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Adsorption of Methylene Blue Dye on Chemically-Modified Agricultural Leaf Waste: Mini-Review

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Abstract: Methylene blue (MB) dye in water sources poses a significant threat to aquatic ecosystems and human health, prompting the search for cost-effective and sustainable adsorbents. This mini-review examines the potential of chemically modified agricultural leaf waste as a sustainable and cost-effective adsorbent for MB removal. Categorized various chemical modification techniques, including acid and alkaline treatments, as well as impregnation with different agents, and evaluated their impact on adsorption performance. This review presents recent findings on the effectiveness of these modifications, with adsorption capacities reaching up to 950 mg/g. In addition to assessing the effectiveness of these modifications, this review also summarizes the key factors influencing the adsorption process, such as pH, temperature, concentration, and dosage. Ultimately, chemically-modified leaf-based agricultural waste provides a viable and promising sustainable solution for the removal of MB from wastewater, highlighting the feasibility of utilizing abundant and renewable agricultural waste for MB removal in wastewater treatment and contributing to a greener and more sustainable future.

Keywords: adsorbent; adsorption; agricultural leaf waste; chemical modification; methylene blue dye.

1. Introduction

The past few decades have witnessed a remarkable surge in global economic growth and development. However, this progress has come at a cost to the environment. Industries, agriculture, and urbanization have increased the demand for water resources and generated hazardous waste, leading to extensive environmental pollution. This pollution affects soil, plants, and water bodies on a large scale, endangering living organisms and necessitating effective wastewater treatment [1].

Among the various pollutants threatening water resources, dyes from industries like textiles, printing, and leather pose a significant challenge [2]. These industries alone consume large quantities of water and utilize various toxic chemicals during production [3], resulting in substantial volumes of highly colored wastewater containing a complex mixture of pollutants [4,5].

Dyes, which are complex organic compounds, are used extensively in these industries, leading to significant water contamination [6]. Global dye production is estimated at 0.7–1.6 million tons annually, with a concerning 10–15% of this production released as wastewater [7-9]. The discharge of untreated dye-laden wastewater into water bodies has severe consequences for aquatic ecosystems and human health. Even at low concentrations, dyes hinder light penetration and disrupt oxygen transmission, affecting vital ecological processes such as photosynthesis and respiration [10-14]. This disruption leads to a decline in biodiversity and can negatively impact human health due to the toxic nature of many dyes.

Methylene blue (MB) is a commonly used heterocyclic aromatic compound that belongs to the thiazine class and significantly contributes to dye pollution in wastewater [15]. Methylene blue has a molecular weight of 319.85 g/mol and maximum light absorption at 663 nm [16,17]. Its diverse applications range from textiles and pharmaceuticals to food coloring [18-22]. Methylene blue also has medical applications, including the treatment of malaria, fungal infections, and methemoglobinemia [23], and it shows promise in treating skin conditions [24,25]. However, recent reports have raised concerns about potential health risks associated with MB exposure, including respiratory problems, digestive issues, and vision impairment [26,27].

Even at low concentrations, the presence of MB in water bodies disrupts aquatic ecosystems. The intense color of MB and its by-products blocks light penetration, affecting photosynthesis and reducing oxygen solubility [28,29]. This reduces biodiversity and negatively impacts the aquatic environment [30,31]. Consequently, the unregulated discharge of dye-laden wastewater poses significant ecological risks, necessitating stringent regulations and comprehensive treatment prior to release [32].

Scientists have developed various treatment methods to address the growing concern over dye contamination, including physicochemical methods like sedimentation, coagulation, and electrochemical processes, as well as biological treatments and advanced techniques like electrocoagulation, reverse osmosis, and oxidation-precipitation [33,34]. However, these techniques have limitations. Physical methods often involve high costs associated with specialized materials and maintenance. Although chemical coagulation is effective, it can generate secondary pollutants. Biological methods are typically sensitive to variations in wastewater composition and can produce large volumes of sludge by-products, which require further management or disposal [35-38].

