Decreasing Methylene Blue Using Expanded Perlite

Mardiana Rimba Utami, Kapti Riyani, Tien Setyaningtyas
Department of Chemistry, Faculty of Sciences and Mathematics, Jenderal Soedirman University
mardianarimba@gmail.com

***Abstract*-The industrial sector and household is a significant contributor to water pollution. Pollutants such as acids, dyes, heavy metals, and other toxic chemicals can have devastating effects on the environment, even at low concentrations. Methylene blue in water can undergo photodegradation with the assistance of sunlight, but this reaction occurs slowly and is not in line with its accumulation rate in the water. This can lead to environmental damage due to the presence of methylene blue, which is difficult to degrade and impairs water quality. To mitigate these risks, adsorption used to wastewater treatment. This study investigates the effectiveness of perlite as an adsorbent for decreasing methylene blue from water, examining the effects of pH, contact time, and initial concentration on adsorption kinetics and isotherms. The optimal pH for methylene blue adsorption is 9, with a contact time of 180 minutes. The maximum adsorption capacity of perlite is 3.87 mg/g, with the adsorption process following pseudo-second-order kinetics and the Langmuir isotherm model, indicating a chemical and homogeneous single-layer adsorption mechanism.**

Keywords: Methylene Blue; Expanded Perlite; Adsorption

I. INTRODUCTION

Water is a vital component of life, and its significance cannot be overstated. Water is the lifeblood of our daily routines – from quenching our thirst and keeping ourselves clean, to cooking our meals and building our homes. Smart water management is the key to guaranteeing the water we use is not only safe but also sustainable, with waste that's harmless to the environment. Fortunately, the Earth's water cycle continuously recycles this precious resource. As technology and science advance, it's imperative that innovations prioritize environmental sustainability. The potential consequences of untreated wastewater posing a threat to the environment are dire. Aquatic ecosystems that receive polluted water are not only compromised but also pose a risk to other living organisms. To mitigate this risk, humans are driven to develop simple, cost-effective solutions to minimize the adverse effects of hazardous waste and promote a safe and sustainable future for our planet.

The industrial sector and household is a significant contributor to water pollution. Even in tiny amounts, pollutants like acids, dyes, heavy metals, and other toxic chemicals can unleash catastrophic damage on the environment. Industrial waste contributes 33.33% of water pollution, while household waste contributes 47.62%, and urban waste accounts for 19.04% [5]. Untreated dyes can be particularly problematic, posing a threat to microorganisms, aquatic ecosystems, and drinking water sources. There are various dyes used as textile colorants with different chemical properties, such as methylene blue, rhodamine B, methyl violet, sunset yellow, and allura red. Methylene blue, in particular, is a cationic dye belonging to the heterocyclic aromatic compound family. It is frequently used in dyeing silk, paint, leather, paper, and cosmetics [8]. Furthermore, research by Pasa et al. highlights the dye's additional applications in medicine, such as a staining agent in histology and as an antiseptic and antifungal treatment. Methylene blue in water can undergo photodegradation with the assistance of sunlight, but this reaction occurs slowly and is not in line with its accumulation rate in the water. This can lead to environmental damage due to the presence of methylene blue, which is difficult to degrade and impairs water quality. Furthermore, exposure to this dye, particularly through contaminated drinking water, can lead to various health issues, including cyanosis, tissue necrosis, nausea, vomiting, jaundice, and hemolysis [11].

