



Valorization of a cheap plant-based biomass for successful and sustainable removal of a common synthetic pollutant from water resource

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Abstract

In recent years, the production of value-added materials from plants has attracted great interest from scientists around the world. In this study, a novel biosorbent from *Prunus laurocerasus* leaves (*Pl*), an herbal waste, material that was eco-friendly and cheap, was successfully produced to remove synthetic dye (methylene blue, MB) from aqueous environment. Optimum bio-adsorption conditions were contact period of 360 min, solution pH of 8, biosorbent amount of 10 mg and MB concentration of 15 mg L⁻¹. Experimental biosorption profile was best fitted by Langmuir isotherm model (R^2 : 1.000, R^2_{adj} : 0.999, $RMSE$: 0.504), showing a bio-adsorption capacity of 70.870 mg g⁻¹, and Elovich kinetic model (R^2 : 0.981, R^2_{adj} : 0.980, $RMSE$: 2.693). Synthetic dye biosorption was a spontaneous, feasible and physical process. *Pl* exhibited a surface structure favorable for retention of synthetic dye. These remarkable experimental data indicate that *Pl* can be successfully used in field of removal of synthetic dyes from aqueous medium.

Keywords *Prunus laurocerasus* leaves · Value-added herbal material production · Bio-adsorption · Synthetic dye

1 Introduction

One of the biggest issues in today's world is undoubtedly environmental pollution. As a result of increasing industrial, agricultural and urban activities of human beings, disregarding nature, large amounts of pollutants have been released into natural environment. One of the most prominent of these pollutants is synthetic dyes. Synthetic dyes come in a wide variety and are produced in incredible quantities to meet increasing demand. Unfortunately, a significant amount of synthetic dyes is released into aquatic environment during the production and usage processes. These synthetic pollutants are substances that are not or have very difficult to biodegrade and have the ability to accumulate. They also have very harmful effects on organism, such as mutagenic, carcinogenic and even teratogenic activities [1, 2]. Due to these properties, synthetic dyes cause numerous

biological activity problems and even deaths in all biota, starting with living creatures in aquatic ecosystem.

Considering that millions of people around the world do not have access to clean water and adequate sanitation, the fact that water contaminated with such pollutants must be thoroughly purified and released into natural environment becomes clear. Synthetic dyes can be removed from aquatic medium by various methods, including ozonation, chemical precipitation, photocatalytic degradation, ion-exchange, membrane separation, and chemical oxidation processes. However, adsorption using green materials from nature, also called bio-adsorption or biosorption, is considered a promising method in synthetic dye purification due to its environmental friendliness, simplicity, low cost and high efficiency [3–5]. With this method offering a sustainable environmental solution to synthetic dye pollution, scientists have focused on the use of bio-adsorbents obtained from plant biomass-derived materials to replace traditional adsorbents in recent decades [6–8].

Prunus laurocerasus is a hardy species belonging to *Rosaceae* plant family. It is present in many countries around the world, especially in Türkiye. It has widespread use as a food, landscaping, decorative, medicinal and barrier plant. *P. laurocerasus* has a rich bioactive content

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and antimicrobial and antioxidant properties [9, 10]. Main objective of this study was to produce a novel biosorbent from *P. laurocerasus* leaves (*Pll*) to successfully remove synthetic dye (MB) from aquatic environment. Its high bio-active compound content indicates that it can be a successful biosorbent. Its above-mentioned properties also pave the way for this material to be used against different pollutants simultaneously. Optimum biosorption conditions were studied by evaluating several important factors influencing purification process. Experimental synthetic dye bio-adsorption profile of the biosorbent was investigated by isotherm, kinetic, thermodynamic and characterization studies.

2 Materials and methods

2.1 Production of *Pll*

Pll was supplied from Samsun province, Türkiye. *Pll* samples were washed with tap water and then with ultrapure water (Merck Direct-Q 3 UV) until no impurities were deposited on them. They were then dried in oven (Mikrotest MST 120D) for 24 h. Dried samples were ground with a grinder and sieved through 0.5 mm stainless steel sieve (Loyka). Powdered material was stored to be used in bio-adsorption experiment.

2.2 Preparation of MB sample

MB (Merck) was provided from a local medical company (Samsun province, Türkiye). Desired MB concentration was obtained by diluting stock MB solution (1000 mg L⁻¹). pH of experimental MB solution samples was set by adding NaOH and/or HCl (0.1 mol L⁻¹, Merck). pH value of samples was determined with a pH meter (Hach HQ40d). A standard curve was created by measuring absorbance of MB (663 nm) using spectrophotometer (Thermo Genesys 10 S) to determine MB concentration in samples.

