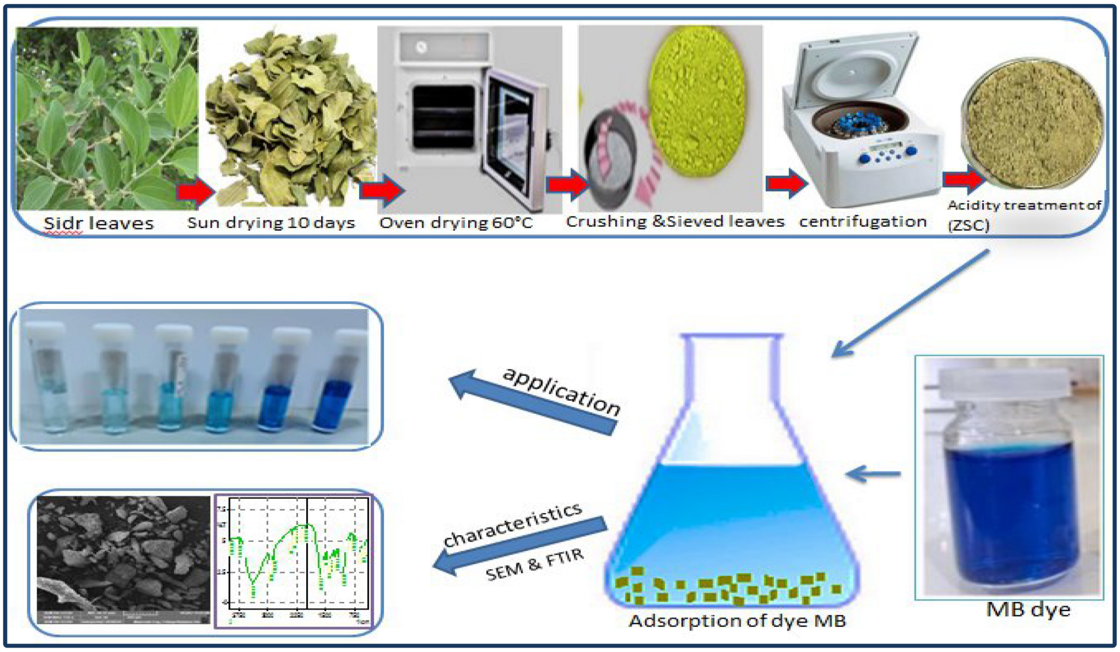


Fig. 2. Experiment design scheme.

and HCl (0.1 mol), and stirring at 150 rpm. Eq. (1), was employed to calculate the removal percentage R% of Mb dyes.[20]

$$R\,(\%) = \frac{Co - Ct}{Co} \times 100 \tag{1}$$

where C0 represents the dyes' starting concentration, Ct represents their concentration at time t, and R% represents their removal efficiency.

Kinetic adsorption models

The kinetics of langmuir-hinshelwood (L-H) was used for characterizing the interaction between adsorbent and adsorbate molecules. The three kinetic models, "zero-order, pseudo-first-order, and pseudo-second -order", are presented by Eqs. (2) to (5), respectively

The rate of dye removal constant was estimated by utilizing Eq. (2).

$$Kt = ln\,(Ci/Ct) \tag{2}$$

K denotes the dye adsorption rate constant, while t indicates the dye adsorption duration. The kinetics of Langmuir-Hinshelwood (L-H) is a common method for characterizing the interaction between adsorbent and adsorbate molecules. The model of (L-H) is affected by numerous parameters like adsorbent characteristics, elements of mass transfer, and operating conditions. The adsorbed particles of dye are consumed at an initial rate as in Eq. (3).[21]

$$-\frac{dC}{dt} = \frac{\mathrm{kKC}}{1 + KC} \tag{3}$$

The adsorbent concentration remains constant during the adsorption process, hence the rate of adsorption is determined by the dye concentration. As a result, the pseudo term may be employed to estimate the rate of reaction. When the dye concentration is high, the adsorbent surface becomes saturated, hence the rate constant is unaffected by the dye concentration. According to Eq. (4).[22]

$$-\frac{dC}{dt} = \mathrm{K} \tag{4}$$

However, when the dye concentration is low, the rate of adsorption is reliant on the dye concentration change; Eq. (5) takes on the following form:

$$-\frac{dC}{dt} = \mathrm{K1}C \tag{5}$$

FTIR and SEM characterization of ZSC

FTIR analysis was performed at the University of Babylon using the (Shimadzu IRAffinity-1S) spectrometer in the 400-3000 cm^{-1} range to determine functional groups responsible for Mb dye absorption. A "scanning electron microscope SEM" (JEOL-JSM-5200 LV, USA) was utilized to analyze the outer