To mitigate these risks, it is essential to develop and implement effective treatment methods that can remove hazardous substances from industrial wastewater before it is released into the environment. Techniques including adsorption [4], membrane technology, biofilm, electrocoagulation, oxidation, microbial fuel cells [3], and filtration [2] are all viable options. Among these, adsorption stands out as one of the most commonly employed methods for treating wastewater.This method is considered low-cost, has a simple process, and high effectiveness. Researchers are continually seeking adsorbents that combine high performance, eco-friendly, and economically viable. Perlite stands out for its exceptional adsorption capacity, low cost, nontoxic, and practical application [7] [4]. Perlite is essentially a volcanic aluminosilicate glass, distinguished by its crystal structure which traps water – making up about 2% to 5% of its composition. When perlite is heated to high temperature (800-1200℃), the water turns to vapor creating a porous, lightweight that called expanded perlite. The silanol groups on perlite's surface enable it to bind with various contaminants, including heavy metals and organic pollutants, while its porous nature enhances its adsorption capabilities, making it effective for multiple uses [4]. Perlite's ability to adsorb substances can be traced back to the silanol groups present on its surface – these groups are formed by silicon atoms [12] [9]. The perlite surface, containing alumina hydrous oxide, also plays a role in adsorption process [12]. The diffusion mechanism occuring in perlite adsorption can vary depending on the adsorbent structure and sometimes multiple mechanism can occur simultaneously. The adsorption process involves several key mechanisms, including the migration of ions from solution to the adsorbent surface – a process known as ion adsorption. Additionally, surface dissociation allows ions to break free from the adsorbent surface, while isomorphic substitution enables ions to swap places within the crystal structure of perlite. With a surface area spanning from 1.22 to 52.9 m^2/g, perlite demonstrates impressive adsorption potential. When perlite is expanded, its surface area and pore volume jump – a massive 70-fold increase compared to its unexpanded counterpart [13]. This remarkable capacity for adsorption has been borne out in previous research, where expanded perlite has been successfully used to adsorb Cu(II) by Pamasengi et al., iron(III) by Kalabegashvili, and the cationic dye rhodamine B by Damiyine et al. The present study seeks to explore perlite's ability to reduce methylene blue levels via adsorption, testing key parameters such as pH, contact time, and initial ion concentration to gauge the adsorbent's effectiveness.

II. EXPERIMENTAL METHOD

A. *Preparation of perlite*

To prepare the perlite, a multi-step process was employed. Initially, 25 grams of perlite with a 120-mesh particle size was obtained. This was followed by a 1-hour immersion in 5% nitric acid. After the acid treatment, the perlite was subjected to oven drying at 80°C for 1 hour. The final step involved calcining the perlite at 500°C for a duration of 2 hours [14].

B. *Test Method*

To kick off the process, the initial step was to pinpoint the peak wavelength for methylene blue. This involved taking a 2 ppm aliquot from a 10 ppm methylene blue standard solution and then running it through a UV-Vis spectrophotometer, scanning the spectrum from 400 to 800 nm to capture the point of maximum absorbance. This critical wavelength value would then serve as the foundation for all subsequent experiments.

To explore how pH influences the adsorption process, we combined 250 mL of a 10 ppm methylene blue solution – adjusted to different pH levels (specifically 3, 5, 7, 9, and 11) – with 0.25 grams of our specially prepared perlite. These mixtures were then agitated for 3 hours using a magnetic stirrer, followed by a 10-minute centrifugation at 1500 rpm to separate the solids. The resulting supernatant was then analyzed for its absorbance at the previously determined maximum wavelength, allowing us to gauge the adsorption efficiency under these varying pH conditions.

After pinpointing the optimal pH, we turned our attention to the impact of contact time on the adsorption process. This involved introducing 0.25 grams of our prepared perlite into 250 mL of a 10 ppm methylene blue solution, which was pre-adjusted to the maximum pH we had previously identified. The mixture was then subjected to magnetic stirring, and samples were drawn at specific intervals: 10, 30, 60, 90, 120, 180, and 240 minutes under visible light conditions. Following centrifugation to separate the solids, we measured the absorbance of the resulting supernatant at the maximum wavelength, enabling us to assess the adsorption performance across these varying contact times.

To gauge the impact of initial ion concentration, we crafted a series of 250 mL methylene blue solutions, spanning a concentration range of 10 to 80 ppm (specifically 10, 20, 30, 40, 50, 60, 70, and 80 ppm), with each solution containing 0.25 grams of prepared perlite. Then fine-tuned each solution to the optimal pH and subjected them to magnetic stirring for the maximum contact time, all under visible light conditions. Post-centrifugation, we measured the absorbance of each solution at the maximum wavelength, allowing us to assess the adsorption capacity across this range of initial concentrations.