2.3 Bio-adsorption experiment

Bio-adsorption study was conducted at ambient temperature (17 °C) under different operational conditions in 100 mL conical flasks containing MB solution. Different amounts of *Pll* were added to MB solution, which was prepared at different MB concentrations according to experimental conditions, and mix was agitated (orbital shaker, 150 rpm) for a certain period of time. When specified experimental time was reached, a certain amount of aqueous phase sample was separated from mix. MB concentration in sample was calculated by spectrophotometer. MB biosorption capacity (q_t , q_e , mg g⁻¹) (Eqs. 1 and 2) and removal yield (r_y , %) (Eq. 3) of *Pll* were determined using equations in Table 1.

2.4 Optimization of bio-adsorption conditions

Optimum working conditions were determined by evaluating several important factors influencing biosorption process such as contact period, pH, biosorbent amount and MB concentration. Experimental studies were conducted by keeping other variables constant while evaluating impact of a variable on bio-adsorption process.

2.5 Isotherm modeling

Relationship between MB amount retained on *Pll* (q_e) and MB concentration remaining in aqueous phase (C_e) in equilibrium at a constant temperature was modeled with Freundlich (Eq. 4), Langmuir (Eq. 5) and Dubinin-Radushkevich (D-R, Eq. 6) equations in Table 2. This table also presents equations for separation factor (R_L) (Eq. 7) and mean free energy (E , kJ mol⁻¹) (Eq. 8).

2.6 Kinetic modeling

Relationship between MB bio-adsorption ability of *Pll* (q_t) and contact period (t , min) was modeled with pseudo-first-order (pfo, Eq. 9), pseudo-second-order (pso, Eq. 10), Elovich (Eq. 11) and intra-particle diffusion (ipd, Eq. 12) equations in Table 3.

Table 1 Equations

Num.	Eq.
1	$q_t = \frac{(C_0 - C_t)V}{m}$
2	$q_e = \frac{(C_0 - C_e)V}{m}$
3	$r_y = \frac{C_0 - C_t}{C_0} \times 100$

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)

Table 2 Isotherm equations

Num.	Eq.	Ref.
4	$q_e = K_F C_e^{\frac{1}{n_F}}$	[11]
5	$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$	[12]
6	$q_e = q_m e^{-B\epsilon^2}$	[13]
7	$R_L = \frac{1}{1 + K_L C_0}$	[14]
8	$E = \frac{1}{\sqrt{2B}}$	[14]

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

Table 3 Kinetic equations

Num.	Eq.	Ref.
9	$q_t = q_e(1 - e^{-k_1 t})$	[15]
10	$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t}$	[16]
11	$q_t = \frac{1}{b} \ln(1 + abt)$	[17]
12	$q_t = C + k_p \sqrt{t}$	[18]

k_1 : pfo rate constant (min^{-1}), k_2 : pso rate constant ($\text{g mg}^{-1} \text{min}^{-1}$), a : initial bio-adsorption rate ($\text{mg g}^{-1} \text{min}^{-1}$), b : desorption constant (g mg^{-1}), C : a parameter about boundary layer thickness (mg g^{-1}), k_p : ipd rate constant ($\text{mg g}^{-1} \text{min}^{-1/2}$)

2.7 Thermodynamic modeling

Thermodynamic behavior of MB biosorption process was modeled using ΔG° (kJ mol^{-1}) (Eq. 13) equation in Table 4.

2.8 Study of characterization

Physiochemical features of *Pll* before and after MB bio-adsorption process were investigated by FTIR and SEM techniques. Functional group profile of *Pll* was studied using FTIR spectrometer (PerkinElmer Spectrum 400). FTIR spectrum was recorded in $4000\text{--}400 \text{ cm}^{-1}$ range. Morphological structure of material was studied by SEM (Zeiss Evo Ls 10) at a magnification range of $100\text{--}5000$ times.

2.9 Statistical analysis

Performance of models in representing experimental bio-adsorption data was assessed using SigmaPlot program (Systat) with R^2 (Eq. 14), adjusted R^2 (R^2_{adj}) (Eq. 15) and $RMSE$ (Eq. 16) statistical equations in Table 5.

3 Results and discussion

3.1 Optimization

3.1.1 Contact period

Impact of contact period on bio-adsorption process was studied in $0\text{--}360$ min range and results are shown in Fig. 1.