Among various physicochemical techniques, adsorption offers a promising solution for dye wastewater treatment due to its exceptional ability to remove a wide range of dye pollutants [33]. This process involves the accumulation and adhesion of dye molecules (adsorbates) onto the surface of a solid material (adsorbent) [39]. Adsorption occurs through mechanisms like physisorption, which involves weak van der Waals forces, and chemisorption, which involves stronger chemical bonds between the adsorbate and adsorbent [39]. The surface area, porosity, and functional groups of the adsorbent determine adsorption effectiveness [40]. A larger surface area provides more interaction sites, porosity facilitates dye penetration, and specific functional groups attract and bind dye molecules. These factors influence the rate and extent of dye removal. In addition, adsorption offers several advantages: simplicity, minimal harmful by-products, high dye removal rates, and ease of operation [1].

Researchers are increasingly investigating agricultural by-products, such as leaf waste, as sustainable and cost-effective adsorbents for dye removal [41]. These abundant and readily available materials often exhibit good adsorption properties due to their natural composition, which includes cellulose, hemicellulose, and lignin [42-44]. Several studies have explored the potential of various agricultural waste materials, such as enset midrib leaf [45], cassava leaves [46], mango leaves [47], and durian leaves [48], as adsorbents for MB. These studies report adsorption capacities ranging from 35 to 250 mg/g, demonstrating the potential of these materials for effectively removing MB from wastewater. Given the promising potential, this mini-review focuses on the effectiveness of modified leaf-based materials for MB adsorption from wastewater. We categorized and assessed the performance of different chemical modification techniques applied to leaf waste in removing MB, thereby contributing to the development of sustainable and cost-effective strategies for dye wastewater treatment.

2. Chemical Modification Techniques

Adsorbents are commonly modified prior to their application in pollutant removal. The primary aim of this modification is to enhance the adsorbent capacity for pollutant uptake [49]. Among the various modification techniques, chemical modification using different reagents is a popular choice due to its straightforward approach. This typically involves a single-step process conducted at lower temperatures, making it a relatively simple and efficient technique compared to other methods [50]. Through these chemical treatments, functional groups are introduced or enhanced on the adsorbent surface, which increases surface area and porosity and creates active binding sites. This combination of effects ultimately improves the overall adsorption capacity of the material [51].

2.1. Acid modification.

Acid modification is widely used to enhance the adsorption capacity of various materials. This process typically involves treatment with mineral acids (hydrochloric acid (HCl), sulfuric acid (H_2SO_4), nitric acid (HNO_3), etc.) or oxidizing agents such as hydrogen peroxide and hypochlorous acid [52-56]. Acidic functional groups (carboxyl, quinone, hydroxyl, carbonyl, etc.) are introduced onto the adsorbent surface through this process [57]. These functional groups contribute to the increased polarity and hydrophilicity of the adsorbent [58]. The overall effect is a more negatively charged surface, enhancing the adsorption of positively charged pollutants through electrostatic interactions. Additionally, H^+ ions can be introduced onto the surface through acid treatment, facilitating the adsorption of certain negatively charged pollutants via a different mechanism [59]. Furthermore, strong acids like H_2SO_4 and HNO_3 can introduce functional groups containing elements like nitrogen, sulfur, and phosphorus, potentially creating additional adsorption sites [60]. The adsorption capacity can be further enhanced through various interactions with pollutants facilitated by these additional functional groups.

The removal of MB dye from aqueous solutions using acid-treated *Hevea brasiliensis* leaves was investigated by Jawad et al. [61]. The removal efficiency was evaluated at various dye concentrations (50–300 mg/L) and a broad pH range (3–11) using batch experiments. The leaves were collected, washed, oven-dried, crushed, sieved, and acid-treated with a 1:1 concentrated H_2SO_4 solution to enhance adsorption. A total pore volume of 0.00342 cm^3/g and a surface area of 1.65 m^2/g were revealed through the characterization of the modified adsorbent (ATRL). Additionally, the presence of carbon, oxygen, and sulfur on the leaf surface was confirmed by energy-dispersive X-ray analysis. The morphology of the adsorbent appeared highly porous and heterogeneous, resembling a tunnel or honeycomb-like structure.

