



Removal of methylene blue from water environment in an environmentally responsible manner: green remediation potential of alkali-treated biomass of *Prunus laurocerasus* L

Fatih Deniz¹

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Abstract

Methylene blue (Mb) is a synthetic dye that is broadly used in many practical applications but also poses many risks to the health of living beings. In this study, for the first time in the literature, the green remediation potential of alkali-treated waste leaf biomass of *Prunus laurocerasus* L. for Mb biosorption from aqueous medium was investigated. The biosorption process was characterized through the techniques of SEM and FTIR. The biosorbent exhibited a favorable surface structure for Mb biosorption. The biosorbent reached peak efficiency at pH of 8, biosorbent dose of 10 mg, biosorption time of 120 min and initial Mb concentration of 15 mg L⁻¹. For Mb biosorption, the isotherm data agreed well with Dubinin-Radushkevich (D-R) isotherm model, while the kinetic data provided a good fit to the pseudo-second-order (PSO) kinetic model. The biosorption was thermodynamically physical, spontaneous and favorable process. Mb biosorption capacity of the biosorbent was calculated to be 141.098 mg g⁻¹. High success of the biosorbent in Mb biosorption indicated its superior environmental remediation potential for large-scale wastewater treatment in an environmentally responsible way.

Keywords Methylene blue · Aqueous environment · Green remediation · Biosorption · *Prunus laurocerasus* L

1 Introduction

A considerable amount of untreated wastewater is discharged directly into immediate environment as a result of numerous human-induced activities. These wastewaters originating from industrial, urban or agricultural activities contain diverse inorganic and organic micropollutants such as micro/nanoplastics, synthetic dyes and toxic metals. Continuous discharge of such micropollutants without prior treatment in a manner that respects nature leads to significant adverse effects on living health. Synthetic dyes, which are the subject of this study, cause visual pollution in water bodies, increase the biochemical and chemical oxygen demands of water sources and weaken the photosynthetic activity of living things. They bioaccumulate through the food chains and exhibit toxic, mutagenic and carcinogenic

effects on living beings [1–3]. Additionally, synthetic dyes, are persistent chemicals with low biodegradability, so they must be properly removed from the contaminated environment to protect living health.

The biosorption method, where the material used to retain the pollutant is of biological origin, has become quite popular in the last few decades for the removal of micropollutants such as synthetic dyes. This wastewater treatment technique has several advantages such as simplicity, cost-effectiveness, high efficiency, rapid result and most importantly, compatibility with nature. Due to wide range of bio-based materials and their very different features, the biosorption process has attracted the attention of many researchers and in recent years, numerous studies have been conducted to remove various toxic synthetic dyes using various biosorbent materials [1, 4–6]. However, more efficient and economical novel biosorbents are still needed for this technique to find practical field-based wide application in the treatment of synthetic dyes from wastewater medium.

This study was performed to examine the biosorptive capability of the waste leaf biomass of *Prunus laurocerasus* L. in the removal of Mb dye from water environment for the

✉ Fatih Deniz
fdeniz@harran.edu.tr

¹ Environmental Protection Technologies Department, Harran University, 63850 Şanlıurfa, Türkiye

first time in literature. *P. laurocerasus* L. is a hardy plant species belonging to the *Rosaceae* family. It is widely cultivated in temperate regions worldwide for various purposes such as decorative, landscaping, barrier, food and medicine [7–9]. The use of the abundant plant leaf biowaste in this way is of great importance both economically and environmentally. Mb is a synthetic dye widely used in many practical applications but it also poses many risks to the health of living beings [6, 10, 11]. The waste biomass of *P. laurocerasus* L. was subjected to alkali treatment in order to improve its Mb biosorption performance. The synthetic dye treatment process was characterized through the analytical techniques of SEM and FTIR. The impact of different biosorption parameters (pH, biosorbent dose, time and initial Mb concentration) on the treatment of Mb by the biosorbent was explored. The isotherm, kinetic and thermodynamic tests were performed for a better understanding of Mb biosorption.

