Research Article
Hittite Journal of Science and Engineering, 2023, 10 (2) 145–151
ISSN NUMBER: 2148–4171
DOI: 10.17350/HJSE19030000301

Adsorption and Kinetic Studies of Methylene Blue from Aqueous Solution Using Modified Spent Tea Leaves

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ABSTRACT

In this study, methylene blue was removed from an aqueous solution using modified waste tea leaves. Temperature, pH, contact time, methylene blue concentration, and other adsorption mechanisms were studied. The characterization of the samples was performed using the remove the FTIR and SEM analyses. The isotherm models were carried out to analyze the adsorption mechanisms and for representing data, the Freundlich isotherm model seemed more appropriate. The findings exhibited the highest adsorption capacity was 3.66 mg/g for the untreated biomass at 298 K, 5.46 mg/g for the 0.1 M HCl group at 318 K, and 5.98 mg/g for the 0.05 M NaOH group at 298 K. The adsorption kinetics was used and the pseudo-second-order model performed well for each group. As a natural substitute biomass for methylene blue adsorption from aqueous solution, used tea leaf residue may be employed.

Article History:
Received: 2023/02/03
Accepted: 2023/05/30
Online: 2023/06/30

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This article has been checked for similarity.

Keywords:
Tea leaves, Adsorption, Methylene blue, Modification

INTRODUCTION

It is observed that water pollution, which occurs as a result of the increase in industrial wastes and applications related to the environment, is gradually increasing (1). The sources of water pollution are mostly classified as organic substances, dye, nutritive salts, microorganisms, inorganic substances, detergents, pesticides, heavy metals, radioactivity, fertilizer, oils remove -petroleum products, and waste heat. These pollutants have significant toxic effects on human health, aquatic habitats, and plant species (2, 3).

While dyestuffs are frequently used in the textile industry and widely used in cosmetics, pharmaceuticals, paper, food and leather (4, 5). Dyes are basically organic substances with different structures and molecular weights. These dyestuffs are harmful to the environment and organisms if they are not treated effectively before being released. Methylene blue (MB) is often preferred for dyeing materials such as cotton and silk in the field of leather, wood, and textiles. MB may cause permanent damage to human and animal eyes and cause respiratory distress, nausea, vomiting or mental confusion (6).

Chemical contaminants are removed using a variety of techniques. Among these are chemical precipitation, oxidation, reduction, ion exchange, electrolysis, flocculation, membrane separation, and reverse osmosis (7). Among these methods, it is known that the adsorption method gives better results than the other methods, because of low cost, easy and high removal power, and the possibility of using environmentally safe and natural adsorbents (8).

In the last few years, researchers have used rice husk, tea waste, sugar beet pulp, soybean husk, clay, natural zeolite, sawdust, peanut shells, and fruit shells as adsorbent (9). Tea waste, which is among these adsorbents, cannot be evaluated in any way and it is a low-cost and natural adsorbent. For that reason, some researchers used it for their studies for adsorption experiments and some researchers modified it with carbon to remove pollutants (10).

In this study, chemical modification of tea (with acid and base) and adsorption experiments were carried out to remove methylene blue, which is dangerous for the environment and human health, from aqueous solution. During the experiments, solution pH, time, temperature, and the concentration of methylene blue, and mass of tea waste, were examined. On adsorption

Cite as:
Ozyaman M. N., Cagsirli S. & Ozudogru Y. Adsorption and kinetic studies of methylene blue from aqueous solution using modified spent tea leaves. Hittite J Sci Eng 2023;10(2):145-151. doi:10.17350/hjse19030000301

mechanisms, the Freundlich and Langmuir isotherm models were used. The Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and EDX (Energy Dispersive Spectroscopy) analyses were applied to determine the morphology and elemental composition of the tea waste.

