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Adsorptive Removal of Methylene Blue Dye Using Low-Cost Adsorbent Prepared From *Azolla Pinnata*

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Abstract
Methylene blue dye is generally used for colouring of the textile products. Acute exposure of the Methylene blue dye containing water has some harmful affects on aquatic as well as terrestrial life. Due to the non- biodegrade nature, there is requirement for the treatment of this dye containing water. Among the various treatment methods, Adsorption method using the biomass can be appropriate and cost effective. Hence, This study was performed in order to investigate the low cost absorbent derived from the azolla pinnata biomass for removing the methylene blue dye from the aqueous solution. The influence of the various adsorption parameters such as initial dye concentration, doses, pH and contact time were studied. The results showed that the adsorption percentage of the methylene blue was reduced with the increment of the initial dye concentration. The dye adsorbed percentage was found to be higher than 90% in lower concentration (50 mg/l to 300 mg/l). Langmuir isotherm best fits the experimental data which suggests the monolayer adsorption of the dye by biomass. The maximum Langmuir adsorption of the dye was found to be 344.827 mg/g. In addition to this, the rate of the adsorption was best fitted with the Pseudo-second order kinetic model. The results show that the biomass of the azolla pinnata can be used as the alternative absorbent for the methylene blue dye removal

Keywords
Azolla Pinnata, Biomass, Methylene blue Dye, Removal Efficiency

1. Introduction

Abundant discharge of the organic or in organic pollutant because of the industrialization and urbanization has shown great environmental impact.The dye consisting effluents are generated from various industries such as leather paper, textile rubber etc for colouring their products. The release of the dye into the environment is a concern for the toxicological reasons as the dye damage the quality of the water streams, affects on the micro organism and carcinogenic to living organism [1].

Among the several dyes used for the industrial purpose, Methylene blue dye is generally used for colouring of the textile products. Acute exposure of the Methylene blue dye has some harmful affects such as increment of heart rate, shock, cyanosis, jundic cyanosis, jaundice, Heinz body formation and eye burns. It also cause the micro-toxicty to the aquatic life such as fishes[2]. The dyes are of the heterogeneous type organic compounds that are hardly biodegradable in nature. Hence, due to these reasons, there is requirement for the treatment of the dye containing solutions. Among the various technologies such as foam flotation, precipitation, coagulation, filtration, membrane technology, chemical oxidation and adsorption method is very appropriate and cost effective [3]. The utilization of the low cost bio-absorbent, biomass generated from agricultural waste, can be used for the waste water treatment due to abundant presence of the amino, hydroxyl and carboxyl group. In addition to this, biomass is composed of cellulose, hemi-cellulose, lignin etc. [4]. Hence, they are used as absorbent for the removal of the dye. There have been various research conducted for the removal of the cationic dye using the biomass of Eucalyptus bark powder[3], rice waste[5],Spent Tea Leaves[6], corn[7], barley[8], fungi and yeast[9], peanut waste[10], timber waste[11], banana peel[12] etc. In addition to this, agricultural biomass are readily available in locally, cost effective and efficient in the removal of the azo dyes and also make the adsorption method more effective and cost efficient. The biomass generated from the synthesis of phyto-remediation plant such as lamina minor[13], Azolla filiculoides[14], Azolla pinnata[15],Salvinia molesta Mitchel[16],Eichhornia crassipes[17] can be used for the efficient adsorption of the cationic dye. There are several researches have been found using biomass of Azolla filiculoides for the removal of the Acid Blue 225[18], Acid black 1[19], Acid Green 3[19],reactive black 5[20] and acid dyes[21, 18, 22]. The adsorption of various kind of the dye in the research papers only focused on using the biomass of Azolla filiculoides. Beside this, very limited research articles focus on the adsorption methylene blue dye using biomass of the azolla pinnata. Not only this, The adsorption parameters, kinetics and adsorption mechanism under the various process conditions are not focused using biomass of an azolla pinnata[23, 15]. Hence, this study focused on the adsorption of the methylene blue dye using biomass of azolla pinnata available locally.

2. Materials and Methods

2.1 Absorbent and Stock solution Preparation

Azolla pinnata was collected from the nearest agriculture farm located in Balaju, Kathmandu district in the month of late July 2022 as they cultivated it for feeding to the chicken. The collected samples was washed using the deionized water till the impurities are clearly removed. The washed azolla pinnata was then dried in the oven using temperature of 75-80 degree Celsius[24]. Then after, the dried sample was grounded to the fine powder using the mortar and pestle. Finally sample are stored in the plastic sample bottle for the adsorption experiment. 1 gram of the Methylene blue dye was dissolved in the distilled water in order to prepare the stock solution of 1,000 mg/l methylene blue dye. The working dye solution of the required concentration were made by diluting the stock solution.