2 Materials and methods

2.1 Mb solution

Mb dye (Merck) was obtained from a local medical company (Samsun, Turkey). The desired initial concentration of the synthetic dye was obtained by diluting 1000 mg L⁻¹ stock Mb solution with ultrapure water (Merck Direct-Q 3 UV). pH of Mb solution was adjusted by adding 0.1 mol L⁻¹ NaOH and/or HCl (Merck). pH value of the synthetic dye solution was measured with a pH meter (Hach HQ40d). A standard curve was created by measuring the absorbance of Mb (663 nm) via a spectrophotometer (Thermo Genesys 10 S) to determine the concentration of Mb in the test medium.

2.2 Biosorbent preparation

P. laurocerasus L. waste leaf biomass was provided from Samsun province of Türkiye. The leaf biomass was first cleaned with ultrapure water and then subjected to oven (Mikrotest MST 120D) drying for 24 h. The waste biomaterial was then passed through a grinder (Waring) and sieved using a sieve (0.5 mm, Loyka). For the alkali treatment, 1 g of the raw powdered biomass was added into 100 mL of NaOH (0.3 mol L⁻¹) solution and the mixture was shaken for 24 h at room temperature with the help of a hot plate magnetic stirrer (Stuart SB162). Then, the biomaterial was separated from the liquid phase and washed with ultrapure water until pH value was neutral. The treated biosorbent was dried again and stored under appropriate conditions to be used in Mb biosorption experiments.

2.3 Characterization

The synthetic dye treatment process was characterized through the analytical techniques of SEM and FTIR. The morphological properties of the treated biosorbent before and after Mb biosorption were studied using a SEM device (Zeiss Evo Ls 10) at a magnification range of 100–5000 times. FTIR spectra of the biosorbent before and after Mb biosorption were recorded in the range of 4000–400 cm⁻¹ with the help of a FTIR spectrometer (PerkinElmer Spectrum 400) to study the surface functional groups responsible for the retention of the molecules of Mb dye.

2.4 Biosorption study

The biosorption study was conducted at ambient temperature (18 °C) under different operating conditions in 100 mL conical flasks containing Mb solution. Different amounts of biosorbent were added to Mb solution, which was prepared at different Mb concentrations according to the experimental conditions, and the mixture was agitated for a certain period of time. When the specified experimental time was reached, a certain amount of liquid phase sample was separated from the mixture. The concentration of Mb in the solution was determined by a spectrophotometer. The amount of Mb retained per gram of the biosorbent at a time t (q_t , mg g⁻¹) (Eq. 1), the amount of Mb retained per gram of the biosorbent at equilibrium (q_e , mg g⁻¹) (Eq. 2) and Mb treatment yield of the biosorbent (R_e , %) (Eq. 3) were calculated using the equations in Table 1.

2.5 Impact of various biosorption parameters

Mb biosorption performance of the biosorbent was evaluated by studying the impacts of various operating parameters. The experiments were conducted by keeping the other operating parameters constant while evaluating the impact of a parameter on the removal process, and thus optimum operating conditions were obtained.

Table 1 Equations

Equation	Number
$q_t = \frac{(C_0 - C_t)V}{m}$	1
$q_e = \frac{(C_0 - C_e)V}{m}$	2
$R_e = \frac{C_0 - C_t}{C_0} \times 100$	3

C_0 : initial Mb concentration in solution (mg L⁻¹), C_t : Mb concentration at a time t in solution (mg L⁻¹), C_e : equilibrium Mb concentration in solution (mg L⁻¹), V : Mb solution volume (L), m : biosorbent mass (g)

2.6 Isotherm (equilibrium), kinetic (rate) and thermodynamic studies

The isotherm, kinetic and thermodynamic studies were conducted for a better understanding of Mb biosorption process. For the isotherm study, the relationship between the amount of Mb retained on the biosorbent (q_e , mg g⁻¹) and Mb concentration remaining in water medium (C_e , mg L⁻¹) in equilibrium at a constant temperature value was analyzed with Langmuir [12] (Eq. 4), Freundlich [13] (Eq. 5) and D-R [14] (Eq. 6) (Table 2) models.