MATERIALS AND METHODS

Methylene blue (MB) was purchased from Merck. Diluted HCl or NaOH was used to pH adjustment. For the adsorption experiments, different concentrations of MB (5 - 150 mg/L) were used to diluted 1000 ppm stock MB solution. In the adsorption experiments, a Mikrotest MSC 30 model shaker was used. The samples were filtered using syringe filter before measured with a spectrophotometer because of clarity. Specord S 600, Analytik Jena was used to measure the amount of MB in the solution. The surface of tea samples was characterized using a Perkin Elmer Spectrum 100 FTIR spectrometer and a scanning electron microscope (Jeol JSM 7100F).

Preparation of the tea waste (*Camellia sinensis*) samples

Black tea samples were obtained from a Turkish producer, Doğadan Company. For adsorption experiments, distilled water was used to clean the black tea samples and obtained by drying at 333 K for 24 hours in an oven until the weight is constant. The adsorbent particle sizes were adjusted to 300-400 µm. and stored to adsorption experiments.

Preparation of chemically modified black tea waste

For the chemically modified black tea waste, one of the dry biomasses was treated with 0.05 M NaOH and one of the dry biomasses was treated with 0.1 M HCl, for 3 hours, then rinsed with distilled water after that. Dried at 333 K for 24 h and was sieved to particle sizes of 300-400 µm.

Method

In order to examine the study, solution pH, contact time, concentration, temperature and quantity tests were carried out. For the adsorption experiments, 200 mg of biomass for each group was placed in Falcon tubes filled with 10 mL of methylene blue (MB) solutions (10 ppm) and then shaken at 200 rpm for one hour. After that, the supernatant was measured with a spectrophotometer at 665 nm. The specific adsorbed amount of MB (Removal %) was founded according to the following equation:

$$\%\text{Removal}=\frac{(C_0-C_e)}{C_0}x100 \quad (1)$$

where Co and Ce are the initial and the equilibrium concentration of MB in the aqueous solution (mg/L).

The effect of initial pH on the adsorption was worked with a different pH (2–9) using 10 mL MB (10 ppm) solution. The adsorption experiments were studied at 200 rpm at room temperature for one hour. After being measured with a spectrophotometer, Eq. 1 was used to find the removal % of MB.

To perform the duration trials, 200 mg of biomass was added to a 10 ML MB (10 ppm) solution of different biomass groups at appropriate pH values due to the maximum removal of MB. The pH value for the untreated tea, treated with HCl and treated with NaOH, was 3, 5 and 9, respectively. The adsorption procedure was carried out at different time intervals (10-300 min) at 298 K. The qt (mg / g) value in MB intake was calculated by giving the equation below:

$$q_t=\frac{(C_0-C_e)}{M}xV \quad (2)$$

The Co value given in the formula gives the initial MB concentration (mg / L), Ce gives the MB concentration (mg /L) at a certain time. V is the volume of MB solution (L) and M is the mass of tea waste (g dry weight).

For effects of the initial MB concentration on adsorption capacity were studied at six different MB solution concentrations (5- 150 ppm) at 298 K and 318 K for a contact time of 100 min. After the measurements, Langmuir and Freundlich isotherm models were used to describe the experimental data.

Adsorption isotherms

The Langmuir and Freundlich isotherms were used at 298K and 318K. The Langmuir model is :

$$\frac{C_e}{q_e}=\frac{1}{q_m a_L}+\frac{C_e}{q_m} \quad (3)$$

where q_e (mg/g) is the amount of M, Ce (mg/L) is the equilibrium concentration of MB, q_m (mg/g) is the maximum adsorption capacity, and a_L is the Langmuir constant.

A linear form of the Freundlich equation is:

$$\log qe=\log Kf + 1/nf \log Ce \quad (4)$$

In this equation, K_f and n_f are constants at given tem-

perature.

Adsorption kinetics

MB adsorption processes can be explained by kinetic models. Lagergren pseudo-first order (Eq. 5) and Ho pseudo-second-order velocity (Eq. 6) equations were utilized to analyze the amount of MB adsorption from tea wastes and to interpret the experimental data. Lagergren's first-order equation is one of the most used equations in adsorption experiments in liquid/solid systems (11).

