









RESEARCH ARTICLE



Valorization of Orange Peel-Derived Adsorbents for Sustainable Removal of Methylene Blue from Textile Industry Wastewater: A Promising Approach to Mitigate Water Pollution and Environmental Litter

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Abstract: Wastewater discharged from the textile industry poses significant environmental and societal challenges due to the presence of hazardous dye pollutants. This study explores a novel approach for the removal of methylene blue, a common textile dye, from water using adsorbents derived from orange peels. The goal is to mitigate water pollution, thereby reducing harm to ecosystems and living organisms, while simultaneously addressing the issue of environmental litter generated by discarded orange peels. In this study, the removal of methylene blue dye from textile

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wastewater using adsorbents made from orange peels was examined. Orange peels were collected, washed, dried, charred at 500 °C, ground into powder, and activated with phosphoric acid to create an

activated carbon with a bulk density of 0.987 g/mL, 15% moisture, 3% ash content, and a surface area of 52.5 m²/g. It was found that adsorption rates increased over time, reaching equilibrium after 120 min, with a maximum removal efficiency of 90% achieved using 0.6 g of adsorbent. The study concluded that orange peel-derived adsorbents could be an effective and eco-friendly method for reducing water pollution and repurposing agricultural waste, presenting a viable solution to the environmental challenges posed by the textile industry.

Keywords: methylene blue, wastewater, valorization, orange-peel, biomass, pollution

1. Introduction

Pollution continues to be a persistent and escalating global challenge, posing substantial health risks and environmental threats [1, 2]. Despite significant efforts to mitigate its effects, pollution remains a formidable issue [2, 3]. Particularly in the developing world, traditional forms of pollution, including inadequate sanitation, substandard waste management, industrial emissions, contaminated water sources, and indoor air pollution from biomass fuels, impose severe health hazards on communities [1]. Notably, environmental contamination is not limited to less developed countries, as even in developed nations, marginalized segments of the population endure its adverse effects [4]. The deleterious impacts of pollution often do not manifest immediately, primarily because the concentrations of pollutants rarely reach levels that can cause immediate harm. Instead, their consequences on health tend to be delayed or inconspicuous [5].

Pollutants come in various forms, including gases, liquids, solids, and energy, and understanding their sources is pivotal for effective pollution management. To this end, pollution sources can be categorized into two main groups: point sources and non-point sources [6]. Point sources are readily identifiable and include instances such as diesel pickup trucks emitting visibly dark exhaust fumes or pipes discharging liquid waste into rivers. In contrast, Non-point sources, also known as “diffuse pollution,” pose a challenge in pinpointing the exact origins of pollution [7]. In the case of non-point sources, determining the responsible individuals, households, or establishments for water pollution becomes complex. These sources encompass a wide range of origins, including domestic activities like sewage from toilets, kitchens, and bathrooms, industrial processes, agricultural practices such as irrigation, pesticide and fertilizer applications, runoff, and erosion, as well as pollution stemming from transportation activities, among others [6].

The repercussions of pollution extend across different sectors and manifest in various forms, including air pollution, water pollution, and soil and land contamination. These effects disrupt and destabilize ecosystems. For instance, water pollution can lead to a decline in fish populations and other aquatic species, thereby disturbing aquatic ecology and depriving local residents of food sources and livelihoods. Simultaneously, air pollution negatively impacts the health of humans, animals, and plants. Air pollutants contain chemicals that can dissolve in cloud water vapor and precipitate as acid rain, contributing to reduced lung function and the prevalence of respiratory conditions like asthma and bronchitis [8].

Given that approximately 75% of the Earth's surface is covered by water, water pollution emerges as a significant concern. It encompasses the contamination of water bodies such as oceans, lakes, rivers, and groundwater due to unwanted foreign substances introduced through human activities. These pollutants pose hazards to various organisms, including humans, animals, and plants [9].