The Freundlich isotherm was found to best describe MB adsorption, suggesting multilayer coverage on the ATRL surface. The maximum adsorption capacity of ATRL for MB was determined to be 263.2 mg/g at 303 K. Furthermore, the adsorption process was better represented by the pseudo-second-order (PSO) kinetic model.

The use of phosphoric acid-treated eucalyptus leaves as an adsorbent for MB dye removal from aqueous solutions was studied by Ghosh et al. [62] using batch and column adsorption studies. Optimal conditions for MB removal, including pH, contact time, initial dye concentration, and adsorbent dosage, were identified through batch adsorption experiments. The equilibrium data were best described by the Langmuir isotherm model, indicating a maximum monolayer adsorption capacity of 52.18 mg/g. Column adsorption studies revealed that the percentage of MB adsorption increased with bed height but decreased with flow rate and initial MB concentration. The breakthrough curves obtained from the column experiments were effectively fitted to the Thomas model. A regeneration efficiency of approximately 55.10% was revealed in the desorption study, suggesting the potential for reusability of the treated eucalyptus leaves.

Acid-treated *Enset ventricosum* midrib leaves (EVML) were explored for MB removal by Mekuria et al. [45]. The raw material was pre-treated with 0.1 M HCl to remove colored components. Subsequently, the treated leaves were washed and oven-dried. The physicochemical characterization of the modified EVML included moisture content, dry matter, surface area, and point of zero charge. Batch adsorption experiments were conducted using a 10 mg/L MB solution, 2.5 g/L adsorbent dosage, and an initial pH of 5.7. An increase in adsorption was observed with a higher initial pH of the MB solution. The adsorption process was best described by the Langmuir isotherm model, indicating a maximum capacity of 35.5 mg/g. Furthermore, significant MB removal efficiency was demonstrated even after five cycles in reusability studies using 0.1 M HCl and sodium hydroxide (NaOH) solutions for regeneration, suggesting the potential of EVML for multiple applications in MB-contaminated water treatment.

2.2. Alkaline modification.

Alkaline modification is a widely used chemical treatment method employed to tailor the surface properties of adsorbents, ultimately enhancing their performance in pollutant removal applications. In this process, the adsorbent is typically treated with various alkaline agents, such as NaOH, potassium hydroxide, lithium hydroxide, sodium silicate, and sodium carbonate [63,64]. This treatment alters the surface functional groups of the adsorbent, leading to an increase in the non-polar character of the adsorbent surface [65]. The adsorption of non-polar pollutants can be improved through van der Waals interactions or other non-covalent interactions due to this enhanced non-polarity [56]. Additionally, positively charged sites can also be introduced onto the adsorbent surface through alkaline treatment [58]. Negatively charged pollutants (anions) in the solution can be electrostatically attracted to these positively charged sites, further enhancing the adsorption capacity.

The potential of clove leaf waste as a low-cost biosorbent for MB dye removal was explored by Kusuma et al. [63]. The effects of initial dye concentration, biosorbent dosage, and contact time were examined to evaluate the adsorption process. The adsorption kinetics were best described by the pseudo-first-order model. The equilibrium data were effectively fitted by Langmuir and Freundlich isotherm models, suggesting a possible heterogeneous adsorption mechanism involving monolayer and multilayer coverage. The thermodynamic parameters revealed an endothermic and non-spontaneous nature of the adsorption process. A maximum adsorption capacity of 9.80 mg/g was reached by the optimized clove leaf biosorbent (CL-NaOH), highlighting its potential as a cost-effective and recyclable biomaterial for MB removal from water.

The effects of NaOH treatment on durian leaf powder (DLP) for MB adsorption were studied by Hussin et al. [66]. Vital interactions between the modified adsorbent (NaOH-DLP) and MB dye were revealed by characterization techniques. The impact of various parameters on MB removal was investigated through batch adsorption experiments. The equilibrium data were best described by the Langmuir isotherm model (R^2 = 0.989), suggesting monolayer adsorption with a high capacity (125 mg/g). Kinetic studies indicated a good fit with the PSO model, implying a chemisorption mechanism. The potential of NaOH-DLP as an effective and high-capacity adsorbent for MB removal from wastewater is highlighted by these findings.