Table 4 Thermodynamic equation

Num.	Eq.	Ref.
13	$\Delta G^\circ = -RT \ln K_D$	[19]

R : gas constant ($\text{J mol}^{-1} \text{K}^{-1}$), T : temperature (K), K_D : distribution coefficient (C_{be}/C_e , -), C_{be} : equilibrium synthetic dye concentration on biosorbent (mg L^{-1})

Table 5 Statistical equations

Num.	Eq.
14	$R^2 = 1 - \frac{\sum_{i=1}^n (x - y)^2}{\sum_{i=1}^n (x - \bar{x})^2}$
15	$R^2_{\text{adj}} = 1 - \frac{(1 - R^2)(n - 1)}{n - p - 1}$
16	$RMSE = \sqrt{\frac{1}{n} \sum_{i=1}^n (x - y)^2}$

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

MB biosorption capacity of *Pll* enhanced with increasing contact period and equilibrium position was reached in 360 min. Initially, MB bio-adsorption increased gradually due to availability of numerous suitable binding sites on biosorbent material for retention of MB molecules. However, after a certain contact period, vacant binding sites were almost completely filled by MB molecules, and biosorption process reached equilibrium [2].

3.1.2 pH

Impact of pH on MB bio-adsorption by *Pll* was examined in range of pH $4\text{--}8$. Figure 2. displays pH effect on MB purification. Biosorption performance of *Pll* increased with enhancing pH. MB binding sites on *Pll* were more protonated at low pH and positive charges surrounded surface of biosorbent. Since MB is a cationic compound, competition between MB molecules and protons for binding sites limited retention of MB on *Pll*. As pH increased, binding sites became deprotonated and negatively charged. Thus, these sites created high electrostatic attraction fields for cationic MB molecules, thus increasing biosorption capacity of *Pll* [20].

3.1.3 Biosorbent amount

Effect of *Pll* amount on MB removal by biosorbent was investigated in range of $10\text{--}30$ mg and results are indicated in Fig. 3. MB bio-adsorption ability of biosorbent decreased with increase in *Pll* amount. As amount of biosorbent increased, the number of binding sites retaining MB molecules increased. However, since MB concentration did not change in aqueous medium, binding sites were not completely occupied by MB molecules, resulting in lower MB removal efficiency at higher biosorbent amounts. Another reason may be overlapping or agglomeration of biosorbent particles [21].

Fig. 1 Impact of contact period on biosorption process

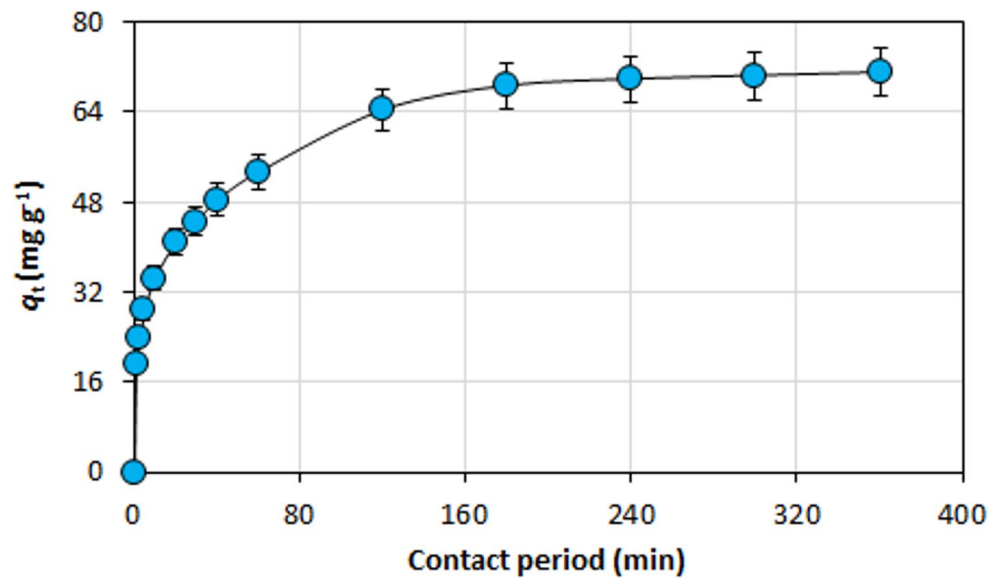
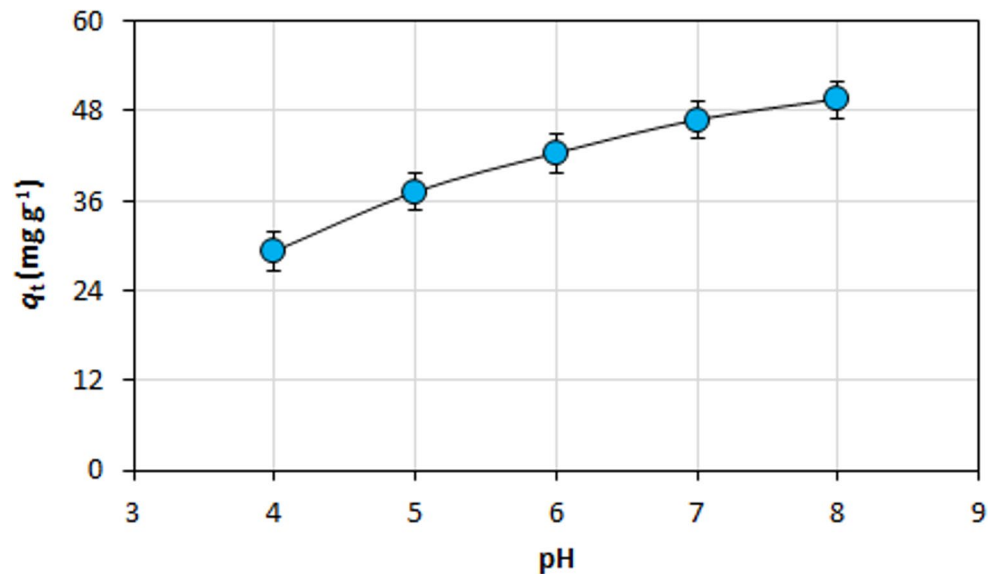