One of the foremost contributors to water and soil pollution is textile industry wastewater (TIWW) [10]. This wastewater, laden with dyes and various contaminants, is typically discharged into

water bodies without proper treatment. The textile industry relies heavily on synthetic dyes, with an annual consumption exceeding 10,000 tons, drawn from a total production of around 7×10^7 tons of various dyes [11]. TIWW contains an array of pollutants, including salts, phenols, heavy metals, volatile organic compounds, binders, surfactants, alkalis, reducing agents, dioxin, phthalates, chlorobenzenes, dispersants, detergents, and pentachlorophenol, often in substantial concentrations [12].

In the textile industry, various types of dyes are utilized to impart color and aesthetics to fabrics. These dyes can be broadly categorized into different classes, such as azo dyes, anthraquinone dyes, reactive dyes, vat dyes, and others, each with unique chemical structures and properties. The choice of a specific dye depends on factors such as the type of fabric, desired color, and application method.

In the context of wastewater treatment studies, the selection of methylene blue as the target pollutant is often due to its widespread use in the textile industry, especially in dyeing cotton and wool. Methylene blue, a heterocyclic aromatic dye, belongs to the thiazine class of dyes. Its chemical structure consists of a central nitrogen-containing heterocycle, making it water-soluble and suitable for dyeing natural fibers.

There are several reasons why methylene blue was chosen as the model pollutant in this study:

1. Methylene blue is extensively used in the textile industry due to its versatility, stability, and vibrant color. Its widespread application makes it a representative dye for studying the efficiency of treatment processes.
2. Methylene blue is a cationic dye, and its chemical properties, such as solubility in water and positive charge, make it a suitable candidate for studying adsorption and removal processes in wastewater treatment.
3. The detection and quantification of methylene blue are relatively straightforward, enabling researchers to accurately measure its concentration in aqueous solutions. This facilitates the assessment of treatment efficiency.

Methylene blue, a cationic dye used in textiles, microbiology, surgery, and diagnostics, is known for its adverse effects when introduced into the environment. While not extremely hazardous, it can lead to an increased heart rate, shock, the formation of Heinz bodies, tissue necrosis, quadriplegia, jaundice, and cyanosis in humans [13]. Notably, it can cause eye burns, potentially resulting in lasting damage to the eyes of both humans and animals. Methylene blue, like many other synthetic dyes, also poses environmental challenges. It can persist in water bodies, leading to water pollution and affecting aquatic life. The dye's presence can hinder light penetration in water, impacting photosynthesis and disrupting the aquatic ecosystem. Additionally, the dye may undergo photodegradation, producing potentially harmful by-products.

Consequently, the treatment of effluents containing methylene blue is a matter of utmost importance. Dyes, in general, exhibit poor biodegradability and resist degradation by environmental factors, making the treatment of dye-contaminated wastewater particularly challenging. Among various techniques, adsorption on activated carbon stands out as a highly efficient and cost-effective method [14].

The production of activated carbon from orange peels through acid activation offers an environmentally friendly and economically viable solution for removing dyes from wastewater. Extensive studies have assessed the efficiency of this process using both batch and continuous models. These investigations have demonstrated the capability of orange peel-based activated carbon to remove dyes from single and binary dye solutions in both batch and continuous modes [15–17]. Equilibrium isotherms for single dyes have been well characterized by the Langmuir–Freundlich model, whereas equilibrium isotherms for binary dyes have been suitably described by single and multi-component models [18, 19]. Orange peels, activated through acid activation, offer substantial potential for the removal of basic dyes. This activated carbon exhibits a high adsorption capacity for dyes like methylene blue and Rhodamine B [18]. Notably, the adsorption mechanism remains largely unaffected by solution pH, a highly desirable feature for practical wastewater treatment, underscoring that the adsorption process is primarily influenced by the textural properties and dispersion interactions of the orange peel-based carbon [20].

The versatility of this process allows for exploring alternative fruit waste materials and alternative acids for fruit waste activation to achieve effective dye adsorption in water. Fruit waste materials, such as *Rapanea ferruginea*, and activation with alternative agents like ethanol and sulfuric acid, offer promising avenues for further research [15, 20]. This environmentally friendly process is both feasible and sustainable, utilizing materials readily available from marketplaces and fruit industries. Employing methylene blue as a target pollutant, this study investigates the impact of initial dye concentration, adsorbent dosage, particle size, and contact time on the adsorption efficiency of the prepared sample [21–23].