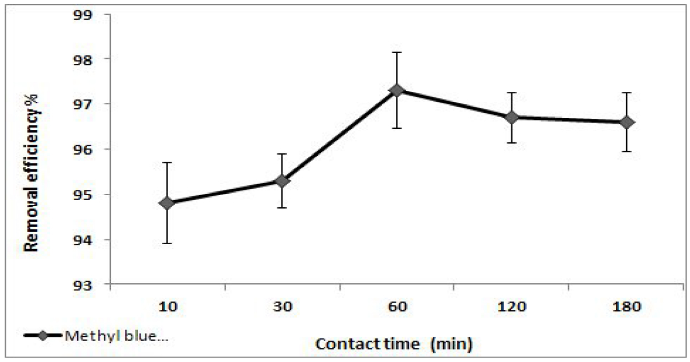


Fig. 3. Influence of contact time on adsorption of Mb.

surface morphology of ZSC particles before and after dye adsorption.[23]

Analysis of statistics

In this study, all experiments were performed in triplicate. SPSS (version 26) software was utilized for statistical analysis, and the results are presented as mean ± standard deviation, with significance considered at $P < 0.05$.

Results and discussion

Impact of contact time

Determining the appropriate time to reach equilibrium is a crucial stage in batch studies, as it helps understand how pollutants disperse throughout the sorbent and solution phases.[20] Fig. 3 demonstrates how the effectiveness of Mb dye adsorption varies with contact time. The impact of contact time on removing methylene blue dye was tested using ZSC for (10 to 180) minutes under constant conditions of 0.65 g sorbent dosage, 20 ppm initial concentration, 40 mL of MB dye(a volume of 40mL of Mb dye solution was used in each experiment), the same pH, 25 °C, and 150 rpm agitation speed. Mb dye adsorption efficiency appears to be relatively rapid. Mb dye's adsorption efficacy appeared to be fairly swift initially, with sorption increasing substantially in the first 30 minutes and reaching equilibrium after 60 minutes. The rate is projected to diminish after an hour and a half owing to a decrease in occupied spaces.[3] As a consequence, the best contact time was determined to be 60 minutes, with a maximum Mb removal rate of 97.3%. The rapid adsorption of (ZSC) is attributed to its increased surface area and functional groups, which facilitate the interaction between the biological material's surface and dye molecules. The efficiency of dye removal also depends on the surface area and functional groups such as -OH, $-NH_2$, and C=O, which facilitate adsorption through hydrogen bonding and electrostatic interactions.[22] These results are consistent with the findings of Salahshour et al.,[23] which recorded the highest adsorption efficiency of methylene blue dye, which was 93.67% after 60 minutes using activated carbon prepared from lotus leaves, while AlMokhalelati et al.[24] achieved the highest R% for methylene blue at high doses, 95% after 50 minutes with sugarcane seeds. In a study conducted by Alabi et al.,[25] methylene blue dye was eliminated from industrial waste employing ground seeds of Albizia lebbeck; the dye reached equilibrium after 360 minutes.

Impact of initial concentration

The initial concentration is a significant factor in the adsorption process since it influences the rate of pollutant adsorption in the medium. Fig. 4 illustrates that the starting concentration significantly affects the removal effectiveness of Mb dye. These elimination efficiencies were dramatically reduced, from 95.7% to 65.6% for Mb at 60 minutes of contact time, 1.5 g/40 mL dosage, and the same pH,. All dye molecules are anticipated to interact with the available binding sites, significantly enhancing dye adsorption efficiency; however, an excessive concentration of dye molecules due to high concentration may lead to a reduction in removal efficiency because of insufficient free binding sites for Mb adsorption in an aqueous solution.[14,20] It is consistent with the findings of several studies as.[26–28]

Impact of PH on Mb adsorption

The pH of a solution often affects both positively and negatively charged surface molecules. Through the process of gaining or losing a proton, their charge changes to become more positively or negatively charged, and the PH of the solution influences the degree of ionization and dissociation of the functional groups of the material.[29] Fig. 5 depicts the influence