Fig. 2 pH effect on MB purification



3.1.4 MB concentration

Impact of MB concentration on bio-adsorption capacity of *Pll* was studied in 5–15 mg L⁻¹ range. Figure 4. shows impact of MB concentration on biosorption process. Removal yield of biosorbent increased with enhancing MB concentration. Higher driving force created by increasing concentration gradient with enhancing MB concentration provided more mass transfer of MB molecules to binding sites [22].

As a result, it was determined that optimum MB bio-adsorption conditions were contact period of 360 min, solution pH of 8, biosorbent amount of 10 mg and MB concentration of 15 mg L⁻¹.

3.2 Isotherm modeling

Experimental data obtained from MB biosorption by *Pll* were modeled with Freundlich, Langmuir and D-R equations and results are indicated in Table 6. Performance of models in representing experimental biosorption isotherm data was assessed using R^2 , R^2_{adj} and $RMSE$. These statistical parameters showed that experimental MB bio-adsorption profile of *Pll* was best fitted by Langmuir isotherm model (R^2 : 1.000, R^2_{adj} : 0.999, $RMSE$: 0.504), showing a monolayer biosorption capacity of 70.870 mg g⁻¹. R_L for MB bio-adsorption was determined in 0.00103–0.00308 range. These values between 0 and 1 showed that MB biosorption by biosorbent was a feasible process. For MB removal, n_F parameter