For the kinetic analysis, the relationship between Mb biosorption yield of the biosorbent (q_t , mg g⁻¹) and the process time (t , min) was studied with the pseudo-first-order (PFO) [15] (Eq. 7), PSO (Eq. 8) [16] and intra-particle diffusion (IPD) [17] (Eq. 9) (Table 3) models.

The thermodynamic study was performed using the parameter of free energy change (Gibbs, ΔG° , kJ mol⁻¹) [3] (Eq. 10) for Mb biosorption.

$$\Delta G^\circ = -RT \ln K_D \quad (10)$$

T is temperature (K), R is gas constant (J mol⁻¹ K⁻¹), K_D is coefficient of distribution (C_{be}/C_e , -) and C_{be} is equilibrium Mb concentration on biosorbent (mg L⁻¹).

The performance of all the models in representing the biosorption data was assessed with the statistical parameters of determination coefficient (R^2) (Eq. 11), adjusted R^2 (R^2_{adj}) (Eq.

Table 2 Isotherm models

Model	Number
$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$	4
$q_e = K_F C_e^{\frac{1}{n_F}}$	5
$q_e = q_m e^{-B\epsilon^2}$	6

q_m : maximum Mb biosorption capacity of biosorbent (mg g⁻¹), K_L : a parameter about biosorption energy (L mg⁻¹), K_F : a parameter about Mb biosorption capacity of biosorbent (mg g⁻¹ (L mg⁻¹)^{1/n_F}), n_F : a parameter about biosorption intensity (-), B : a parameter about biosorption energy (mol² kJ⁻²), ϵ : Polanyi biosorption potential (J mol⁻¹)

Table 3 Kinetic models

Model	Number
$q_t = q_e(1 - e^{-k_1 t})$	7
$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t}$	8
$q_t = C + k_p \sqrt{t}$	9

k_1 : PFO rate constant (min⁻¹), k_2 : PSO rate constant (g mg⁻¹ min⁻¹), C : a parameter about boundary layer thickness (mg g⁻¹), k_p : IPD rate constant (mg g⁻¹ min^{-1/2})

Table 4 Statistical parameters

Parameter	Number
$R^2 = 1 - \frac{\sum_{i=1}^n (x-y)^2}{\sum_{i=1}^n (x-\bar{x})^2}$	11
$R^2_{adj} = 1 - \frac{(1-R^2)(n-1)}{n-p-1}$	12
$RMSE = \sqrt{\frac{1}{n} \sum_{i=1}^n (x-y)^2}$	13

n : number of tests, x : experimental values, y : predicted values, $\bar{x}$: mean of actual values, p : number of independent variables

12) and root mean square error ($RMSE$) (Eq. 13) (Table 4) using SigmaPlot program (Systat software).

3 Results and discussion

3.1 Characterization study

The morphological features of the alkali-treated material before and after Mb treatment were studied using a SEM. Figure 1. displays SEM pictures of the material before and after Mb removal. It was seen from the figures that the material had a wavy heterogeneous surface structure containing cavities of various sizes. This surface structure indicated the suitability of the material for Mb biosorption [2, 6, 18, 19]. The changes in SEM image after the biosorption of Mb (Fig. 1b) indicated that the synthetic dye molecules were retained on the biosorbent surface.

To study the active groups responsible for the retention of the molecules of Mb dye, the analysis of FTIR spectrometer was conducted. Figure 2. shows FTIR spectra of the biosorbent before and after Mb biosorption. The band at 3335 cm⁻¹ was due to O-H group. The bands at 2919 cm⁻¹ and 2850 cm⁻¹ were related to C-H group. The bands at 1731 cm⁻¹ and 1608 cm⁻¹ were attributed to the groups of C=O and C=C. The bands at 1455 cm⁻¹, 1412 cm⁻¹, 1377 cm⁻¹, 1315 cm⁻¹, 1245 cm⁻¹ and 1162 cm⁻¹ were associated with C-O, C-H, C-O-C, C-N, C=C and O-H groups. The strong band at 1043 cm⁻¹ was due to C-O-C group. The small bands at 895 cm⁻¹, 820 cm⁻¹, 778 cm⁻¹ and 719 cm⁻¹ were associated with the groups of C-H and C-O [2, 5, 6, 18, 19]. This characterization study revealed that the biosorbent was quite rich in terms of the functional groups. The changes in FTIR spectrum after the treatment of Mb (Fig. 2b) indicated that the rich functional group profile of the biosorbent played a very important role in Mb biosorption.