The aim of this study is to remove dye (methylene blue) from textile industry wastewater using adsorbents prepared from orange peels.

The following goals were used to achieve the study's aim:

1. Carry out the proximate analysis on prepared adsorbent to determine the levels of various components present in the activated carbon, such as moisture and ash.
2. Characterize the prepared adsorbent to determine surface area, total pore surface, average pore size, and bulk density.
3. Study the adsorption characteristics of methylene blue onto the prepared activated carbon with varying contact time, adsorbent dosage, and initial concentration.
4. Carry out adsorption isotherm study of the adsorption process.

This research is poised to advance our understanding of effective, eco-friendly solutions to combat water and soil pollution, while simultaneously addressing environmental waste through the valorization of fruit peels. It offers a promising approach to mitigate water pollution and reduce the environmental impact of industrial processes, emphasizing the importance of sustainable practices for a cleaner and healthier planet.

2. Materials and Methods

2.1. Sample collection

Orange peels were collected from neighborhood markets and fruit juice stands where they are readily available. The wastewater from the textile industry was produced with high-purity methylene blue sourced from Fisher Scientific. This makes it possible to analyze adsorption's impact on methylene blue in great detail.

2.2. Experimental materials

The following materials were used to carry out this study; 50 mL, 125 mL, and 500 mL conical flasks sourced from luco laboratory, Shimadzu digital weighing balance, pestle and mortar, 100-mesh screen sieve, thermo fisher scientific drying oven and muffle furnace, measuring cylinders, beakers and test tubes, 400 nm UV spectrophotometer sourced from shimadzu, and cyclomixer cycling vibrator.

2.3. Pretreatment of samples

2.3.1. Preparation of adsorbent

Oranges (*Citrus sinensis*) were obtained from a nearby market, washed with running water to remove debris and other remains, and then peeled. Following the procedures suggested by [15, 24, 25], the orange peels were sliced and broken into little pieces, sun-dried, oven dried at a temperature of 105 °C for 4 h. After the pieces were dried, they were ground into fine powder using a pestle and mortar. The dried sample was added to the crucible and heated for two h at 500 °C in a muffle furnace. In a 1000 ml beaker, 200 g of the carbonized material was placed. The sample was activated using an activating agent (phosphoric acid) and 1.0 M H₃PO₄ at a ratio of 1:3. A magnetic stirrer with a heating element was used to agitate the mixture, and the temperature was maintained at 200 °C for 2 h. The final product (activated carbon) was left to cool down for roughly 5 h after the activating process. The activated sample was dried in a 105 °C oven for 5 h, cooled to room temperature in a desiccator, and then placed in an airtight container for further use. But before storing, to make sure that the powder had uniformly sized particles, it was sieved in 0.5 mm sieve.

2.3.2. Preparation of adsorbate

As described by [25, 26], an amount of methylene blue dye of mass 0.1 g was suitably dissolved in 1 L of distilled water in a 1000 mL standard flask to create a stock solution. After that the stock solution was then used to extract the required volume of solutions that were then diluted with distilled water to produce different concentrations of adsorbate (20 mg/L to 100 mg/L).

2.4. Batch adsorption studies

Using the batch adsorption method adopted from [24–27], the adsorption studies for evaluating the adsorbent for removing methylene blue from aqueous solutions were conducted. In these experiments, different doses of adsorbent (0.2, 0.4, 0.6, 0.8, and 1 g) were added to 250 ml conical flasks that contained 100 ml of methylene blue aqueous solutions with an initial concentration of 20 mg/L. The mixtures were then stirred for 40 min at room temperature and pH 4.5 while being subjected to agitation.

The process was then repeated using an adsorbent dose of 1 g, pH of 4.5, and a contact time of 40 min but with varying initial concentration of methylene blue solution ranging from 20 mg/L to 100 mg/L.

At varying contact times of 20, 40, and 60 min, the adsorption method was repeated using an aqueous solution of methylene blue with an initial concentration of 20 mg/L, a pH value of 4.5, and an adsorbent dose of 1 g.