2.2 Characterization

The FT-IR characterization was done using the perkin elmer Spectrum IR Spectrometer in order to determine the functional group associated with biomass. The concentration of the residual dye in the aqueous medium was determined by using the UV/visible (CECIL-CE-100) spectrometer. The wavelength $(\lambda_m = 664nm)$ was taken in which the absorbance value of the methylene blue dye was maximum. This wavelength and stock solution of 1-10 mg/l were used for calibration of the dye solution and then quantification of the dye was performed.

2.3 Adsorption experiments

The adsorption experiments were performed by varying the absorbent doses, initial dye concentration, pH and time. The mixture was shaken in laboratory temperature condition 30 ± 2 degree Celsius. The dye of 25 ml having concentration of 50, 100, 200, 300, 500 and 600mg/l were filled in the Erlenmeyer flask. The dose of the absorbent was taken as 25 mg (1gm/l) in the adsorption experiment. Then shaking was done by using the 200 RPM in the rotatory shaker for the time of 180 minutes. The solutions with absorbent were filtered using Wattman filter paper in order to separate the absorbent form the solution. Then after, the quantification of the residual dye solution was done by UV-Vis spectroscopy. Similarly, the effect of the absorbent doses was determined by varying the doses from 10 mg to 60 mg in the dye volume of 25ml and 300 mg/l concentration.

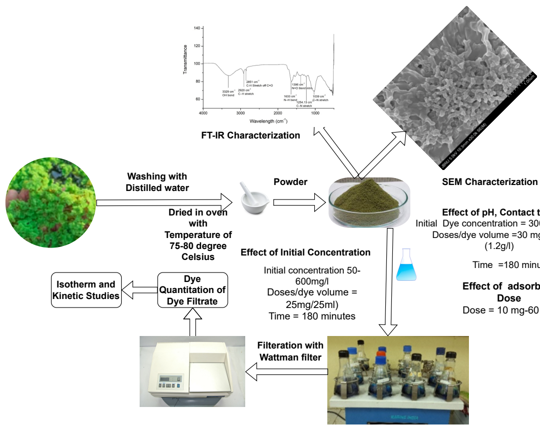

Figure 1: Graphical Representation of Experimental Methods for Methylene blue Dye removal

2.3.1 Kinetics and Adsorption Isotherm Experiments

For the investigation of the adsorption and transient behaviour of the adsorption process, the adsorption kinematic needs to be determined and analyzed using the pseudo-second order and pseudo first order models. The linear equation of the pseudo first order model is expressed as in equation 1.

$$log(q_e - q_t) = log(q_e) - \frac{k_1}{2.303}t \quad (1)$$

The linear order model of the pseudo second order is also expressed as in the equation 2.

$$\frac{t}{q_t} = \frac{1}{(K_2)(q_e)^2} + \frac{t}{q_e} \quad (2)$$

where q_e (mg/g) and q_t (mg/g) are the amount of the dye adsorbed onto the biomass of the azolla pinnata at equilibrium state and time. k_1 and k_2 are the equilibrium rate constant of pseudo first order and second order adsorption respectively. The equation for determined the amount of the dye adsorbed with the respect to time and equilibrium are expressed in equations 3 and 4 respectively

$$q_t = \frac{C_0 - C_t}{m} \times V \quad (3)$$

$$q_e = \frac{C_0 - C_e}{m} \times V \quad (4)$$

C_0 and C_e are the initial and equilibrium concentration of the dye solution. m and V are the mass of the absorbent and Volume of the dye solution respectively. The dye removal efficiency is expressed as

$$Removal percentage = \frac{C_0 - C_e}{C_0} \times 100 \quad (5)$$

The adsorption Isotherm are expressed in two models such as Langmuir and Freundlich. These models are selected in order to explicate the dye interaction with biomass and also evaluate the adsorbent capacity. The linear equation of the freundlich isotherm and Langmuir isotherm are expressed as in equation 6 and equation 7

$$log(q_e) = logK_f + \frac{1}{n}logC_e \quad (6)$$

$$\frac{C_e}{q_e} = \frac{1}{k_a q_m} + \frac{1}{q_m} \quad (7)$$

Where K_f and K_a are the isotherm constant for freundlich and Langmuir isotherm.