Fig. 3 Effect of biosorbent amount on MB removal by *Pll*

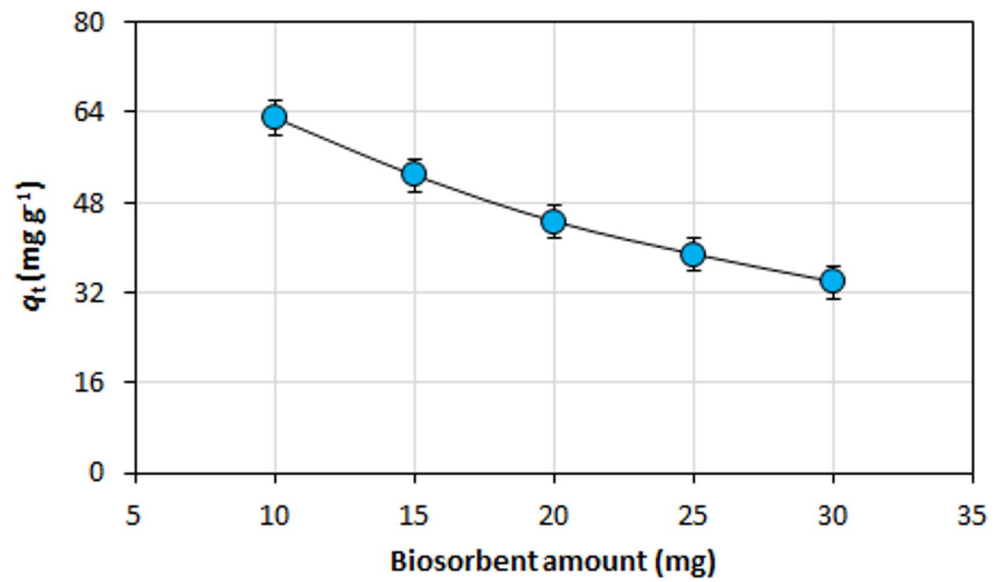


Fig. 4 Impact of MB concentration on bio-adsorption process

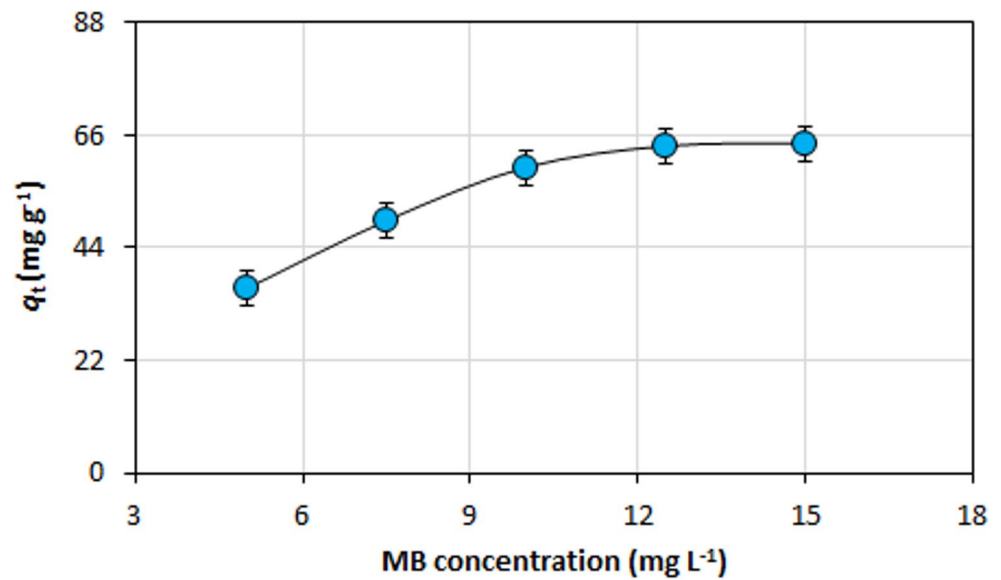


Table 6 Isotherm modeling

Model	MB con. (mg L ⁻¹)	Parameter				
Freundlich		K_F	n_F	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹ (L mg ⁻¹) ^{1/n_F}	-	-	-	-
Langmuir		62.762	12.706	0.730	0.460	12.718
		q_m	R_L	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	-	-	-	-
	5	70.870	0.00308	1.000	0.999	0.504
	10		0.00154			
	15		0.00103			
D-R		q_m	E	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	kJ mol ⁻¹	-	-	-
		72.326	8.771	0.991	0.981	2.392

Table 7 Kinetic modeling

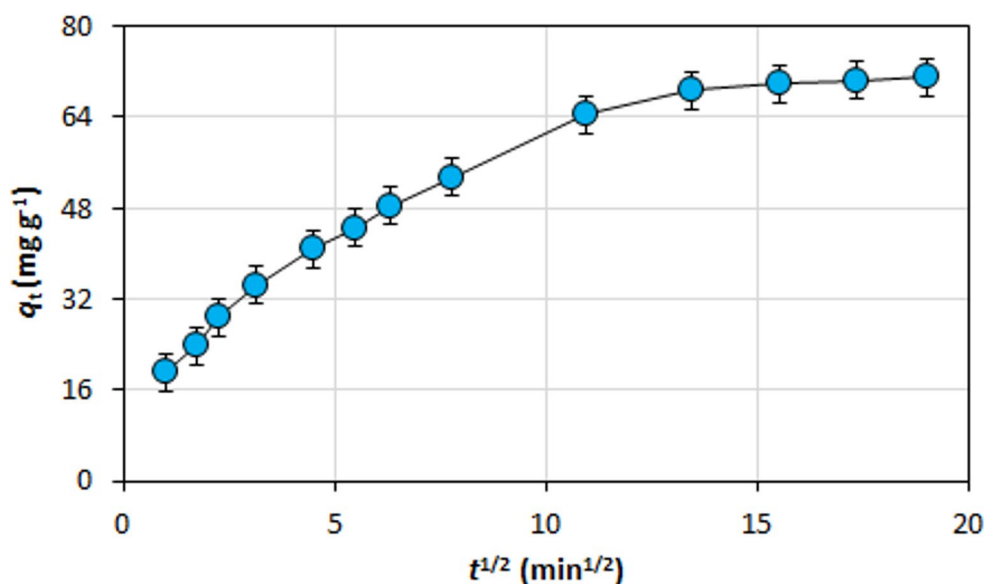
Model	MB con. (mg L ⁻¹)	Parameter				
pfo		q_e	k_1	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	min ⁻¹	-	-	-
	5	33.129	0.361	0.504	0.459	5.025
	10	54.928	0.120	0.509	0.464	11.127
pso	15	64.948	0.058	0.764	0.742	9.555
		q_e	k_2	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	g mg ⁻¹ min ⁻¹	-	-	-
	5	34.884	0.01494	0.747	0.724	3.588
Elovich	10	59.799	0.00264	0.728	0.703	8.278
	15	69.027	0.00145	0.889	0.879	6.543
		a	b	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹ min ⁻¹	g mg ⁻¹	-	-	-
ipd	5	3.605	0.277	0.990	0.989	0.732
	10	7.834	0.128	0.944	0.939	3.743
	15	9.893	0.101	0.981	0.980	2.693
		C	k_p	R^2	R^2_{adj}	$RMSE$
		mg g ⁻¹	mg g ⁻¹ min ^{-1/2}	-	-	-
	5	21.782	1.029	0.883	0.872	2.440
	10	26.253	2.359	0.937	0.932	3.978
	15	25.093	2.877	0.908	0.900	5.950