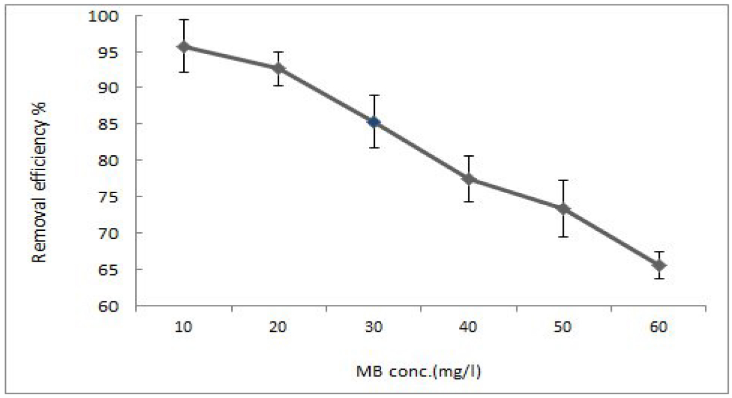


Fig. 4. Influence of initial concentration on adsorption of Mb.

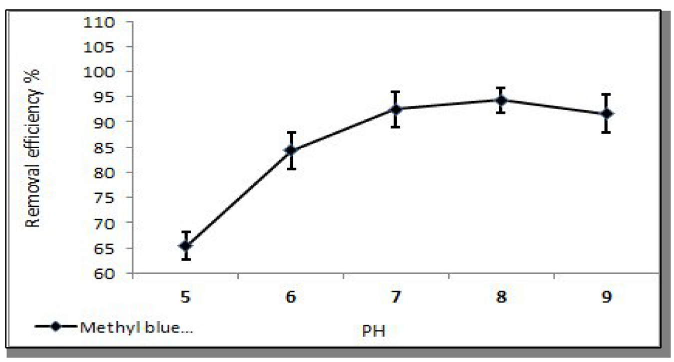


Fig. 5. Influence of PH valve on adsorption of Mb.

of pH on the elimination efficiency of Mb in aqueous solution. The results indicated that the dye had the maximum elimination efficiency (94.3%) at a pH of 8, at 60 minutes of contact time. Each experiment used 1.5g of adsorbent with 40mL of MB dye solution, at 25°C. Adsorption of Mb dye ions increases under basic conditions at pH 8. This is because, being a cationic dye, MB produces positively charged ions when it dissolves in aqueous liquids.[30] Increasing the solution's pH encourages the adsorbent surfaces to deprotonate, which raises the negative charges on the surface and, consequently, increases the biosorption capacity of Mb through electrostatic attractions.[26] On the other hand, the removal rate of Mb dye was observed to decrease at acidic pH levels, due to competition between hydrogen ions and positively charged dye molecules.[31] The results of this study are consistent with those of.[20,32,33]

Impact of temperature

The temperature is one of the most critical components impacting the process of adsorption.[34] Fig. 6 shows that the efficacy of eliminating Mb dye increased as the temperature increased; the best elimination efficiency was recorded at 50°C and was 96.2%, at 60 minutes of contact time, 1.5 g/40 mL dosage, PH of 8, and a speed of 150 rpm. This is because raising the temperature causes the dye molecules' kinetic energy or the adsorbent material's surface activity to increase, i.e., increasing the active sites for adsorbing methylene dye. Temperature is a significant physicochemical factor that affects the adsorption capacity of pollutants in a solution, and the adsorption mechanism in this experiment was endothermic, as the removal efficiency increased with rising temperature.[26] It is consistent with many studies that show that increasing temperature affects the adsorption process of Mb dye as.[35,36]

Influence of sorbent doses

To establish the optimal dose, the influence of biomass((ZSC) on Mb dye absorption was tested separately at concentrations ranging from 0.25 to 2 g per 40 ml of dye solution. Fig. 7 shows that it was observed that by increasing the dose of the sorbent from 0.25 to 1.5 g/L, the dye adsorption efficiency increased from 70.3% to 96.5% at a pH of 8, at 60 minutes of contact time, 25°C, and a speed of

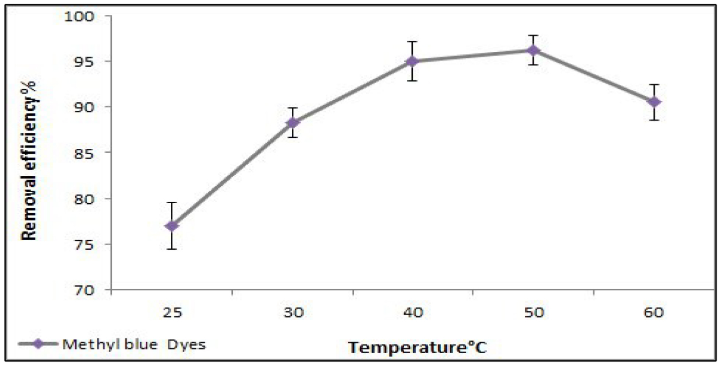


Fig. 6. Influence of temperature on adsorption of Mb.