Room temperature was used for the experiment. To achieve proper adsorption, the liquids were then agitated with a cycling vibrator. Samples were withdrawn from the stirrer at various intervals. The adsorbents were then separated from the sample using a filter paper or a sieve. A UV-spectrophotometer was utilized to calculate the supernatant solution's absorbance. With

the help of the calibration plot, dye concentration in the supernatant was determined. The absorbance values are converted to the final dye concentration using the equation of line on the calibration plot. To ascertain the dye's absorption, a series of experiments were conducted at various times. Several different adsorbents were also employed in this experiment. The percentage removal of dye was calculated using Equation (1):

$$\text{dye removal (\%)} = \frac{(C_o - C_f)}{C_o} * 100\% \quad (1)$$

where;

C_o = initial dye concentration

C_f = final dye concentration

The methylene blue uptake was calculated by applying Equation (2);

$$q_e = \frac{V_s(C_o - C_e)}{m} \quad (2)$$

where;

q_e = amount of adsorbent adsorbed at equilibrium time

V_s = volume of solution

C_o = initial concentration

C_e = concentration at equilibrium time

2.4.1. Characterization of sample

The surface area, total pore surface, and average pore size of the prepared adsorbent were estimated using iodine number of the adsorbent.

2.5. Proximate analysis

2.5.1. Ash content

The weight of a crucible was taken. A sample of 1 g was obtained and weighed in the crucible. A muffle furnace was used to keep the sample at 500 °C for an hour and a half. It was then removed and allowed to cool for 30 min in a desiccator. The weight is measured once more as Equation (3) [24, 28].

$$\text{Ash content} = \frac{(W_A - W_o)}{(W_s - W_o)} * 100\% \quad (3)$$

where;

W_o = mass of empty crucible

W_s = mass of crucible plus sample

W_A = mass of crucible plus ash sample

2.5.2. Bulk density

According to the procedure described by [29], the product was placed into a graduated cylinder and crushed by tapping on the bench top until an anticipated volume (cm³) was occupied by mass (g). The cylinder was banged against the bench top until the sample volume stopped dropping. Bulk density was determined using Equation (4) after recording the mass and volume;

$$\text{Bulk density (g/mL)} = \frac{\text{weight of dry material (g)}}{\text{volume of carbon in cylinder}} \quad (4)$$

2.5.3. Moisture content

According to [30], the weight of a crucible with a lid was taken. In the crucible with the cover, 1 g of the sample was removed and weighed. It spent one and a half hours in a hot air oven set to 105 °C. It was removed and stored inside the desiccator. The weight was then determined using Equation (5).

$$\text{Moisture content} = \frac{(M_s - M_d)}{(M_s - M_o)} * 100\% \quad (5)$$

where;

M_o = Mass of crucible with lid

M_s = Mass of crucible with lid plus sample

M_d = Mass of crucible with lid plus dried sample

3. Results and Discussion

3.1. Characterization of carbonized activation of orange peel

According to the information in Table 1, the activated carbon adsorbent made from orange peel has a moderate bulk density of 0.987 g/mL, a moderate moisture content of 15%, and a comparatively low ash level of 3%. The substance has a pH of 7.25, which is somewhat basic, and an average pore size of just 46.8 angstroms. Additionally, the substance has a moderate total pore surface of 0.632 mL/g and a comparatively large surface area per gram of 52.5 m².

Table 1
Result of characterization of carbonized activation of orange peel

Parameters	Values
Ash content (%)	3
Bulk density (g/mL)	0.987
Moisture content (%)	15
Average pore size (A)	46.8
Surface area (m ² /g)	52.5
Total pore surface (mL/g)	0.632

According to one possible interpretation of the low ash level, the activated carbon adsorbent may be relatively pure and devoid of impurities, which would be advantageous for its usage as an adsorbent [31]. The material may have acceptable flowability and be ideal for use in situations where it needs to be handled or transported because of the moderate bulk density [32]. The material's mild moisture content would suggest that it is quite stable in humid conditions [33].