was determined as 12.706. $1/n_F$ (0.08) value in range of 0–1 indicated that a favorable bio-adsorption process occurred. E parameter for MB biosorption was determined as 8.771 kJ mol⁻¹. This value, which was slightly greater than 8 kJ mol⁻¹, showed that MB removal by *Pll* could be achieved by both a chemical and physical process [14, 23, 24].

3.3 Kinetic modeling

Experimental data obtained for MB bio-adsorption were modeled with pfo, pso, Elovich and ipd equations. Results of kinetic modeling are shown in Table 7. Performance

of models in representing experimental MB biosorption kinetic data was evaluated with R^2 , R^2_{adj} and $RMSE$. Statistical results indicated that experimental MB biosorption profile of *Pll* was best fitted by Elovich kinetic model (R^2 : 0.981, R^2_{adj} : 0.980, $RMSE$: 2.693), indicating chemical interactions between MB molecules and binding sites of biosorbent heterogeneous surface [20, 25]. Effect of ipd step on MB bio-adsorption is shown in Fig. 5. It was observed that ipd plot obtained for MB biosorption was non-linear and did not pass through origin. These results indicated that mechanisms other than ipd may also be effective in MB bio-adsorption [26].

Fig. 5 Effect of ipd step on MB biosorption

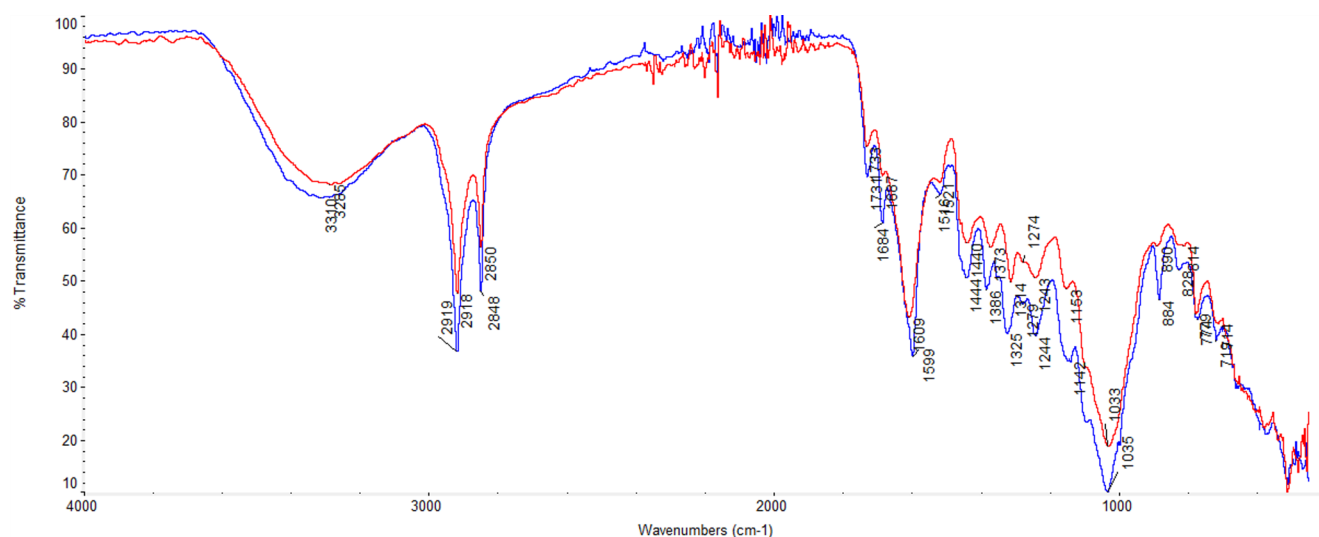


Fig. 6 FTIR spectra of *PII* before (red) and after (blue) MB bio-adsorption

3.4 Thermodynamic modeling