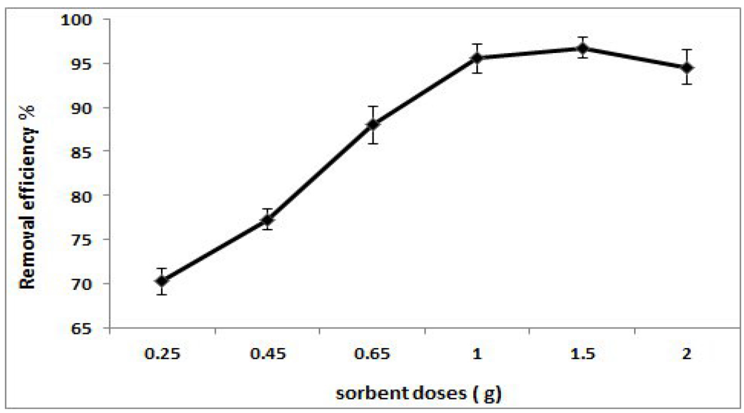


Fig. 7. Influence of sorbent doses on adsorption of Mb.

150 rpm. This may be due to the increase in the surface area of the sorbent, which increases the number of adsorption sites accessible for adsorption, as previously reported.[7,18] However, once the sorbent reaches its optimal concentration, a slight decrease in its adsorption capacity was observed. This decline is explained by the sorbent molecules' tendency to aggregate, resulting in a reduction of the available surface area with active site, hence lowering adsorption capacity.[37] The findings are consistent with.[38,39]

Kinetics modeling of methylene blue (Mb) adsorption

To assess the experimental results, three distinct kinetic models were examined in this study: a zero-order model, a pseudo-first-order model, and a pseudo-second-order model. Table 1 displays the properties of each kinetic model along with the corresponding correlation coefficient (R^2). The linear graphs in Fig. 8. (A, B, C) indicate that all three models are applicable. The correlation coefficients for the zero-order reaction, pseudo-first-order, and pseudo-second-order models are 0.6448, 0.9695, and 0.8813, respectively. The adsorption kinetics of Mb

Table 1. Adsorption kinetic parameters of Mb onto activated (ZSC).

Kinetics model	Parameters	
Zero-order kinetics	R^2 0.6448	K (C). (t^{-1}) 0.256
First-order kinetics	R^2 0.9695	K (t^{-1}) 0.1151
Second-order kinetics	R^2 0.8813	K (C^{-1}). (t^{-1}) 0.1176

dye were analyzed using different models. The correlation coefficients for the Mb dye indicate that the pseudo-first-order model is the most appropriate, as it showed the highest correlation coefficient (R^2 = 0.9695), accurately describing the adsorption process. Kinetics affects the adsorption rate, which in turn influences the time required to reach equilibrium in the adsorption process, thereby providing a deeper understanding of the adsorption process and its underlying mechanism.[20]

Scanning electron microscopy (SEM)

As seen in Fig. 9, SEM was employed to examine the surface morphology of the changed (ZSC) leaves. The pre-treatment of the leaves led to alterations in the surface composition and increased roughness and