3. Results and Discussion

3.1 FT-IR analysis

The FT-IR spectra of the AP biomass (figure 2) shows several peaks which representing the consisting of the of various functional groups. The broad peak at $3329 cm^{-1}$ which indicate the presence of the OH stretch with alcohol and phenol. The peak at $2920 cm^{-1}$ and $2851 cm^{-1}$ represent the C-H stretch alkenes and C=O aldehydes. The sharp peaks at the $1633 cm^{-1}$ and $1039 cm^{-1}$ indicate the presence of the N-H bond with amine and aliphatic amines.

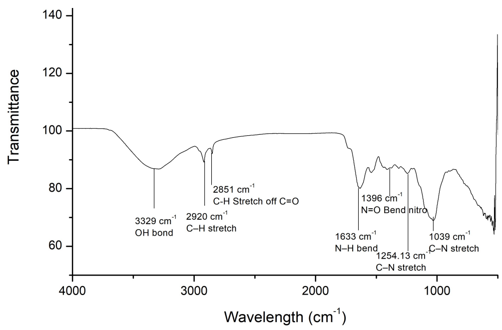

Figure 2: FTIR results of Azolla pinnata Biomass

These functional groups interact with the methylene blue dye with the electrostatic interaction, H- bond and vanderwall force. This leads to the rapid adsorption of the cationic dye into it's pore and surfaces[25] .

3.2 SEM analysis of Biomass

The SEM characterization of Azolla was done in order investigate the morphology of the biomass. The SEM image of the biomass (figure 3) shows that it consists of the deep pores and voids. This active sites can enhance the adsorption of the dye molecules[26].

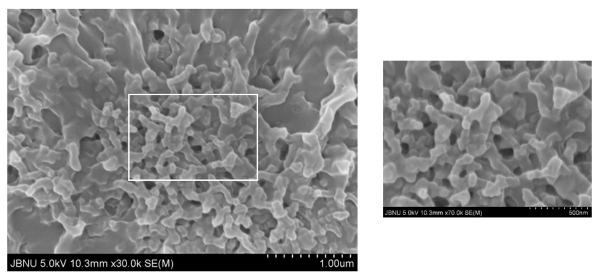

Figure 3: SEM image of Azolla pinnata Biomass

3.3 Effect of initial dye concentration on dye adsorption

In order to investigate the effect of the initial dye concentration, the experiment was performed by varying the initial dye concentration from 50 mg/l to 600 mg/l by taking the absorbent doses as 1g/l. The figure 4 shows that the adsorption of the dye increased with the increment of the initial dye concentration and later remains constant with the respect to higher dye concentration. However, the removal efficiency decreases with the from 97% – 55% when initial concentration of the dye increased from 50 mg/l to 600 mg/l

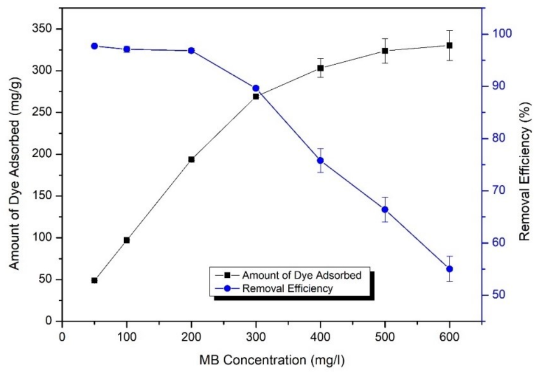

Figure 4: Effect of initial dye concentration on the amount of dye adsorbed and removal efficiency

3.4 Effect of absorbent doses on dye adsorption

The absorbent dosage is a crucial parameters due to fact that this factor determines the absorbent capacity for a given concentration. The AP biomass were varied from 10 mg to 60 mg in a 25 ml of 300 mg/l dye solution.The graph (figure 5) between absorbent doses, dye adsorbed and removal efficiency shows that as the absorbent dosage increased, dye removal efficiency also escalated. However, the amount of the dye adsorbed per unit mass of AP biomass is decreased considerably. This was due to the unsaturated of the adsorption sites during adsorption reactions.