In applications where a large surface area is required for adsorption, including the removal of dyes or pollutants from water, the material's tiny average pore size and high surface area may make it ideal for utilization [34]. The material might also be ideal for use in applications that need a certain amount of porosity due to its modest total pore surface [35], including the filtering of gases.

Overall, the evidence points to a range of physical and chemical qualities in the activated carbon adsorbent made from orange peel that may make it appropriate for use in a number of adsorption applications [25]. To better comprehend the material's adsorption capabilities, additional study may be required, including methylene blue adsorption studies.

3.2. Batch adsorption studies

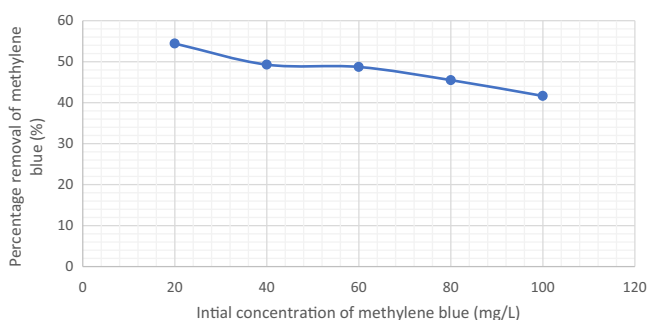
3.2.1. Effect of initial concentration of methylene blue

It can be observed from Table A1 and Figure 1 how the initial concentrations affect how much methylene blue is absorbed. The experiment utilized methylene blue concentrations of 20 mg/L, 40 mg/L, 60 mg/L, 80 mg/L, and 100 mg/L. According to the information in Figure 1, it seems that as the initial concentration of the dye increases, a lesser percentage of methylene blue dye is being adsorbed onto the prepared adsorbent. The active sites that are present on the surface of the carbon can be used to explain this phenomenon [36]. There are a lot of active sites on the activated carbon prepared that may interact with the dye ions at low concentrations of the adsorbate, in this instance the methylene blue dye. This indicates that there are a lot of active sites compared to the overall quantity of dye, which allows the dye ions to interact with the adsorbent and be drawn out of the solution. There are, however, fewer active sites on the activated carbon that are available to interact with the dye ions as the dye concentration rises. As a result, there are fewer active sites relative to the overall quantity of dye, which makes it harder to remove dye ions from solutions [37, 38]. This finding is in line with what has been written about adsorption in the literature.

Table A1
Experimental data for adsorption study on effect of initial concentration

Initial concentration (mg/L)	Concentration after adsorption (mg/L)	Removal (%)
20	9.114	54.431
40	20.286	49.284
60	30.769	48.718
80	43.597	45.504
100	58.355	41.645

Figure 1
Plot of percentage removal of methylene blue against initial concentration



3.2.2. Effect of adsorbent dosage

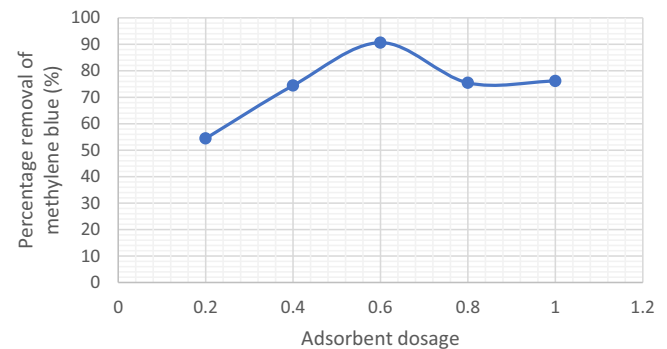
For varied dosages of 0.2 g, 0.4 g, 0.6 g, 0.8 g, and 1.0 g in 100 mL solution, the effects of the adsorbent dose on the percentage removal of dye at an initial concentration of 20 mg/L at room temperature with a contact period of 20 min were studied. According to Table A2 and Figure 2, the activated orange peels' ability to absorb methylene blue improves as the amount of adsorbent used rises from 0.1 g to 0.6 g. However, it was shown

that an increase of up to 1.0 g reduced the adsorption efficiency. Such patterning outcomes for other dyes have been reported from earlier research [39, 40]. The primary reason for the discrepancies in this finding can be attributed to the quantity of active sites. In fact, when the dosage of adsorbent was raised, the overall number of active sites grew. However, it was shown that an increase of up to 1.0 g reduced the adsorption efficiency. This may be because the active sites may bind to one another when adsorbent levels rise over the ideal level [36].