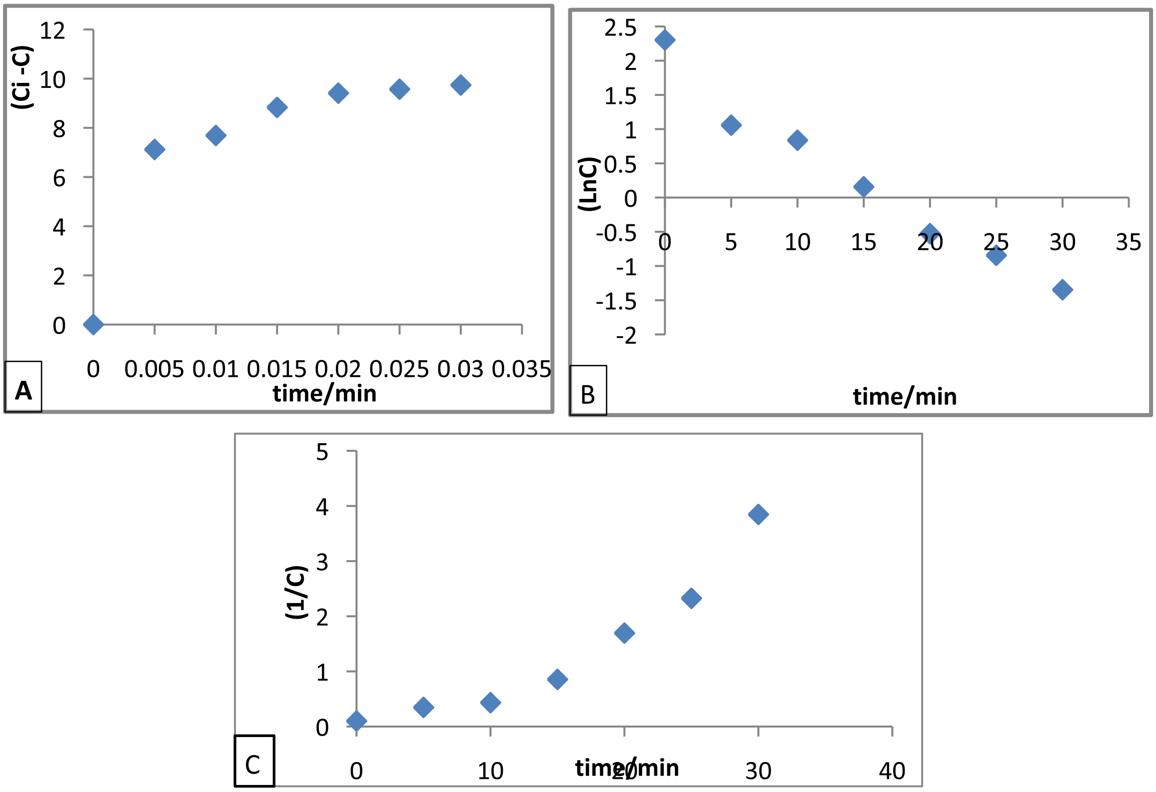
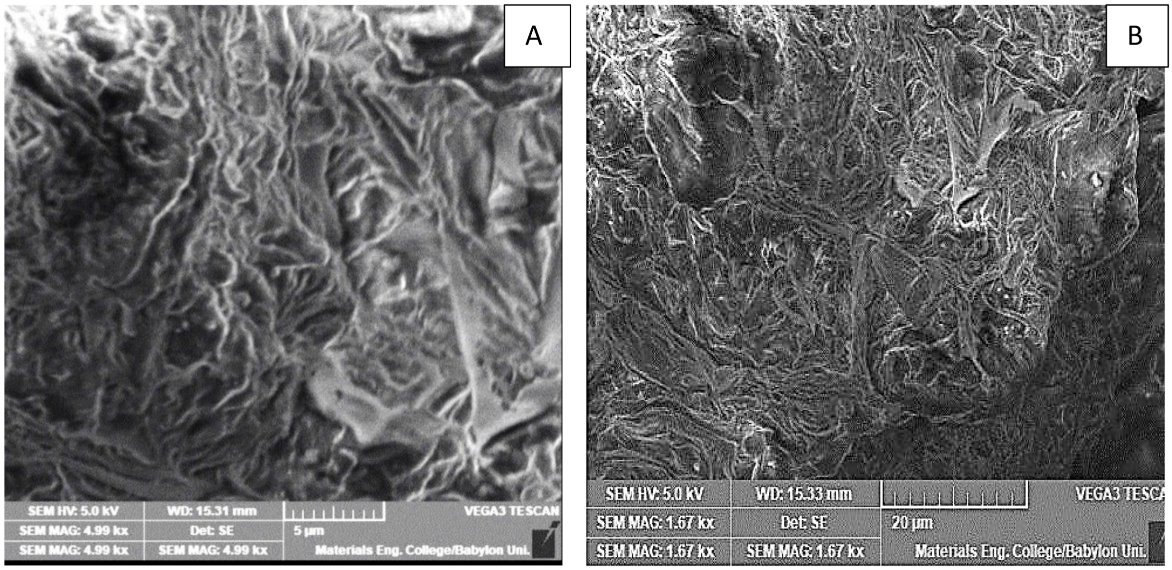