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (5)$$

$$\left(\frac{t}{q_t}\right) = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (6)$$

where q_e and q_t are the amounts of MB adsorbed at the equilibrium (mg/g) and time *t*, respectively. For the pseudo-first order adsorption, k_1 is the pseudo-first-order rate (1/min) and k_2 is the rate constant of the pseudo-second-order (g/mg. min).

RESULTS AND DISCUSSION

Characterizing biomass

FTIR Analyses

Various functional groups on biomass were determined using FTIR absorption spectrometry. The untreated and treated with HCl and NaOH tea wastes (before and after MB adsorption) are shown in Figure 1. The adsorption bands at approximately 3350 and 2900 cm^{-1} for biomass were assigned to the -OH groups in water and -CH stretching, respectively (12). The sharp peaks at 1731 cm^{-1} were due to the carbonyl groups (13). The adsorption bands at approximately 1630 cm^{-1} were the C=C and C=N stretches in amide groups. The 1516 cm^{-1} and 1465 cm^{-1} peaks are belonged to the aromatic C-C stretches (14). The symmetric stretching vibration peak at 1161 cm^{-1} belongs to the C-O-C bond (15), the peaks at 1027 cm^{-1} belong to aromatic C-O stretching and the peak at 719 cm^{-1} belongs to the S-O stretching. We can say that the -OH, -CH, C=C, C=N groups are responsible for the adsorption.

SEM Analyses

The porosity characteristics of the biomass is important for adsorption processes. The SEM images and EDX analyses of different tea waste groups (untreated and treated with MB) are given in Fig. 2. According to the results,

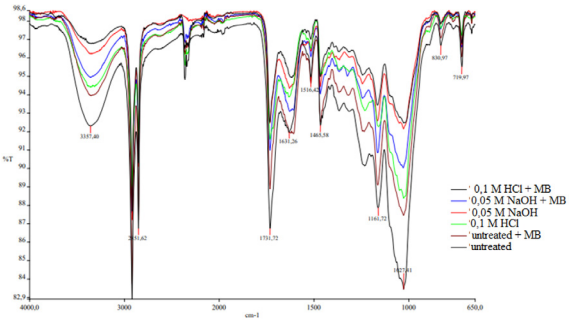

Figure 1. FTIR spectrum of biomass before and after adsorption of MB for each group

different materials caused different physical changes. When tea waste interacted with HCl or NaOH, it showed that the surface was more indented. The pores were found to be even more rounded when interacting most with NaOH. In the EDX analysis, the presence of C and O in the structure of the tea was observed. The Cl- ions were observed in the EDX analyses after pretreatment with HCl. Similarly, the presence of Na+ ions appeared in the EDX analyses after pretreatment with NaOH (Figure 2).

Effect of pH

The negative or positive charge movements of the adsorbent in the solution are important for adsorption. Because pH affects the physicochemical behavior of the solution. In this way, it also affects the load value of the charge (16). The results of the removal of MB solution with tea waste with different pH values are shown in Figure 1.

It was determined that different pH values in MB solution changed the removal rate. MB competes with the H^+ ions in the solution as the pH changes. As the pH increased from 2 to 3 in the untreated group, the % removal value first increased and then decreased.

After pH 5, the % removal value increased again. In the HCl group, the % removal value increased between pH 2-5 and then decreased. It is seen that the % removal value decreased very much with the rise of pH from 5 to 7. In the NaOH group, the removal % value increased with the increase in pH. It is thought that the reason for the change in the removal capacity due to the pH change is the competition between MB and H^+ and mainly due to the formation of soluble hydroxyl complexes (17). The highest % removal values were found as 90% at pH 3 for the untreated biomass, 85% at pH 5 in the 0.1 M HCl group, and 73% at pH 9 in the 0.05 M NaOH group.