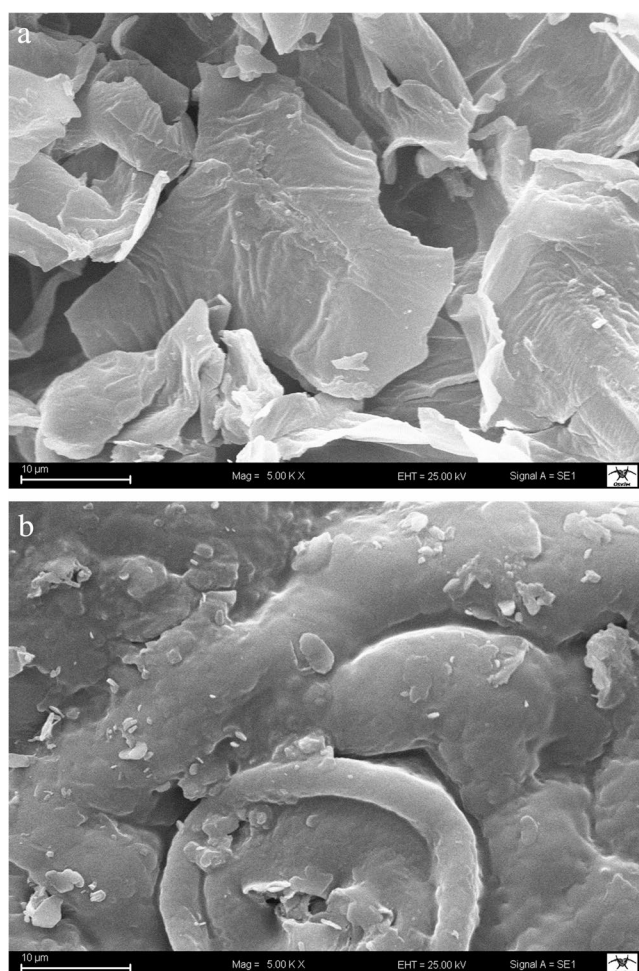


Fig. 1 SEM pictures of biosorbent before (a) and after Mb biosorption (b)

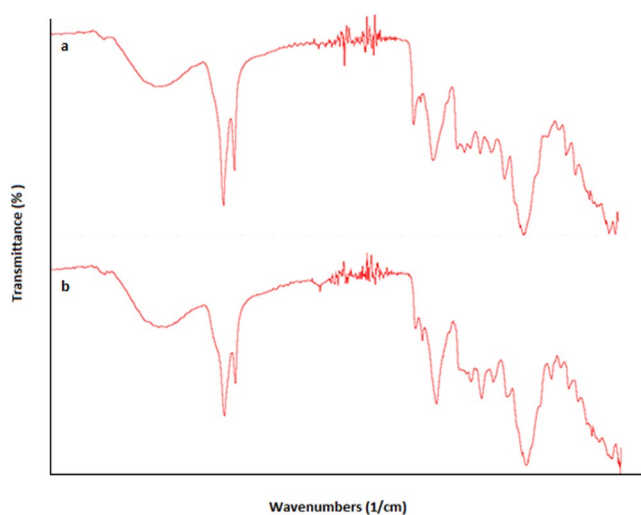


Fig. 2 FTIR spectra of biosorbent before (a) and after Mb biosorption (b)

3.2 Impact of pH

The impact of pH on Mb biosorption was investigated by changing the initial solution pH from 4 to 8. Figure 3. presents the impact of pH on the treatment of the synthetic dye. Mb biosorption yield of the material increased with the change of solution pH from 4 to 8. The active sites on the biosorbent surface were more protonated at low solution pH and thus positive charges surround the surface of the biosorbent. Since Mb is a cationic dye, the competition between Mb molecules and protons for the active sites on the biosorbent limited the retention of Mb on the biosorbent. As pH increased, the active sites on the surface were deprotonated and became negatively charged. These sites created high electrostatic attraction fields for the cationic Mb molecules, resulting in an increase in the performance of the material [20, 21]. Thus, it was found that the most suitable pH value for Mb biosorption was 8.