Thermodynamic evaluation of MB bio-adsorption by *PII* was performed using ΔG° parameter. ΔG° for MB biosorption were determined in -18.472 – 5.364 kJ mol $^{-1}$ range. Negative ΔG° values mean that MB biosorption on biosorbent was favorable and spontaneous. In addition, negative ΔG° values determined for MB removal were > -20 kJ mol $^{-1}$, indicating that bio-adsorption process was due to physisorption mechanism [27].

3.5 Characterization

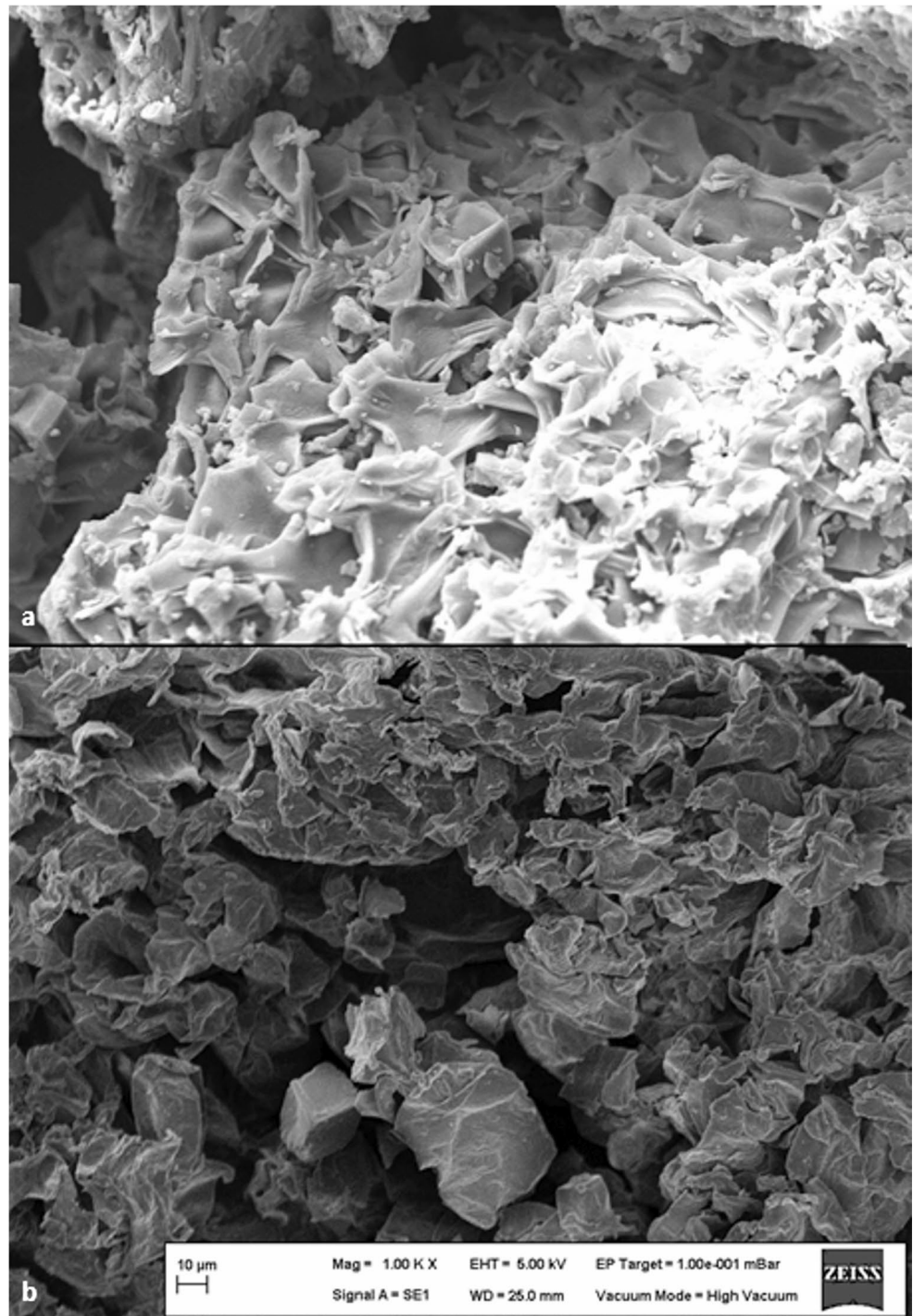
Physiochemical features of *PII* before and after MB bio-adsorption were investigated by FTIR and SEM techniques. Functional group profile of *PII* was studied using FTIR spectrometer. FTIR spectra of biosorbent before (red) and after (blue) MB biosorption are shown in Fig. 6. In addition, changes in the spectral positions of FTIR bands are displayed in Table 8. For unloaded biosorbent (red), broad band at 3285 cm $^{-1}$ referred to O-H chemical group. Sharp bands at 2918 and 2850 cm $^{-1}$ were attributed to C-H bond [28–30]. Bands at 1733 , 1687 and 1609 cm $^{-1}$ corresponded to C = O and C = C bonds [31, 32]. Small bands at 1521 , 1440 , 1373 , 1314 , 1274 , 1243 and 1153 cm $^{-1}$ were assigned to C-O-C, O-H, C-H, C-O, C = C and C-N chemical bonds [26, 30]. Strong band at 1033 cm $^{-1}$ was originated from bond of C-O-C. Finally, small bands at 890 , 814 , 779 and 714 cm $^{-1}$ were associated with C-O and C-H bonds [2, 26, 33]. Morphological changes in FTIR spectrum of *PII* after MB removal (blue) indicated that different chemical binding sites on biosorbent were involved in MB bio-adsorption.