III. RESULT AND DISCUSSION

Perlite is primarily composed of silica (SiO_2) at 72-76% and alumina (Al_2O_3) at 13.5-16.9%, along with potassium oxide (3-6%), sodium oxide (3-5%), and smaller amounts of other oxides. The surface of perlite features silanol groups, including silanol (–SiOH), silanediol (–$Si(OH)_2$), and silanetriol (–$Si(OH)_3$), as well as hydroxyl groups from aluminum hydroxide Al(OH) or $Al(OH)_3$). These functional groups enhance perlite's ability to adsorb metal ions from aqueous solutions, underscoring its adsorptive properties [15].

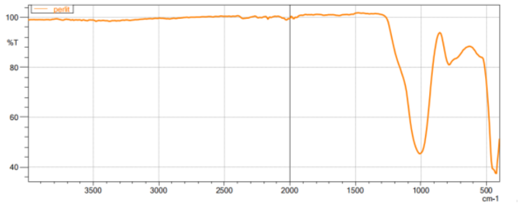

Fig. 1. FTIR Spectra of Expanded Perlite

To gain deeper insights, we turned to FTIR for a qualitative analysis. The goal here was to pinpoint the interconnected bonds within the prepared perlite specifically, the expanded perlite adsorbent by interpreting the distinct bands that emerge on the FTIR diagram. By FTIR spectroscopy, we aimed to identify the key functional groups present in the expanded perlite. A visual representation of the infrared spectrum for this material can be found in Figure 1. Our FTIR analysis revealed some telling adsorption bands: the band at 424.34 cm^{-1} corresponds to the bending vibration of the siloxane group (Si-O-Si); the 786.96 cm^{-1} band is indicative of aluminosilicate characteristics; and the 1010.7 cm^{-1} band is tied to Si-O-M stretching vibrations [16].

A. Maximum pH

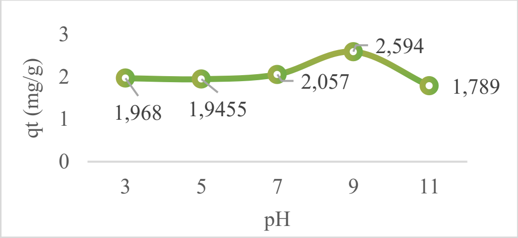

Fig. 2. Most Effective pH for Decreasing Methylene Blue

As illustrated in Figure 2, the peak adsorption capacity (qt) for methylene blue was achieved at a pH of 9, hitting a high of 2.59 mg/g. When methylene blue is in solution, it breaks down into cationic species. At lower pH levels, the expanded perlite surface takes on a positive charge due to the prevalence of H+ ions [17], triggering electrostatic repulsion that actually hinders the adsorption of these cationic methylene blue species. However, as the pH climbs, the surface of the expanded perlite picks up a negative charge, which in turn facilitates the adsorption of those methylene blue cations [18]. This process is further amplified by the ionization of silanol groups on the expanded perlite surface at higher pH levels, resulting in the release of H^+ ions into the solution and boosting adsorption [19]. The negatively charged surface of the expanded perlite enables electrostatic interactions with the cationic methylene blue, and the strength of this attraction intensifies as the negative charge on the adsorbent surface grows [16]. That being said, at extremely alkaline conditions, the dye starts to decompose, rendering further pH increases ineffective. The high silica content in expanded perlite is a key player in its adsorption capacity. As Acemioglu (2005) noted, silanol groups formed by silicon atoms on the perlite surface are the primary agents responsible for adsorbing methylene blue. The reactive oxygen atoms (O^-) in these silanol and siloxane groups can effectively bind to the positively charged nitrogen atoms (N^+) in methylene blue, driving the adsorption process forward. A simplified reaction equation for this mechanism is provided below [9].