Effect of contact time

In order to examine the effect of contact time on adsorption efficiency, the experiments were followed through

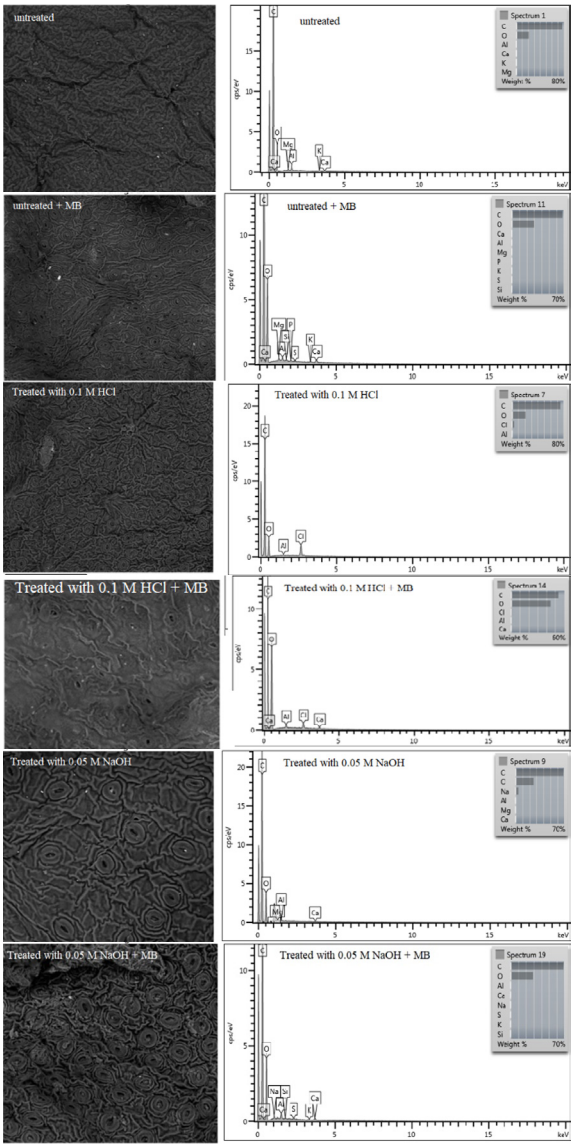

Figure 2. SEM micrographs of biomass (untreated and treated) with MB for each group

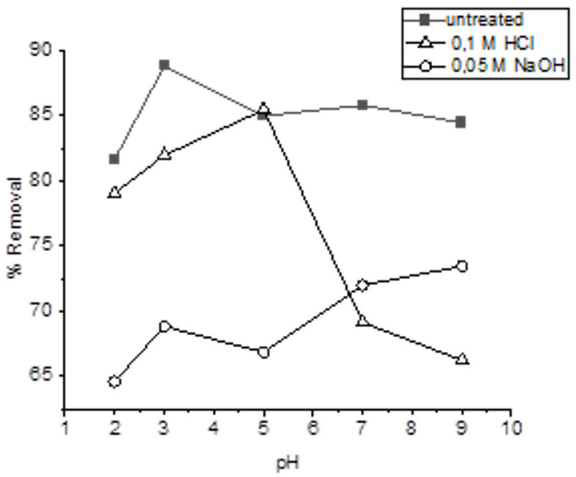

Figure 3. Effect of pH on Removal % of tea waste.

between 10 minutes and 300 minutes at the pH where the % removal values of each group were the highest. In this context, pH 3 for the untreated group, pH 5 for the HCl group and pH 9 for the NaOH group were selected.