3.3 Impact of biosorbent dose

The impact of the biosorbent dose on the removal of Mb by the biosorbent was studied in the range of the material dose varying from 10 to 30 mg. The impact of the material dose on Mb biosorption is shown in Fig. 4. Mb biosorption yield

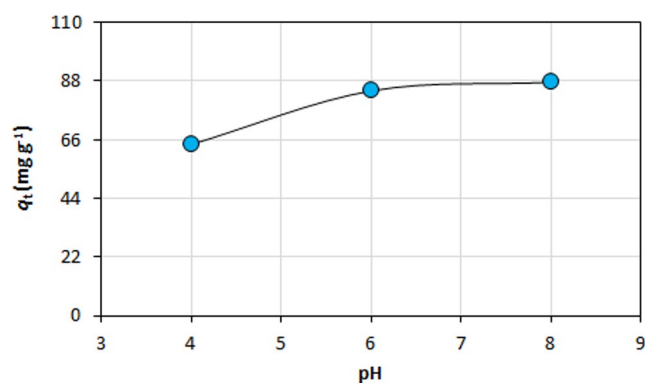


Fig. 3 Impact of pH on biosorption of synthetic dye

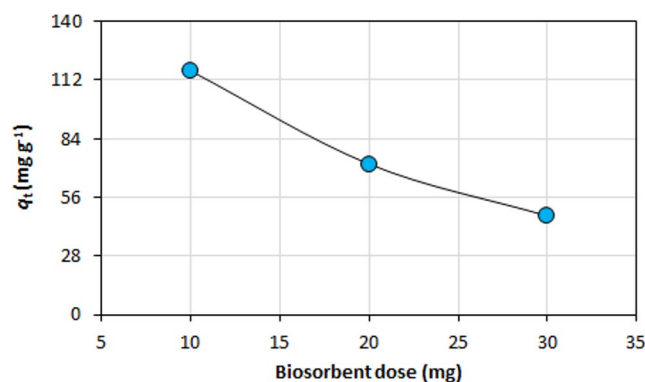


Fig. 4 Impact of biosorbent dose on Mb biosorption

decreased with the dose increase. As the biosorbent dose increased, the number of binding sites to retain the molecules of Mb increased. However, since the concentration of Mb did not change in aqueous medium, the active sites were not completely occupied by Mb molecules, resulting in lower biosorption yield at higher biosorbent doses. The overlapping of the surfaces of the biosorbent particles might be another reason for this situation [2, 22]. Thus, the optimum biosorbent dose for Mb removal was found to be 10 mg.

3.4 Impact of time

The impact of the contact time on the treatment of Mb was examined in the range of 0–360 min. The results are demonstrated in Fig. 5. Mb biosorption increased with increasing contact time and the equilibrium position was reached within 120 min. Initially, Mb biosorption increased gradually due to the availability of numerous suitable active sites on the biosorbent for Mb molecules retention. However,

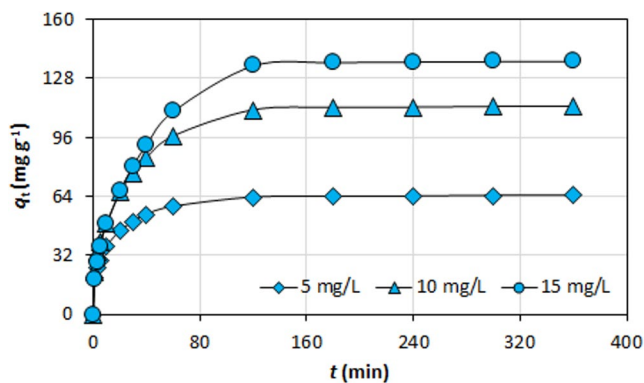


Fig. 5 Impacts of contact time and initial Mb concentration on biosorption of Mb

after a certain time, the vacant active sites were almost completely filled by Mb molecules, and Mb removal reached the equilibrium position [11, 23]. Thus, a time of 120 min was found to be the optimum contact time value for Mb biosorption.