$$–MO^- + dyes^+ \leftrightarrow M–O–dyes \qquad (1)$$

B. Maximum Contact Time

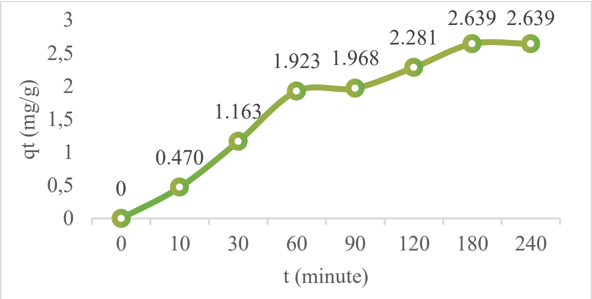

Fig. 3. Most Effective Contact Time for Decreasing Methylene Blue

As depicted in Figure 3, the optimal contact time for expanded perlite adsorption is 180 minutes, at which point it reaches an adsorption capacity of 2.64 mg/g. The expanded perlite's ability to reduce methylene blue becomes more pronounced with extended contact times, simply because more and more active sites on the expanded perlite surface get occupied by methylene blue molecules. As time goes on, the reaction starts to plateau due to the dwindling number of available active sites on the expanded perlite that can still be filled by methylene blue cations. The data collected from this process is then utilized to pinpoint the most fitting kinetic model for describing the adsorption process.

C. Adsorption Kinetics

To assess the adsorption kinetics, we matched our data against the most appropriate kinetic model, judging by the coefficient of determination (R^2) or linearity. We zeroed in on two key models: the pseudo-first order model (Equation 2) and the pseudo-second order model (Equation 3) [10]. To gauge the pseudo-first order model, we created a plot of log (qe - qt) versus time (t), whereas for the pseudo-second order model, we plotted t/qt against time (t). The resulting kinetic model plots for the adsorption of methylene blue onto expanded perlite are visually represented in Figure 4.

$$\log(\mathrm{qe} - \mathrm{qt}) = \log \mathrm{qe} - \frac{\mathrm{k}_1}{2{,}303}\mathrm{t} \qquad (2)$$

Within this equation, qt and qe signify the quantity of methylene blue adsorbed at a given time t and at equilibrium, respectively. Meanwhile, k symbolizes the rate constant that governs the adsorption process.

$$\frac{t}{qt} = \frac{1}{q_e^2 k_2} + \frac{1}{qe}t \qquad (3)$$

The adsorption kinetic were conducted at pH 9 with varying concentrations of methylene blue ranging from 10-80 ppm.

TABLE I. ADSORPTION KINETICS DATA

				Pseudo first order	Pseudo second order
time (t)	**qe (mg/g)**	**qt (mg/g)**	**qe-qt**	**log (qe-qt)**	**t/qt**
10	2,639	0,47	2,169	0,336	21,277
30		1,163	1,476	0,169	25,795
60		1,923	0,716	-0,145	31,201
90		1,968	0,671	-0,173	45,732
120		2,281	0,358	-0,446	52,609
180		2,639	0	0	68,208
240		2,639	0	0	90,944