Similarly, NaOH-treated *Leucaena leucocephala* leaf powder for MB removal from aqueous solutions was investigated by Hanafiah et al. [67], with an examination of the impact of various factors on the adsorption process. Batch adsorption experiments were conducted using a 100 mg/L MB solution, 2.5 g/L adsorbent dosage, a neutral pH of 7, and an ambient temperature. The adsorption capacity of leaf powder was significantly enhanced by NaOH treatment, with 97.94% removal efficiency and a maximum removal capacity of 203.28 mg/g. This suggests a higher affinity for MB, likely due to changes in the leaf powder surface properties. Kinetic and isotherm studies revealed that the adsorption process followed Langmuir and PSO models, suggesting efficient MB adsorption.

2.3. Modification by other chemical methods (impregnation and organic agents).

Beyond acid-base modifications, impregnation offers another avenue for tailoring adsorbent surfaces. This technique involves the uniform distribution of a desired chemical within the porous structure of the adsorbent. Research has demonstrated that impregnation can enhance stability, facilitate regeneration, and ultimately improve the practical utility of the adsorbent [58, 63]. Impregnating agents encompass a wide range of materials, including oxidizing agents (e.g., potassium permanganate), neutral agents (e.g., zinc chloride ($ZnCl_2$), sodium chloride), metal cations (e.g., iron(III) chloride, cerium), and even organic solvents like ethanol [64]. Specific functionalities can be introduced to the surface by each type of impregnant, potentially improving adsorption capacity. However, limitations are posed by organic solvents due to cost, safety concerns, and instability [68].

The surface area, yield, and adsorption capacity of adsorbents have been shown to increase through impregnation with neutral agents, particularly when using agricultural waste materials [69]. Despite their effectiveness, environmental concerns are often raised by these activators. They can be caustic, hazardous, and toxic at high concentrations, and explosions can result from improper handling after impregnation, such as heating. Water hardness issues can be caused by the dissociation of some neutral agents, like calcium chloride, upon disposal [69].

In an endeavor to further enhance the adsorption properties of various adsorbents using greener chemical activators, additives such as urea, cetyltrimethylammonium bromide (CTAB), polyacrylamide, sodium methylate, and sodium dodecyl sulfate were introduced by Yeneneh et al. [70] for binding with the adsorbent and enhancing its adsorption capacity. Although some improvements in adsorption efficiency were observed, further research is needed to identify even more environmentally friendly activators and to elucidate the precise mechanisms by which these additives enhance adsorption. Impregnated adsorbents generally exhibit improved stability compared to unmodified materials, making them more resistant to degradation during use [71]. Reusable and long-lasting adsorbents can be created through this improved stability and easier regeneration, making impregnation a valuable technique.

$ZnCl_2$/HCl-modified *Ocimum basilicum* leaves for MB adsorption were investigated by Bani-Atta et al. [72]. The leaves were boiled, refluxed with $ZnCl_2$, and boiled again with HCl for modification. A high surface area (117.27 m^2/g) and well-developed porosity (total pore volume of 2,570.951 cm^3/g and average pore diameter of 264.144 Å) were revealed through characterization. Temperature was identified as a critical factor in batch experiments, with adsorption enhanced by higher MB concentrations but hindered by increased temperatures. The adsorption equilibrium data fit well with the Langmuir isotherm model, suggesting monolayer coverage on the adsorbent surface. Kinetic studies indicated a PSO model. An exothermic and spontaneous adsorption process was revealed by thermodynamic analysis.

Ethanol-modified cassava leaves for MB adsorption were investigated by Theng et al. [46]. Pigment interference was minimized through microwave pre-treatment, and the leaves were soaked in 80°C ethanol for 6 h to enhance adsorption. Increased MB removal with a higher adsorbent dosage was revealed by batch experiments, reaching 99.9% under optimized conditions (pH 11.0, 45°C, 0.5 g, 60 min). It was suggested by adsorption isotherm studies that the MB adsorption process followed both Langmuir and Freundlich models, indicating a combination of monolayer and heterogeneous surface adsorption.