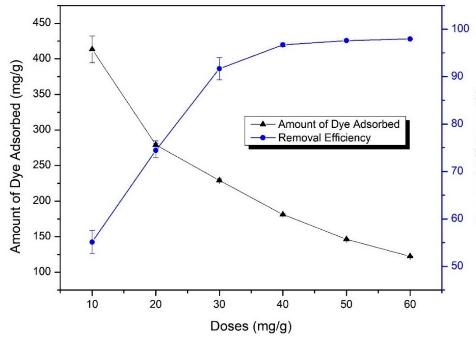

Figure 5: Effect of biomass doses on the amount of dye adsorbed and removal efficiency

3.5 Effect of contact time on dye adsorption

The adsorption of the methylene blue dye using the biomass of the azolla pinnata was studied as the function of the time for determining equilibrium condition. For this, 1.2g/l (30 mg in 25 ml) of the absorbent, 300 mg/l dye concentration were taken and experiment was performed with different contact time 30 minutes to 180 minutes.

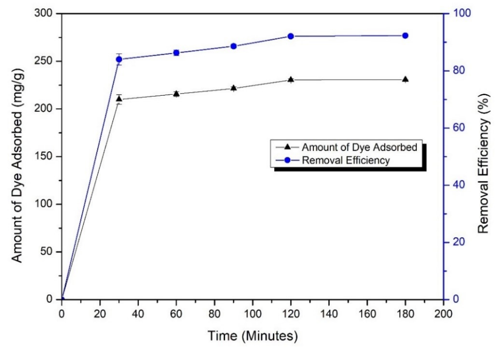

Figure 6: Effect of contact time on the amount of dye adsorbed and removal efficiency

Figure 6 shows that the dye adsorption by the azolla pinnata biomass was increased from 84% to 93% when contact time rises from 30 minutes to 180 minutes respectively. The adsorption rate is high at the initial stage of the experiment because the adsorption sites are more available and methylene blue dye ion is efficiently adsorbed in these sites.

3.6 Effect of pH on dye adsorption

The chemical surface of the adsorbent has huge influence on the absorbate adsorption. This can be done changing the pH value. Figure 7 depicts the effect of the Initial pH value on the MB dye absorption. The removal efficiency and amount of dye adsorbed uplifted with the increment of the pH ranging from 2-12.

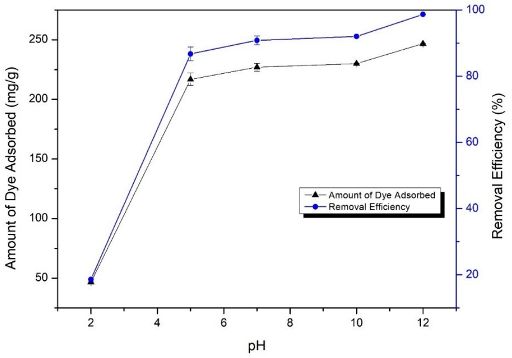

Figure 7: Effect of pH on Dye Adsorption and Removal Efficiency

From the research, it is found that there can be an electrostatic repulsion with a positively charged absorbent and cationic methylene blue dye molecules at small pH values. Hence, it inhibits the adsorption of the dye due to the completes of the hydrogen ion of the methylene blue dye[27, 28, 29].When the pH increases, the biomass surface possesses the negative charge and facilitates the methylene blue dye adsorption by biomass. Hence, the adsorption is more efficient when the negative surface charges is presented. Hence high pH value is suitable for the methylene blue dye removal[30, 31]. Hence,7-12 pH of the dye solution can be appropriate for the MB dye removal form the solution using biomass of azolla pinnata.

3.7 Isotherm and Kinetic Studies

3.7.1 Isotherm Studies

The adsorption isotherm gives the sufficient information about the dye molecules distribution in the absorbent at the equilibrium condition. This studies are performed in order to determine correlation between the adsorption capacity and and residual concentration of the absorbate.The table 1 and figure 8-9 show the parameters of the isotherm model including the regression coefficient R^2. The parameters from the table shows that the langmuir isotherm model ($R^2 = 0.9944$) fits the experimental data better than the freundlich isotherm model($R^2 = 0.8229$). This suggests that the Azolla pinnata biomass follows the mono-layer adsorption mechanism on the homogeneous surface

Table 1: Parameters of Langmuir and Frendlich isotherm

Langmuir Isotherm			Freundlich isotherm		
Q_m(mg/g)	K_a	R^2	n	k_f	R^2
344.8275862	0.144064	0.9944	3.14	69.554	0.8229