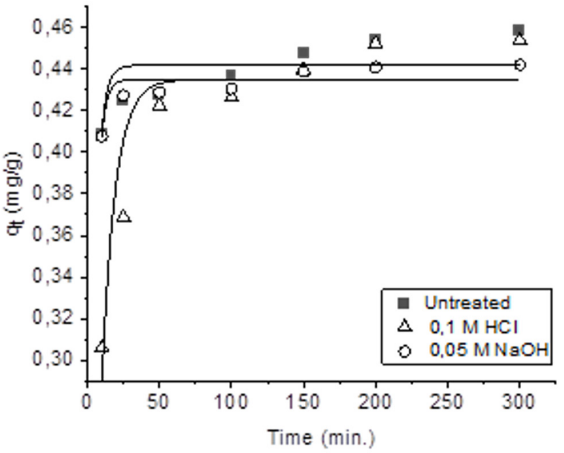

Figure 4. Effects of adsorbent time on adsorption of tea waste.

The relationship between MB and dyestuff in the solution with different contact time is given in Figure 2.

It has been concluded that the removal efficiency of MB dyestuff using waste black tea powder increases with the contact time. In first 50 min., it was seen that, the qt value was rapid and then increased slowly. It was observed that the system came to equilibrium after an average of 150 minutes. The reason the system remained stable after a certain time was related to that the biomass reached saturation (18).

Effect of initial MB concentration

Initial concentration and temperature were effective on adsorption at a given temperature (19). Temperature affects both the movement of the particles and the adsorption capacity depending on whether the mechanism is endothermic or exothermic. For this reason, the isotherm models were used at two different temperatures. The Langmuir isotherm for MB adsorption in tea waste at 298 K and 318 K is given in Figure 3, while the Freundlich isotherm for MB adsorption in tea waste at 298 K and 318K is given in Figure 4. The isotherm parameters results are in Table 1. It goes to showed that, the adsorption of MB in tea waste fitted well to the Freundlich isotherm models having correlation coefficients higher than 0.96 at all temperature. The value defined in the Freundlich isotherm model was calculated as 0.45-1.28. This means that the adsorption of MB was favored and positive. The highest adsorption capacity was 3.66 mg/g for the untreated biomass at 298 K, 5.46 mg/g for the 0.1 M HCl group at 318 K, and 5.98 mg/g for the 0.05 M NaOH group at 298 K.

We can say that the adsorption capacity increases when tea waste is modified with acid or base. Although the ad-

sorption capacity of the waste tea was not higher than the other chemical adsorbents, it was higher than corncob, almond shell, walnut, and fire wood when compared to natural adsorbents (Table 2).

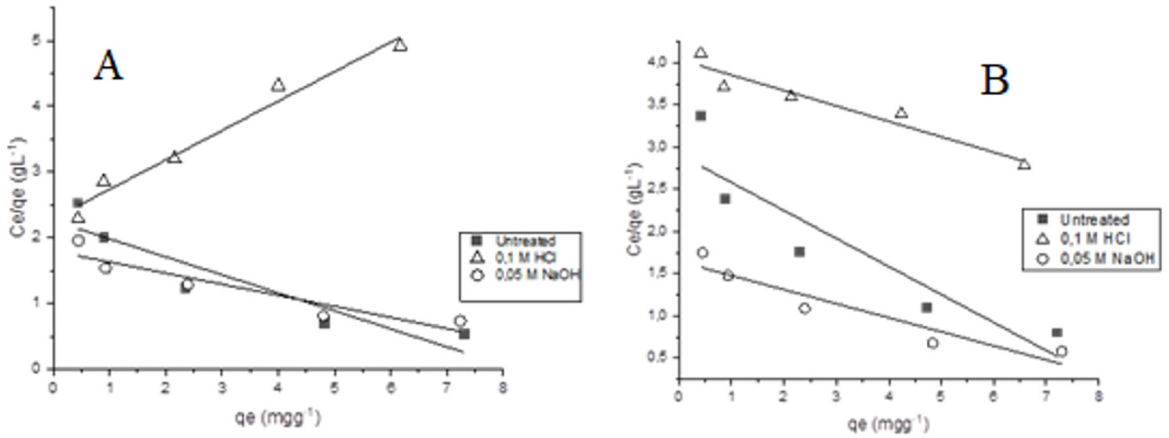

Figure 5. Langmuir isotherm for MB adsorption at 298 K (A) and 318K (B).