Cetyltrimethylammonium bromide as a cationic surfactant for modifying mango leaves (*Mangifera indica* L) for MB removal was explored by Musawwa et al. [47]. The leaves were first activated with NaOH to remove color, followed by CTAB modification. The study revealed a significant impact of pH on adsorption, with an optimum pH observed in the basic region (pH 8). Although the optimal adsorbent mass and MB concentration were not definitively identified from the presented data, the modified leaves exhibited a high adsorption capacity of 950.75 mg/g. Kinetic studies indicated a PSO model for the adsorption process. Table 1 presents a compilation of diverse agricultural waste materials employed as low-cost adsorbents in various research studies.

Table 1. Leaf waste is utilized as a low-cost adsorbent for MB removal.

Leaf waste	Initial conditions	Removal efficiency (%)	Isotherm models	Kinetic models	Maximum adsorption (mg/g)	References
Hevea brasiliensis leaves	Dosage: 0.02–0.30 g Time: 1,440 min pH: 3–11 Temperature: 303 K MB concentration: 50–300 mg/L	Nil	F	PSO	263.2	[61]
Eucalyptus leaves	Dosage: Nil Time: 360 min pH: 2–10 Temperature: 25 °C MB concentration: 100 mg/L	Nil	L	Nil	52.18	[62]
Enset ventricosum midrib leaves	Dosage: 2.5 g/L Time: 24 h pH: 5.7 MB concentration: 10 mg/L	95	L	PSO	35.5	[45]
Clove leaf	Dosage: 0.01–5.0 g/L Time: 30 min pH: 11 Temperature: 303.15 K	Nil	L, F	PFO	9.80	[66]

Leaf waste	Initial conditions	Removal efficiency (%)	Isotherm models	Kinetic models	Maximum adsorption (mg/g)	References
	MB concentration: 0.05 mg/L					
Durian leaf	Dosage: 0.02–0.10 g Time: 200 min pH: 2–10 Temperature: 30 °C MB concentration: 10–40 mg/L	Nil	L	PSO	125	[48]
Leucaena leucocephala leaf	Dosage: 0.02–1.00 g Time: 90 min pH: 2–11 Temperature: 308 k MB concentration: 10–30 mg/L	97.94	L	PSO	208.33	[67]
Ocimum basilicum leaves	Dosage: 00.5–0.035 g Time: 26 h pH: 2–12 Temperature: 20–50 °C MB concentration: 500 mg/L	76.4	L	PSO	714.29 666.67 625.00 555.56	[72]
Cassava leaves	Dosage: 0.5 g Time: 60 min pH: 11 Temperature: 45 °C MB concentration: 100 mg/L	99.9	L, F	Nil	Nil	[46]
Mango leaves	Dosage: 0.1–0.7 g Time: 60 min pH: 8–11 Temperature: Nil MB concentration: 25–200 mg/L	99.38	Nil	PSO	950.75	[47]

L = Langmuir; F = Freundlich; PFO = Pseudo-first order; PSO = Pseudo-second order; RT = Room temperature; Nil: Not available.

3. Factors Influencing Adsorption Performance

The effectiveness of an adsorbent in dye removal depends on a combination of its inherent properties and the external factors present during adsorption. The availability of adsorption sites is influenced by fundamental intrinsic properties, including surface area and porosity [50]. Additionally, the stability and ability of the adsorbent to retain its adsorption capacity under varying environmental conditions are crucial. Adsorption is significantly affected by external factors, such as solution pH, temperature, initial dye concentration, and adsorbent dosage [40]. These parameters must be optimized to scale up dye removal processes for industrial applications.

3.1. Effect of pH.

Electrostatic interactions between adsorbents and dye molecules are governed by solution pH, which ultimately affects adsorption efficiency [73,74]. The adsorption of anionic dyes is favored in acidic solutions due to electrostatic attraction to the positively charged surface of the adsorbent, while the removal of cationic dyes is hindered. Conversely, cationic dye adsorption is promoted in a basic environment due to the negatively charged surface of the adsorbent [73,74]. The influence of pH on dye adsorption is demonstrated in practical applications. For example, anionic dye adsorption is facilitated through protonation in acidic media using HCl, whereas the surface is deprotonated by NaOH in basic media, which hinders anionic dye uptake but promotes the adsorption of cationic dyes [40].