Table A2
Experimental data for adsorption study on effect of adsorbent dosage

Adsorbent dosage (g)	Concentration after adsorption (mg/L)	Removal (%)
0.2	9.114	54.431
0.4	5.114	74.431
0.6	1.872	90.638
0.8	4.907	75.466
1.0	4.769	76.155

Figure 2
Plot of percentage removal of methylene blue against adsorbent dosage



3.2.3. Effect of contact time

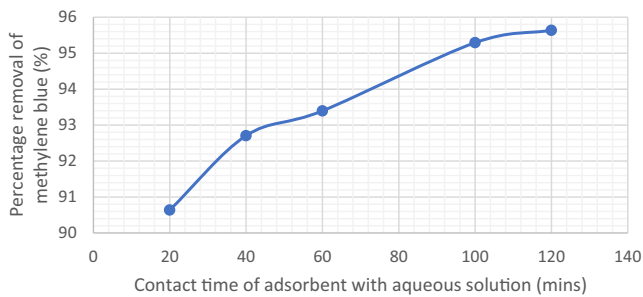
At room temperature, 0.2 g of adsorbent was added into 20 mg/L dye solution for several intervals of contact time (20 min, 40 min, 60 min, 100 min, and 120 min) as seen in Table A3 and Figure 3. The adsorbents were proven to remove more color with longer contact times. It was discovered that the dye removal process via adsorption utilizing activated orange peels was quick during the first few minutes of contact time before becoming slower as contact time increased [16, 41]. This can be as a result of the

Table A3
Experimental data for adsorption study on effect of contact time

Time (min)	Concentration after adsorption (mg/L)	Removal (%)
20	1.872	90.638
40	1.459	92.707
60	1.321	93.397
100	0.941	95.293
120	0.872	95.638

strong forces of attraction between the dye molecules and the adsorbent. When contact duration was extended, percentage removal initially rose as well, this is due to the fact that the active sites of the adsorbent are available and vacant initially [23], with time, these available sites become occupied, and the percentage removal begins to progressively approach a constant value as equilibrium was attained [42]. It can be observed that equilibrium occurs around 120 mg/L.

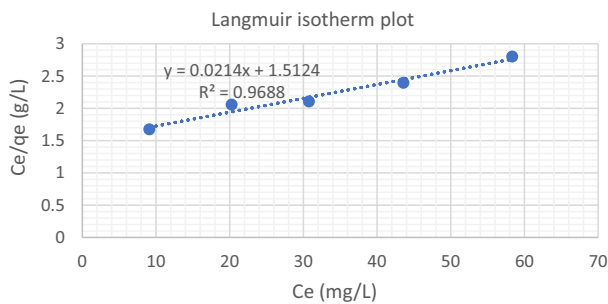
Figure 3
Plot of percentage removal of methylene blue against contact time with adsorbent



3.2.4. Adsorption isotherm study

The adsorption isotherm of methylene blue on the prepared adsorbent from orange peel was analyzed with Langmuir and Freundlich isotherm model as shown in Figures 4 and 5.

Figure 4
Langmuir isotherm plot

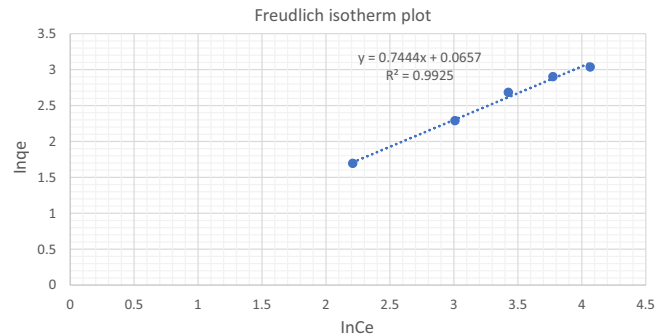


The adsorption capacity of the adsorbent material was determined by fitting the experimental data to the Langmuir isotherm model as seen in Table A4. The maximum adsorption capacity (q_{max}) was found to be 46.723 mg/g, which was higher than the values reported for other adsorbents as seen in Table 2 such as corn stalk (21.27 mg/g) and wheat straw (41.84 mg/g) [43].