3.5 Impact of initial Mb concentration

The impact of the initial Mb concentration on the biosorption efficiency was investigated by varying Mb concentration from 5 to 15 mg L⁻¹. Figure 5. also displays the impact of Mb concentration on the biosorption capacity. The biosorption efficiency increased with increasing Mb concentration. The higher driving force created by the increasing concentration gradient with increasing Mb concentration provided more mass transfer of Mb molecules to the biosorbent binding sites [2, 23]. Thus, the optimum initial Mb concentration was found to be 15 mg L⁻¹.

3.6 Isotherm study

The equilibrium data for Mb biosorption were analyzed using Langmuir, Freundlich and D-R models. The results are given in Table 5. The performance of these models in representing the equilibrium data was assessed with the statistical parameters of R^2 , R^2_{adj} and $RMSE$. The results indicated that the isotherm data for Mb biosorption agreed well with D-R model. B parameter of this isotherm model is used to calculate a significant parameter known as the mean free energy (E) (Eq. 14).

$$E = \frac{1}{\sqrt{2B}} \quad (14)$$

Table 5 Isotherm study results for biosorption of Mb

Model	MB concentration (mg L ⁻¹)	Parameter				
Langmuir		q_m	R_L	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	-	-	-	-
	5	141.098	0.101	0.965	0.930	9.820
	10		0.053			
Freundlich	15		0.036			
		K_F	n_F	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹ (L mg ⁻¹) ^{1/n_F}	-	-	-	-
		152.580	1.869	0.931	0.863	13.743
D-R		q_m	E	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	kJ mol ⁻¹	-	-	-
		142.072	2.865	0.989	0.977	5.616

Table 6 Mb biosorption ability of various materials

Material	Biosorption ability (mg g ⁻¹)	Reference
Magnetized tea waste	3.06	[6]
Magnetized peanut shells	19.68	[6]
Nontronite/ <i>Enteromorpha</i> sp. biocomposite	115.6	[10]
Wheat hull (Siyez)	62.500	[11]
Rice hull (Sarı Kılçık)	54.945	[11]
Garlic stalk (Taşköprü)	370.370	[11]
Alkaline-treated <i>Carpobrotus edulis</i>	153.9	[21]
<i>Wodyetia bifurcata</i> leaves	58.74	[28]
Alcali-treated waste leaf of <i>P. laurocerasus</i> L.	141.098	Here

The value of E for Mb biosorption was determined as 2.865 kJ mol⁻¹. E was less than 8 kJ mol⁻¹. These results indicated a multi-layer physical biosorption process [24, 25].

K_L parameter from Langmuir model is used to compute the separation factor (R_L) (Eq. 15).

$$R_L = \frac{1}{1 + K_L C_0} \quad (15)$$

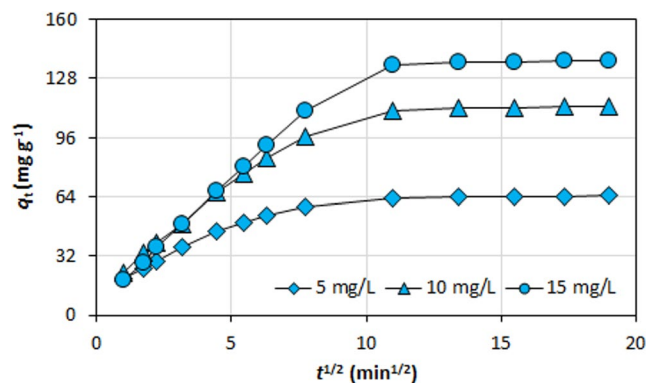
R_L value for Mb removal was found to be in the range of 0.036–0.101. These values between 0 and 1 indicated that Mb biosorption process was favorable [5, 19]. n_F parameter from Freundlich model was found to be 1.869 for Mb biosorption. It was in the range of 1–10. This result disclosed a favorable process [4, 26, 27].