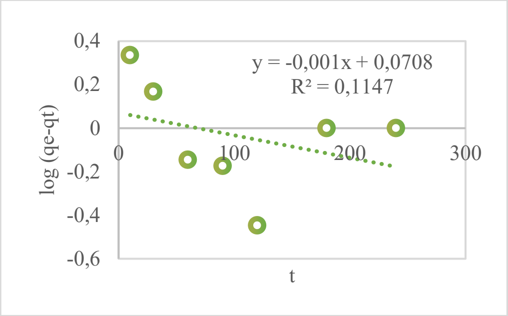

Fig. 4. Pseudo-first Order Kinetic Model of Decreasing Methylene Blue

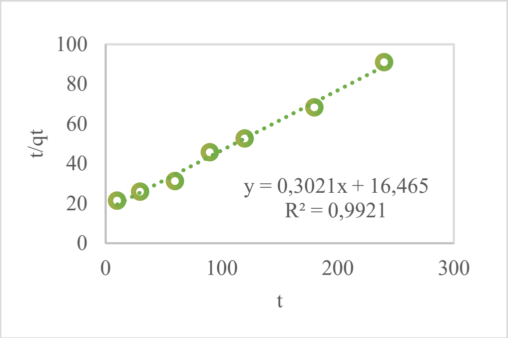

Fig. 5. Pseudo-second Order Kinetic Model of Decreasing Methylene Blue

Figures 4 and 5 visually represent the kinetic models for methylene blue adsorption. For years, linear regression and least squares methods have been go-to techniques for pinpointing the most fitting model and estimating its parameters [1]. When we look at the R^2 values, the pseudo-first order model clocks in at 0.1147, whereas the pseudo-second order model boasts an R^2 value of 0.9921. This stellar R^2 value for the pseudo-second order model implies it's the perfect match for describing the adsorption kinetics, suggesting the adsorption rate is proportional to the square of the remaining adsorption capacity. What this means is the concentration drops rapidly at the outset of adsorption, followed by a more gradual slowdown in the adsorption rate until equilibrium is finally reached. We leverage this kinetic model to calculate key metrics like the adsorption rate constant (k) and the maximum adsorption capacity (q). A higher rate constant is indicative of faster adsorption kinetics [8]. For a concise overview, all the kinetic results are neatly summarized in Table II.

TABLE II. ADSORPTION KINETICS RESULT

Pseudo-first Order	k	0,002 g/mg.min
	q	1,117 mg/g
	R^2	0,1147
Pseudo-second Order	k	0,665 g/mg.min
	q	3,310 mg/g
	R^2	0,9921

D. *Adsorption Isotherm*

We delved into how the initial methylene blue concentration impacts the adsorption capacity of expanded perlite. Our findings revealed that at a concentration of 10 ppm, we achieved the highest adsorption percentage – a notable 41.199%. In stark contrast, the highest concentration we tested, 80 ppm, resulted in the lowest adsorption percentage, coming in at just 7.382%. This trend implies that the adsorption percentage goes up as the methylene blue concentration goes down. The data we gathered from this investigation is then utilized to pinpoint the most fitting isotherm model for describing the adsorption process.

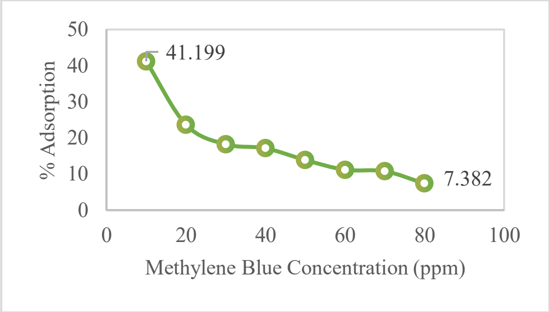

Fig. 6. Effect of Initial Ion Concentration of Decreasing Methylene Blue

Adsorption isotherms essentially map out the equilibrium relationship between the amount of adsorbate clinging to the adsorbent and the pressure or concentration of that adsorbate in the surrounding environment, all at a constant temperature. The Langmuir isotherm operates on the premise of a uniform surface where adsorption occurs in a single layer. In contrast, the Freundlich isotherm paints a picture of adsorption on surfaces that are heterogeneous, featuring multiple types of adsorption sites – this allows for the formation of multiple layers and reversible adsorption. According to the Freundlich model, the adsorbent's surface is made up of diverse adsorption zones, each with its own unique characteristics [20]. To determine the Langmuir isotherm, we plotted Ce/Qe against Ce, whereas for the Freundlich isotherm, we went with a plot of log Qe versus log Ce. These adsorption isotherm experiments were carried out at a pH of 9, with methylene blue concentrations spanning from 10 to 80 ppm.