3.2. Effect of initial dye concentration.

Adsorption is significantly influenced by the initial dye concentration in wastewater. The mass transfer of dye molecules to the adsorbent is affected by the initial concentration, which ultimately impacts the attainable adsorption capacity [75]. An exponential increase in adsorption is generally observed with higher initial concentrations due to the greater availability of active sites on the adsorbent surface [2]. However, a plateau in adsorption capacity is reached as these sites become saturated, and a decrease may even occur at extremely high dye concentrations [44, 76].

3.3. Effect of adsorbent dosage.

Adsorption is influenced by adsorbent dosage, which is a critical parameter. As the amount of adsorbent increases, the total available surface area and the number of adsorption sites also increase, typically resulting in a higher percentage of dye removal [77,78]. However, this increase is accompanied by a decrease in the amount of dye adsorbed per unit mass of adsorbent [79]. This phenomenon can be attributed to two possible mechanisms: the incomplete saturation of adsorption sites at higher adsorbent concentrations [80-83] and the potential aggregation of adsorbent particles, which reduces the effective surface area and hinders dye diffusion [84].

3.4. Effect of temperature.

Physicochemical adsorption is influenced by temperature, which is another crucial parameter. This influence arises from the impact of temperature on both adsorption capacity and the nature of the process, which may be endothermic or exothermic [85]. Higher adsorption capacity is achieved in endothermic processes with increased temperature. This phenomenon can be attributed to the activation of the adsorbent surface at higher temperatures, resulting in greater availability of active sites and enhanced mobility of dye molecules [86,87]. Conversely, adsorption capacity decreases with increasing temperature for exothermic processes. This is explained by the weakening of interactions between the active sites of the adsorbent and the dye molecules at higher temperatures, leading to a decline in dye removal efficiency [88].

4. Conclusions and Future Perspectives

This mini-review aims to evaluate the potential of chemically modified agricultural leaf waste as a cost-effective and sustainable adsorbent for MB removal from wastewater. The review establishes that chemical modification significantly enhances the adsorption capacity of leaf waste for MB, with acid treatment, alkaline treatment, and impregnation identified as effective modification methods. Based on the analysis of the research, the following conclusions are drawn: chemical modification of agricultural leaf waste significantly increases its adsorption capacity for MB, acid treatment, alkaline treatment, and impregnation with various agents are effective methods for modifying leaf waste for MB adsorption, modified leaf waste exhibits increased active sites for MB adsorption, leading to enhanced MB removal, the effectiveness of these modifications is influenced by factors such as pH, temperature, and initial dye concentration, highlighting the importance of optimizing these parameters, chemically-modified leaf-based agricultural waste is a viable, promising sustainable, and cost-effective adsorbent for MB removal from wastewater.

These conclusions highlight the significant potential of chemically modified leaf waste for MB removal, but further research is needed to optimize this approach and realize its full potential in the following areas: systematically investigate and compare the effectiveness of various modifying agents and reaction conditions to maximize adsorption capacity and efficiency, develop and evaluate cost-effective and environmentally friendly methods for regenerating spent leaf-waste adsorbents, enabling their reuse and reducing waste generation, investigate and establish safe and sustainable disposal or utilization strategies for spent leaf-waste adsorbents, such as composting, incineration, or repurposing as soil amendments, to minimize environmental impact, and explore novel modification techniques, such as grafting, and investigate the synergistic effects of combining existing methods to further enhance MB removal capacity and selectivity.

Author Contributions

Conceptualization, A.F. and A.A.; methodology, A.F.; validation, A.A., S.H., and A.F.; writing—original draft preparation, A.F.; writing—review and editing, A.F. and F.A.; visualization, A.A. and S.H.; project administration, A.A.; funding acquisition, A.F. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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