Universidad Tecnológica Nacional
MAYO - AGOSTO 2026 / Año 24 - Nº 56

Revista Tecnología y Ciencia
DOI: https://doi.org/10.33414/rtyc.56.147-162.2026 - ISSN 1666-6933

Use of *Moringa oleifera* residues as a raw and activated adsorbent for the removal of crystal violet in aqueous solution

Uso de residuos de *Moringa oleífera* como adsorbente crudo y activado para la eliminación de violeta cristal en solución acuosa

Presentado: 22/01/2026

Aprobado: 22/07/2026

Publicado: 25/08/2026

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Revista Tecnología y Ciencia
DOI: https://doi.org/10.33414/rtyc.56.147-162.2026 - ISSN 1666-6933

Abstract

The persistence, toxicity, and low biodegradability of synthetic dyes in aquatic systems constitute a relevant environmental challenge. This study evaluated the adsorption of crystal violet (CV) in aqueous solution using *Moringa oleífera* trunk waste in its raw state (Tr) and after activation as carbon (CAT). The materials were characterised by determining their point of zero charge (pHpzc) and by scanning electron microscopy (SEM). Adsorptive performance was analyzed using Langmuir and Freundlich isotherms and pseudo-first- and pseudo-second-order kinetic models. The results showed greater capacity and efficiency in CAT. Tr kinetics fitted the pseudo-second-order model, while CAT did not provide a conclusive fit. Activated *Moringa oleífera* is proposed as a sustainable and efficient adsorbent for the removal of cationic dyes in water.

keywords: adsorption, *Moringa oleifera*, activated carbon, crystal violet.

Resumen

La persistencia, toxicidad y baja biodegradabilidad de colorantes sintéticos en sistemas acuáticos constituyen un desafío ambiental relevante. Este estudio evaluó la adsorción del violeta cristal (CV) empleando biomasa de tronco de *Moringa oleífera* en estado crudo (Tr) y tras activación química como carbón (CAT). Las propiedades superficiales se caracterizaron mediante punto de cero carga y microscopía electrónica de barrido. El desempeño adsortivo se analizó con isotermas de Langmuir y Freundlich y modelos cinéticos de pseudo-primer y pseudo-segundo orden. Los resultados evidenciaron mayor capacidad y eficiencia en CAT. La cinética de Tr se ajustó al modelo de pseudo-segundo orden, mientras que para el CAT no se obtuvo un ajuste concluyente. La *Moringa oleífera* activada se propone como adsorbente sostenible y eficiente para la remoción de colorantes catiónicos en agua.

Palabras claves: adsorción, *Moringa oleífera*, carbón activado, violeta cristal.

Introduction

The contamination of aquatic environments by synthetic dyes, such as crystal violet, has become a pressing environmental and public health issue due to their high toxicity, persistence, and limited biodegradability. Major industrial contributors include the textile, cosmetic, leather, food, pharmaceutical, paint, varnish, pulp, and paper sectors (Dutta et al., 2021). Effluents from these industries typically exhibit high color loads and elevated biological oxygen demand, both of which impair water quality and hinder aquatic photosynthesis. In addition, such pollutants can exert severe toxic effects on aquatic organisms and pose health risks to humans (Jaramillo et al., 2013). Among the various treatment strategies developed for dye removal, adsorption has emerged as one of the most effective and versatile approaches (Arthy & Saravanakumar, 2013). Within the framework of a new economic paradigm, in which the circular economy plays a central role in addressing environmental challenges, agro-industrial residues have attracted increasing attention as precursors for the development of low-cost adsorbents. Among these residues, *Moringa oleífera* has particular relevance. This species, which has been expanding in northeastern Argentina, was officially approved in 2018 for use in infusions (Secretaría De Regulación Y Gestión Sanitaria Y Secretaría De Alimentos Y Bioeconomía, 2019); this approval has promoted its consumption

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and, consequently, its large-scale cultivation.

The production of moringa leaves for infusions, together with the processing of other derivatives, generates considerable quantities of solid by-products, including bark, shells, and trunks, which account for approximately 80% of the plant biomass and are typically discarded (Leibaschoff, 2024). These residues are characterised by their high lignocellulosic content (Ochoa Torres et al., 2026) and wide local availability (Province of Misiones, Argentina), making them promising candidates for valorisation through adsorption applications or conversion into activated carbon. Its use would not only add economic value to the cultivation of moringa, but would also provide a sustainable and locally accessible approach to the remediation of industrial effluents contaminated with synthetic dyes. From a circular economy perspective, the conversion of large volumes of contaminated liquid effluents into more easily managed solid phases (contaminated moringa biomass) represents an important step toward effective pollution control and long-term environmental solutions.

In this context, the present study investigates the adsorption capacity of *Moringa oleífera* trunk in both its raw and activation as carbon forms for the removal of crystal violet from aqueous solutions. The materials were characterised by point of zero charge (pHpzc) and scanning electron microscopy (SEM). Adsorption performance was evaluated using equilibrium isotherm models (Langmuir and Freundlich) and kinetic models (pseudo-first and pseudo-second order) with the aim of establishing their feasibility as sustainable and low-cost biosorbents for wastewater treatment.

Materials and Methodology

Moringa oleífera residues were provided by the company "El Moringuero S.A." and initially subjected to sun-drying. The material was then size-reduced using a milling process and subsequently sieved through a 3.35 mm mesh to obtain the raw trunk fraction (Fig. 1). Based on this material, two adsorbents were prepared: the untreated trunk residue (Tr) and the activated carbon derived from it (CAT).