Fig. 8. Kinetics models for Mb dye: (A. zero-order-kinetic; B Pseudo-first-order kinetic model; and C) pseudo-second-order kinetics.

porosity of the leaves. Chemical treatment increases the leaves' capacity to absorb dyes from aqueous solutions by cleaning and removing the natural colors in the leaves and expanding the pores that they contain.[40] Fig. 9 (a, b) shows the before and after adsorption of Mb dye from aqueous solution. Before adsorption, the outer surface of the adsorbent ZSC leaves had a rough and uneven shape, with cavities and medium-porous structures. This heterogeneous surface plays a key role in the R% by providing multiple active sites that enhance adsorption, which are important for the molecular bonding of Mb to the components of the different materials, as in Fig. 9(a). In contrast, after adsorption, the gaps, cavities, and pores on the adsorbent surface decrease, resulting in a less porous structure. The removal efficiency is influenced by both the external surface and internal pores, which provide additional sites for dye adsorption and enhance overall capacity, as in Fig. 9(b). It is consistent with previous research that has demonstrated that the outer surface form of the material is crucial for the adsorption of the dye Mb.[41,42]

Fourier transform -infrared spectroscopy (FTIR)

In Fig. 10(a,b), The FTIR spectra (600–4000 cm-1) of the (ZSC) leaves before and after the sorption of Mb are shown. The major functional groups that improve dye adsorption at peak levels are taken into account in the spectra. FT-IR analysis showed that the leaves of the ZSC plant contain many active functional groups (C=O, C=C, C-O, and hydroxyl) that contribute to the Mb dye's adsorption. In general, there are many bands attributed to groups O-H and C-H. The peak at position 3410.15 cm^{-1} represents the stretching vibration of the (O-H) alcohol group, where this group appears between 3300 and 3400 cm^{-1}. The peak at 1627.92 cm^{-1} signifies the bending vibration of water because the fibers of hemicellulose have a high affinity for water. The peak at position range 2924.09-2854.65 cm^{-1}, attributed to stretching vibration of (C-H) in the CH_2 and CH_3 groups, respectively. The absorption peak at 1056.99 cm^{-1} represents the bending vibration of the C-OH group. There is no big change in the areas or positions of the peak when comparing the two spectra after and before adsorption. A slight shift in the location of certain peaks was observed following the adsorption of the Mb dye. This is attributed to interactions between the dye and functional groups (–OH, –NH_2, C=O) on the adsorbent surface. Still, no structural alteration was pointed out as a result of the emergence or disappearance of other peaks. This might be explained by the ZCS adsorbent's preservation of



A. before- adsorption B. after- adsorption

Fig. 9. SEM for (ZSC) leaves before and after treatment of methylene blue.

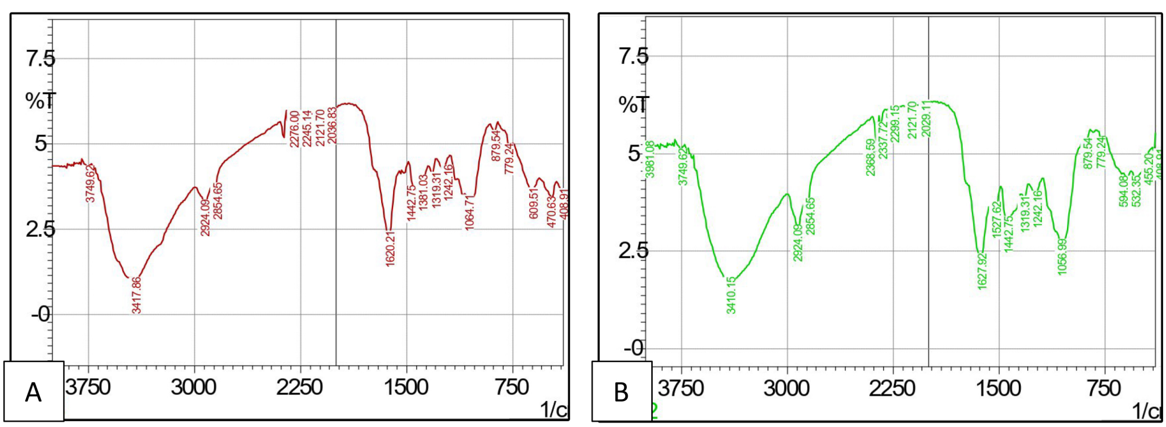


Fig. 10. Spectrum of infrared of activated (ZSC) leaves before (A) and after (B) adsorption.