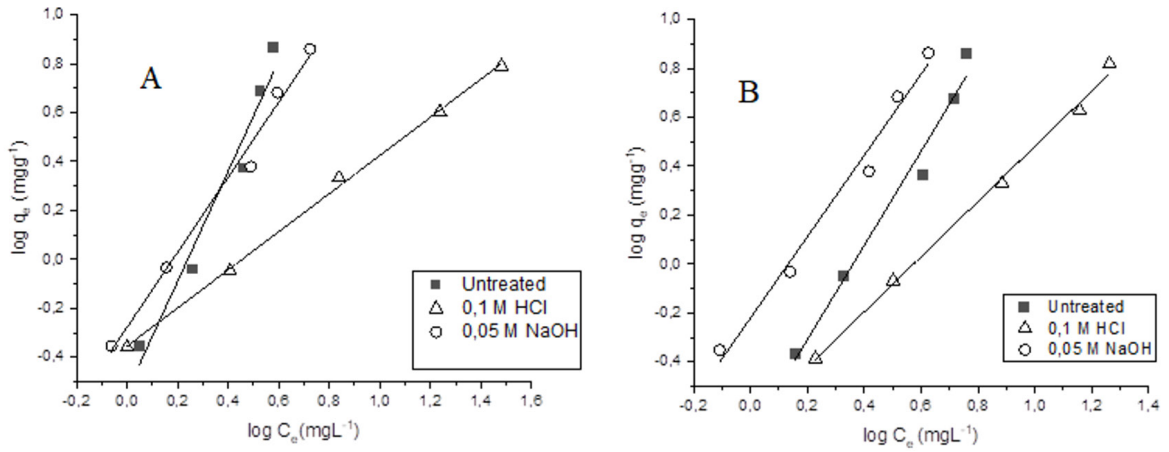

Figure 6. Freundlich isotherm for MB adsorption at 298 K (A) and 318 K (B).

Table 1. Parameters of Langmuir and Freundlich isotherms for adsorption of MB in tea waste

		Langmuir			Freundlich		
Biomass	**Temperature (K)**	**q_{max} (mg/g)**	**a_L**	**R_L^2**	**n_f**	**K_f(mg/g)**	**R_F^2**
Untreated	298	3.66	0.121	0.8525	0.45	0.29	0.9626
	318	3.01	0.11	0.8363	0.47	0.16	0.9765
0.1 M HCl	298	2.24	0.195	0.9701	1.28	0.44	0.9984
	318	5.46	0.045	0.9229	0.89	0.23	0.9966
0.05 M NaOH	298	5.94	0.094	0.8758	0.65	0.53	0.9878
	318	5.98	0.10	0.8989	0.60	0.60	0.9834

Adsorption Kinetics

Fitting the experimental data to different kinetic models, adsorption rates, process models, and relationships between adsorbent/adsorbate allows us to examine predictive information about whether the interaction is physical or chemical (27). The kinetics of MB adsorption were followed with Pseudo-first-order and Pseudo-second-order models. The results are given in Table 3. In the table, the Pseudo-second-order kinetic model is the most appropriate for MB adsorption. The correlation coefficient R2 of the Pseudo-second-order equation is very close to 1 (0.999) for each group of biomasses. In addition, it was observed that it was a good agreement between the calculated qe values with experimental qe values for the 0.1 M HCl group.

Table 2. Comparison of adsorption capacities of MB in various adsorbents.