TABLE III. ADSORPTION ISOTHERM DATA

Concetration (ppm)	**ce**	**qe**	**ce/qe**	**log ce**	**log qe**
10	3,638	2,549	1,427	0,561	0,406
20	8,871	2,75	3,226	0,945	0,439
30	13,343	2,974	4,487	1,125	0,473
40	19,582	4,047	4,839	1,292	0,607
50	25,396	4,092	6,206	1,405	0,612
60	29,844	3,758	7,941	1,475	0,575
70	35,37	4,27	8,283	1,547	0,630
80	39,841	3,175	12,548	1,600	0,502

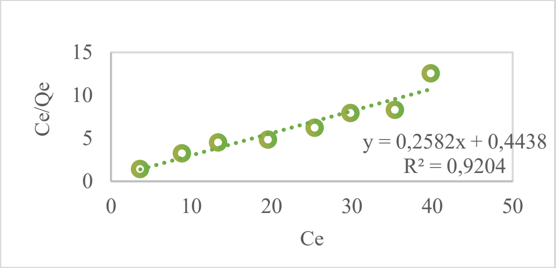

Fig. 7. Langmuir Isotherm Model of Decreasing Methylene Blue

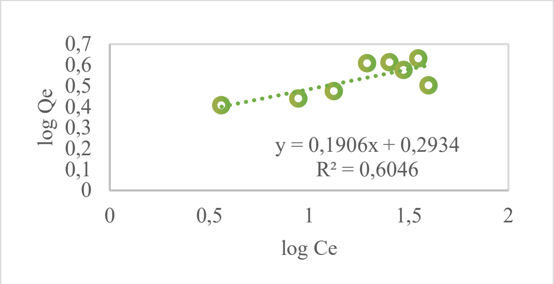

Fig. 8. Freundlich Isotherm Model of Decreasing Methylene Blue

Figures 7 and 8 lay out the adsorption isotherm models for expanded perlite. Here, the Langmuir model spits out an R^2 value of 0.9204, while the Freundlich model gives us an R^2 value of 0.6046. The Langmuir model's higher R^2 value indicates it's the more suitable model for our adsorption data, pointing towards a chemical adsorption process characterized by a uniform distribution of active sites on the expanded perlite surface [21]. The core assumption of the Langmuir model is that each active site can only latch onto one molecule, and when all these sites are filled, the adsorption process hits equilibrium due to the formation of a single molecular layer [22] [23]. Digging into the numbers, the maximum adsorption capacity derived from the Langmuir isotherm model stands at 3.87 mg/g. For a clear snapshot of these adsorption isotherm results, check out Table IV.

TABLE IV. ADSORPTION ISOTHERM RESULT

Isotherm	Data	Value
Langmuir y = 0,2582x + 0,4438 R^2 = 0,9204 $\frac{C_e}{Q_e} = \frac{C_e}{Q_m} + \frac{1}{Qm.K_L}$ [24]	Q_{max} (mg/g) $\frac{1}{qmaks} = b$	3,87
	K_L (L/mg) $\frac{1}{qmaks\,K_L} = a$	0,582
Freundlich y = 0,1906x + 0,2934 R^2 = 0,6046 $\log Q_e = \log K_f + \frac{1}{N}\log C_e$ [25]	N (mg/g) $\frac{1}{N} = b$	5,247
	K_F (L/mg) Log K_F = a	1,965

IV. CONCLUSION

Perlite stands out as one of the adsorbents that really packs a punch when it comes to reducing methylene blue levels. In this particular study, we zeroed in on expanded perlite to gauge its effectiveness. Our findings pinpointed the most effective for decreasing methylene blue is pH 9, coupled with a contact time of 180 minutes. We achieved a maximum adsorption capacity of 3.87 mg/g by using 0.25 g of expanded perlite. This process followed pseudo-second order kinetics and aligned neatly with the Langmuir isotherm model. Looking ahead, there's a clear need for more research to flesh out how perlite performs against a broader range of pollutants.

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