its physicochemical characteristics following the adsorption of Mb. This agrees with that found in the study of.[43] The FTIR analysis revealed that the adsorbed ZSC leaf powder had almost identical main absorption bands before and after methylene blue dye adsorption, indicating the stability of the main active groups. However, slight shifts in the peak positions and changes in their intensity were observed after adsorption.[44] These changes reflect the interaction between the methylene blue dye molecules and the active groups (such as -OH, -C=O, and -NH) on the adsorbent surface, confirming their role in the adsorption process. This indicates that the adsorption process was physical or surface chemical. It occurred through surface interactions without the formation of new chemical bonds.[45,46]

Conclusion

The main objective of this work is to investigate the ability of hydrochloric acid-modified Ziziphus leaves (ZSC) to adsorb and remove the cationic dye Mb from aqueous solutions. This study demonstrated the high efficiency of the ZSC adsorbent in removing Mb dye under different laboratory conditions. The highest rate of removal efficiency was 97.51% under the optimal circumstances, highlighting the suitability of the material for practical applications in wastewater treatment. These results indicate that *Ziziphus Spina Christi* powdered leaves (ZSC) can be an environmentally friendly and cost-effective solution for treating dye-contaminated water. The results of the current study also provide a foundation for further research to improve the adsorption process and explore its practical applications in the treatment of diverse environmental pollutants

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images that are not ours have been included with the necessary permission for re-publication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Babylon.

Authors' contributions statement

Z. H. O. created the approach, conducted the data analysis, and wrote the first draft, Sample collection and laboratory work were done by Z. H. O., N. S.N. and A. R. O.; J. M. S. supervised the discussion of the findings and made edits to the final draft.

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دراسة الامتزاز والحركية لإزالة الصبغة السامة (الميثيلين الأزرق) من مياه الصرف الصناعي المحاكاة باستخدام مواد مستدامة

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الخلاصة

يُعد تلوث المياه مشكلة عالمية تؤثر على صحة الإنسان والبيئة. ويشكل الاستخدام المتزايد لصبغة الميثيلين الأزرق (Mb) في مختلف التطبيقات الصناعية مخاطر كبيرة. هدفت الدراسة إلى استخدام مسحوق أوراق نبات السدر (Zisiphus Spina Christi) المعدلة بحمض الهيدروكلوريك كممتز فعال ومنخفض التكلفة لإزالة صبغة الميثيلين الأزرق من النفايات السائلة. أُجريت تجارب على دفعات، بأوقات تلامس تتراوح بين 10 و180 دقيقة، وتركيزات أولية للصبغة تتراوح بين 10 و60 ملغم/لتر، ودرجات حرارة تتراوح بين 25 و60 درجة مئوية، وقيم pH تتراوح بين 5 و9، وجرعات ماصة تتراوح بين 0.25 و2 غرام لكل 40 مل من محلول الصبغة. أظهرت النتائج أن أعلى كفاءة إزالة لصبغة MB بلغت 97.51% باستخدام تركيز ابتدائي قدره 10 ملغم/لتر، و1.5 غرام من المادة الماصة، ودرجة حموضة 8، عند درجة حرارة 50 درجة مئوية، وزمن تلامس 60 دقيقة، وحجم جسيمات 0.075 ميكرومتر، وسرعة تحريك 150 دورة في الدقيقة. وُصفت مادة ZSC باستخدام تقنيتي المجهر الإلكتروني الماسح (SEM) وتحويل فورييه للأشعة تحت الحمراء (FTIR)، وتبعت حركية الامتزاز نموذجًا شبه من الدرجة الأولى، بمعامل ارتباط قدره 0.969. بشكل عام، تُعد مادة ZSC المعدلة مادة ماصة فعالة وسهلة الاستخدام واقتصادية لإزالة صبغة Mb من المحاليل المائية.

الكلمات المفتاحية: المخاوف الصحية، أزرق الميثيلين(Mb)، الاستدامة، تلوث المياه، أوراق شجرة الزيزفون (ZSC).