Morphological structure of *PII* was studied by SEM and SEM pictures of biosorbent before and after MB bio-adsorption are displayed in Fig. 7. It appears that *PII* has a rough superficial structure containing cavities of varying diameters (Fig. 7a). This morphological structure can provide a good advantage for practical use of biosorbent in MB biosorption as it would serve as a binding site for MB molecules [19]. Morphological changes in SEM image of *PII* after MB biosorption (Fig. 7b) showed that MB molecules were retained on surface.

Table 8 Changes in spectral positions of FTIR bands

Wavenumbers (cm $^{-1}$)	
Before	After
3285	3310
2918	2919
2850	2848
1733	1731
1687	1684
1609	1599
1521	1516
1440	11,444
1373	1386
1314	1325
1274	1279
1243	1244
1153	1142
1033	1035
890	884
814	828
779	777
714	717

Fig. 7 SEM pictures of *Pll* before (a) and after (b) MB biosorption



3.6 Comparison

A lot of studies have been conducted on MB removal. q_m value obtained from Langmuir isotherm model was used to investigate efficiency of biosorbents in this study. Table 9 shows comparison of MB removal from aqueous

medium using *Pll* and other biosorbents. Although *Pll* was not subjected to an activation process, it has a very good MB removal capacity compared to other biosorbents. Therefore, it can be easily said that this biosorbent has a significant potential in removal of MB from aquatic environment.

Table 9 MB removal efficiency of biosorbents

Biosorbent	q_m (mg g ⁻¹)	Refer- ence
Alkaline-activated <i>Detarium senegalense</i> fruit hulls	96.453	[2]
Hibiscus plant waste	35.34	[34]
Hibiscus plant waste-based charcoal	99.01	[34]
<i>Wodyetia bifurcata</i> leaves	58.74	[35]
Brine-activated bamboo stalk	16.32	[36]
<i>Gracilaria bursa-pastoris</i>	8.3612	[37]
<i>Eichhornia carassipes</i> -based biochar	98.57	[38]
<i>Astrocaryum aculeatum</i> seeds	26.8721	[39]
NaOH-treated <i>Astrocaryum aculeatum</i> seeds	37.8105	[39]
H ₃ PO ₄ -treated <i>Astrocaryum aculeatum</i> seeds	23.9887	[39]
<i>Pll</i>	70.870	This study

4 Conclusions

A novel biosorbent from *P. laurocerasus* leaves was successfully produced to remove MB from aqueous medium. Optimum removal conditions were contact period of 360 min, pH of 8, biosorbent amount of 10 mg and MB concentration of 15 mg L⁻¹. Experimental MB removal data was best fitted by Langmuir isotherm model, showing a bio-adsorption capacity of 70.870 mg g⁻¹, and Elovich kinetic model. MB biosorption was a spontaneous, feasible and physical process. *Pll* exhibited a surface structure favorable for biosorption of MB molecules. Results support valorization of cheap plant-based biomasses for successful and sustainable removal of common synthetic pollutants such as MB from water resources. For field-based use, further research is needed on regeneration, continuous system, actual wastewater, different pollutants, stability, life cycle, scalability, and cost.

Author contributions Not Applicable.

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

Declarations

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

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