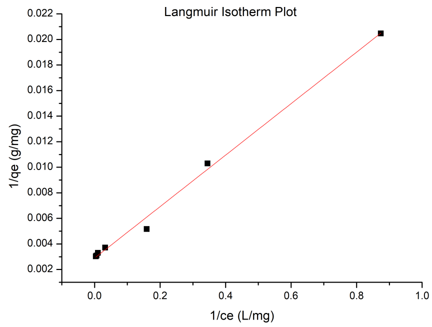

Figure 8: Plot of Langmuir Isotherm

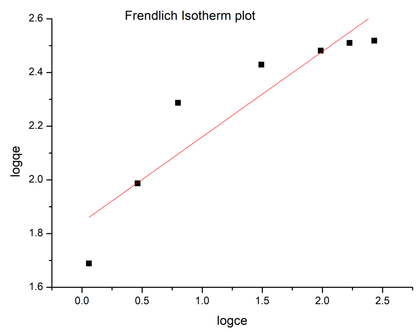

Figure 9: Plot of freundlich isotherm

3.7.2 Kinetic studies

For determining the mechanism and rate controlling process in overall adsorption process, the two kinetic models such as Pseudo first order and second order. From the table 2 and figure 10-11, it is seen that pseudo second order kinetic model ($R^2 = 0.999$) represents the experimental data more precisely than pseudo first order kinetic model ($R^2 = 0.85602$).

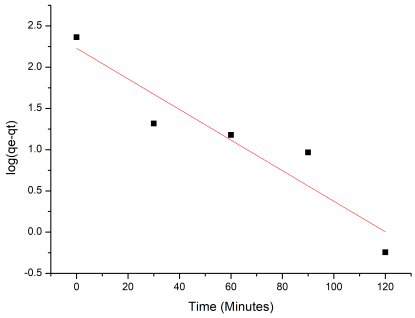

Figure 10: Plot of Pseudo First order model

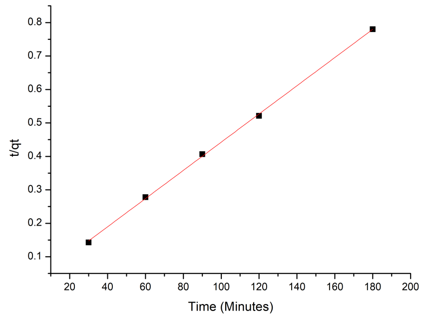

Figure 11: Plot of Pseudo Second order model

Table 2: Parameters of Pseudo first order and Second order kinetic model

model	q_e (mg/g)	k	R^2
First order	169.457	$k_1 = 0.0426$	0.85602
Second order	236.966	$k_2 = 2.232$	0.99941

3.7.3 Comparison with Previous Literature

Adsorption capacity of the biomass of the azolla is found to be higher than the biomass of the other plants such as Ecalyptus Sheathiana bark, Corn, Neem leaf, saw dust and canola residue. Hence, biomass of the azolla pinnata can be good adsorbent for the methylene blue dye removal.

Table 3: Comparison with Previous Studies

SNo	Biomass	Max.Adsorption Capacity(mg/g)	Reference
1	Eucalyptus sheathiana bark	204.08	[32]
2	Corn	47.95	[7]
3	Neem leaf	8.76–19.61	[33]
4	Banana Waste	243.90	[34]
5	Indian Rosewood sawdust	56.4	[11]
6	Cyanobacterial Biomass	238.1	[35]
7	Canola Residue	16.7	[12]
8	Azolla Pinnata biomass	344.8275862	This study

4. Conclusion

The effectiveness of the methylene blue dye adsorption from the aqueous solution by using the biomass of the azolla pinnata was investigated. The effect of the adsorption parameters such as initial dye concentration,contact time and absorbent doses were evaluated. The dye removal efficiency was found to be more than 90% in lower concentration (50 mg/l to 300 mg/l).The best concentration for the dye removal is 100-300 mg/l with the dose of 1 g/l - 1.2 g/l in 120 minutes of contact time. Langmuir isotherm best fits the experimental data than freunlich isotherm model which suggest the monolayer adsorption of the dye by biomass. The maximum Langmuir adsorption of the dye was found to be 344.827 mg/g. In addition to this, the rate of the adsorption was best fitted with the Pseudo-second order kinetic model. Finally, the biomass of the azolla pinnata can be used as the alternative absorbent for the methylene blue dye removal.

Acknowledgments

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