Adsorbent	q_m (mg/g)	Reference
Corncob	0.809	(20)
Hazelnut shell	8.82	(21)
Almond shell	1.33	(21)
Walnut	3.53	(21)
Jute processing waste	22.47	(22)
Water hyacinth root powder	8.04	(23)
Orange peel	20.50	(24)
Fire wood based activated carbon	1.21	(25)
Natural Pumpkin Seed Shell	12.61	(26)
Untreated tea waste	3.66	This study
Tea waste (treated with 0.1 M HCl)	5.46	This study
Tea waste (treated with 0.05 M NaOH)	5.98	This study

determine the characteristic properties of biomass, FTIR, SEM and EDX analyses were preferred. It has been observed that the pre-concentration process is affected by the change in pH value. The highest adsorption capacity was found as 90% at pH 3 for the untreated group, 85% at pH 5 for the 0.1 M HCl group, and 73 % at pH 9 for the 0.05 M NaOH group. The calculated maximal adsorption capacity was found to be 3.66 mg/g for untreated biomass at 298 K, while the 0.1 M HCl group at 318 K increased by calculating 5.46 mg / g and the 0.05 M NaOH group at 298 K increased by calculating 5.98 mg / g. According to the results of the analysis of kinetic and isotherm models, the so-called quadratic model was found to be better adapted to different initial MB concentrations of each study group. The Freundlich model was suitable for the equilibrium data. When we examined the FTIR analysis,

Table 3. Kinetic parameters of MB adsorption in biomass

	Pseudo-First Order Kinetic			Pseudo- Second Order Kinetic		
Dye concentration (mg/L)	**R^2**	**k_1 (1/min)x10^{-4}**	**q_e (mg/g)×10^{-3}**	**R^2**	**k_2 (g/(mg/min))**	**q_e (mg/g)**
Untreated	0.9843	0.26	1.22	0.9998	0.66803	0.461
0.1 M HCl	0.9169	0.4	5.82	0.9998	0.354398	0.462
0.05 M NaOH	0.9525	0.4	0.44	0.9999	1.453058	0.444

Effect of Adsorbent Dosage

The effect of the adsorbent dosage on the adsorption of MB was carried out varying amounts of adsorbent in the range of 0.1-1.5 g. The results are shown in Figure 7. As shown, the fit curves increased with biomass dosage for the three biomass groups. The highest adsorption capacity was reached at 150 g/L.

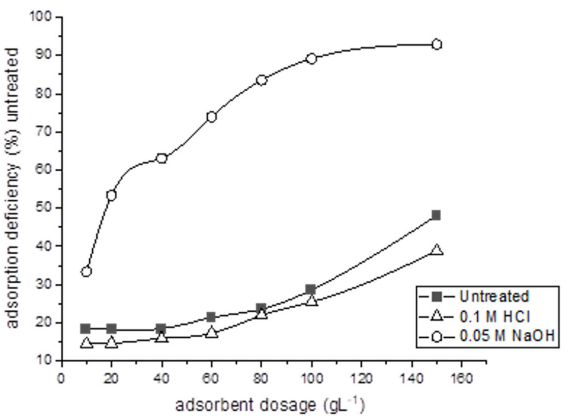

Figure 7. Effects of adsorbent time on adsorption of tea waste.

CONCLUSION

In this work, black tea waste powder was used to remove methylene blue (MB) from the aqueous solution. pH value, contact time, initial MB concentration and temperature were studied. FTIR, SEM, and EDX analyses were preferred to identify the properties of biomass. In order to the -OH, -CH, C=C and C=N groups were responsible for the adsorption. Considering the SEM analysis, the surface of the biomass changed with pretreatment. The study confirmed that black waste tea residue could be used as a natural alternative biomass for methylene blue adsorption from an aqueous solution.

Acknowledgements

This study was funded by the Çanakkale Onsekiz Mart University research foundation (BAP, project no: FLÖ-AP-2022-3973).

The author denies any conflict of interest.

CONFLICT OF INTEREST

The authors denies any conflict of interest.

AUTHOR CONTRIBUTION

The original research article titled "Adsorption and kinetic studies of methylene blue from aqueous solution using modified spent tea leaves" prepared by us has not been published in any journal before and is not in the publication stage in any other journal. All authors contributed equally to the submission of our article for consideration in the Hittite Journal of Science and Engineering.

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