Table A4
Isotherm data for Langmuir and Freundlich

C _e (mg/L)	q _e (mg/g)	C _e /q _e (g/L)	ln(C _e)	ln(q _e)
9.113793103	5.4431034	1.674374406	2.209788992	1.694349385
20.2862069	9.8568966	2.058072416	3.009941192	2.288171368
30.76896552	14.615517	2.105225905	3.426506569	2.682083789
43.59655172	18.201724	2.395188027	3.774978058	2.901516322
58.35517241	20.822414	2.802517181	4.066547999	3.036029993

Figure 5
Freundlich isotherm plot



This indicates that orange peel adsorbent had a superior performance in the removal of methylene blue from waste water.

The equilibrium methylene blue concentration (C_e, mg/L) is shown in Table A4, and Figures 4 and 5 matched to the adsorption model suggested by Langmuir and Freundlich.

Table 2
Compares adsorption capacity of orange peel to other adsorbents reported in literature

S/No	Absorbents	Maximum adsorption capacity (mg/g)	References
1	Sugarcane leaves straw	8.0	Choi et al., [44]
	Sugarcane bagasse	6.6	
2	Wheat straw	41.84	Dhir & Kumar, [43]
3	Sunflower stalk	39	Hashem et al., [45]
4	Wool fiber	12.0	Radetić et al., [46]
5	Corn stalk	21.37	Zheng et al., [47]
6	Hazel nutshell	28.18	Pehlivan et al., [48]
7	Rice husk	19.66	Vlaev et al., [49]

Table 3 lists the values of the constants for the two isotherms, Freundlich and Langmuir. It is obvious that the Freundlich model fits the adsorption isotherm model better. This demonstrates that the dye is absorbed following heterogeneous coverage with overlapping layers.

Table 3
Values of constant for Langmuir and Freundlich isotherm

Langmuir isotherm	Freundlich isotherm
R ² = 0.9688	R ² = 0.9925
K _L = 0.141	K _f = 1.068
q _{max} = 46.723	n = 1.343

4. Conclusion

From the methylene blue adsorption utilizing an adsorbent made from orange peel, the following conclusions may be drawn.

1. The activated carbon was found to have a moisture content of 15%, which shows it can be stable in humid conditions. The adsorbent ash content was gotten to be 3%.
2. The activated carbon adsorbent made from orange peel was found to have a bulk density of 0.987 g/mL, an average pore size of 46.8 angstroms, a total pore surface of 0.632 mL/g, and a surface area of 52.5 m²/g.
3. Methylene blue adsorption rate increases over time until it reaches equilibrium, at which no discernible increase in adsorption. The measured equilibrium time was 120 min. An increase in the dosage of the adsorbent led to a greater removal of methylene blue. Methylene blue adsorption peaked at 0.6 g of adsorbent dose. However, the percentage of methylene blue elimination decreased for adsorbent dosages greater than 0.6 g. It was shown that the initial concentration of methylene blue was inversely proportional to the rate at which methylene blue adsorbs to the produced adsorbent.
4. The concentration of methylene blue showed it was a better fit for Freundlich isotherm model than the Langmuir isotherm model.

This material has the potential applications in the treatment of wastewater containing dye pollutants, which are harmful to the environment and human health. The adsorption performance of this material can be further improved by modifying its surface properties, such as introducing functional groups or coating with metal nanoparticles. The effect of other parameters, such as temperature, salinity, and coexisting substances, on the adsorption process can also be investigated in future studies.

Ethical Statement

This study does not contain any studies with human or animal subjects performed by any of the authors.

Conflicts of Interest

The authors declare that they have no conflicts of interest to this work.

Data Availability Statement

Data available on request from the corresponding author upon reasonable request.

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