Mb biosorption capacity of the material used was calculated to be 141.098 mg g⁻¹. In Table 6, its Mb removal ability was compared with that of several other materials. From the table it was seen that the biosorbent exhibited high biosorption capacity. In addition to its high efficiency, the

biosorbent also has favorable features such as rapid treatment, low cost and easy production. These features make biosorbent a good choice for synthetic dye removal.

3.7 Kinetic study

The biosorption kinetic data was examined using the PFO, PSO and IPD models. The kinetic analysis results are given in Table 7. The performance of these models in representing the kinetic data for Mb removal was assessed with R^2 , R^2_{adj} and $RMSE$. The results showed that Mb biosorption kinetic data provided a good fit to the PSO model. This agreement indicated that Mb biosorption was mainly dependent on chemical interactions [10, 23, 29]. Additionally, the impact of the IPD stage on the removal of Mb from aquatic environment was studied by IPD model. Figure 6. shows the plots of the IPD model. The model plots did not pass via the origin point. This result showed that Mb removal process

**Fig. 6** Plots of IPD model for biosorption of Mb**Table 7** Results of kinetic analysis for Mb biosorption

Model	Mb concentration (mg L ⁻¹)	Parameter				
PFO		q_e	k_1	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	min ⁻¹	-	-	-
	5	60.200	0.111	0.838	0.824	6.840
	10	108.939	0.051	0.914	0.907	10.315
PSO	15	135.491	0.034	0.961	0.957	9.507
		q_e	k_2	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	g mg ⁻¹ min ⁻¹	-	-	-
	5	64.175	0.00266	0.941	0.936	4.126
IPD	10	116.570	0.00070	0.965	0.962	6.588
	15	148.427	0.00032	0.979	0.977	6.985
		C	k_p	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	mg g ⁻¹ min ^{-1/2}	-	-	-
	5	30.133	2.288	0.767	0.746	8.218
	10	38.289	4.910	0.823	0.807	14.821
	15	33.070	6.809	0.860	0.847	17.885

occurred by several biosorption stages, including intra-particle diffusion [26, 27].

3.8 Thermodynamic study

The thermodynamic study was performed using ΔG° parameter. The values were determined in the range of -13.482 – -12.255 kJ mol $^{-1}$. The negative ΔG° values showed that Mb removal was a favorable and spontaneous process. These negative values (-20 to 0 kJ mol $^{-1}$) indicated a physical biosorption, involving electrostatic interaction between the binding sites and Mb molecules [18, 23, 26].

4 Conclusions

This study demonstrated the potential of alkali-treated waste leaf biomass of *P. laurocerasus* L. as a cheap, effective and environmentally friendly biosorbent for Mb removal from aqueous medium. The characterization analysis revealed that the biosorbent exhibited favorable structural properties for synthetic dye biosorption. The biosorbent reached maximum biosorption efficiency under the conditions of pH 8, 10 mg biosorbent dose, 120 min and 15 mg L $^{-1}$ initial Mb concentration. The isotherm data agreed well with Dubinin-Radushkevich model, indicating a multi-layer physical biosorption process. The kinetic data provided a good fit to the PSO model, indicating that the removal operation mainly depended on chemical interactions. Thermodynamic work showed the biosorption was physical, favorable and spontaneous. The biosorbent exhibited high biosorption capacity for Mb. The results of this study provides a sustainable approach for effective wastewater treatment as well as other environmental remediation applications such as the valorization of waste biomass.

Author contributions Not Applicable.

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Data availability All data generated or analyzed during the study are included in the article.

Declarations

Ethical approval Not Applicable.

Use of generative AI and AI-assisted technologies There is no generative AI and AI-assisted technologies usage to declare.

Conflict of interest There is no conflict of interest to declare.

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