Fig. 1: *Moringa oleifera* residue

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The Tr material was washed five times with running water at 100°C for 10 minutes per cycle, followed by two additional washes with distilled water under the same conditions (Torre et al., 2021). After washing, the residue was oven-dried at 60°C for 24 hours and then sieved through 0.850 mm and 0.500 mm meshes to obtain a standardized particle size (Boeykens et al., 2018). To obtain CAT, the raw material was chemically impregnated with a solution of 50% w/w phosphoric acid (H_3PO_4, Biopack®) at an acid-to-precursor mass ratio of 2:1, followed by thermal treatment in an oven at 110°C for 2 hours. The impregnated material was then carbonized in a muffle furnace at 400°C for 1 hours following a previously published procedure (de Celis et al., 2009). Finally, the activated carbon was oven-dried at 60°C for 24 hours and then sieved through 0.850 mm and 0.500 mm meshes.

Adsorbate:

A 2.451mmolL^{-1} stock solution of Cicarelli® Crystal Violet (CV) was used, and the determinations were performed with a PerkinElmer® Lambda 365+ UV/VIS Spectrometer. The maximum wavelength of light absorbed (λ=600nm) was obtained by means of spectral scanning.

Characterization of adsorbent materials:

Point of zero charge: The zero charge point (ZCP) is defined as the pH at which the net surface charge of the particles becomes zero (Nasiruddin Khan & Sarwar, 2007).

The PZC was determined using the salt addition method. For this purpose, 0.1000 g of adsorbent was placed in contact with 50.0 mL of 0.10 M NaCl (Biopack) at different pH values (from 4 to 10). The mixture was stirred at room temperature for 24 hours using an orbital shaker (LCD® – HS-AJ-0660-PRO) at 200 RPM. The samples were then filtered, and the pH of the final solution was determined. The 0.1M NaCl solutions were previously adjusted to the appropriate pH using 0.10 M NaOH (Biopack) or 0.10 M HCl (Cicarelli) solutions, as applicable. The pH of the initial and final solutions was determined using a Hanna® 221 pH meter, calibrated with Biopack® brand buffer solutions at pH 4 and pH 7.

All determinations were performed in duplicate. To determine the PZC, the ΔpH = final pH – initial pH vs. initial pH was plotted. The PZC corresponds to the initial pH at which the resulting line passes through the ordinate = 0

Scanning electron microscopy (SEM) was employed to obtain images of each material under investigation. The present study was conducted using a Zeiss Gemini 560 scanning electron microscope.

Adsorption dosage and equilibrium study:

In order to estimate the optimal mass to be used in the adsorption equilibrium tests, the adsorption dosage was evaluated. The percentage of removal (%R) and the adsorption capacity (q_e) of each contaminant were evaluated using different mass of adsorbent material and applying Equations 1 and 2 in Table 1:

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Parameters	Equation
%Removal	$\%\text{Removal} = \frac{(C_i - C_e)}{C_i}.100$ (1)
Adsorption Capacity	$q_e = \frac{(C_i - C_e).V}{m_{ads}}$ (2)

Table 1. Adsorption dosage equations

In this equation, Ci and Ce represent the initial and equilibrium concentrations (mmolL^{-1}), respectively. V is the volume (L) and mads is the mass of the adsorbent (g) (Boeykens et al., 2017).

The dosage experiment was conducted with continuous stirring, using an orbital shaker (LCD® – HS-AJ-0660-PRO), at 200RPM and a temperature of 30 ± 2°C, with the natural pH of the analyzed system (pH = 6.5 ± 0.2) maintained, for 24 hours. After removing the system from its operational environment and allowing the adsorbent to settle, the fluid was separated using a Pasteur pipette. The equilibrium dye concentration was finally determined for each analyzed sample using the supernatant. The experiments were conducted in triplicate. The contaminant concentration was found to be 0.7 mmol.L-1 of VC, with a range of 100-600 mg of adsorbent.

In order to evaluate the adsorption equilibrium, 100 mg of each adsorbent material was brought into contact with 50.0 mL of VC solutions with initial concentrations ranging from 0.23 to 1.10 mmol.L^{-1}, under controlled working conditions at temperatures of 10, 20, 30, and 40°C. The experimental data were fitted to the Langmuir (Langmuir, 1918) (equation 3) and Freundlich (Freundlich, 1907) (equation 4) adsorption models, as described in Table 2, using Origin 8.0®.

Models	Equation
Langmuir	$q_e = \frac{Q_m.b.C_e}{1 + b.C_e}$ (1)
Freundlich	$q_e = K_f.C_e^{\left(\frac{1}{n}\right)}$ (2)

Table 2. Adsorption isotherm models

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Where q_e is the amount of dye adsorbed on the adsorbent (mmol.g^{-1}); Ce is the concentration of the VC solution at equilibrium (mmol.L^{-1}); Qm is the adsorption capacity of the monolayer (mmol.g^{-1}); b is the Langmuir constant related to the free energy of adsorption; and K_f and n are the Freundlich constants.

Adsorption kinetics study:

The kinetics of the processes were evaluated by placing 100 mg of the adsorbent material in contact with 50.0 mL of VC solution with an initial concentration of 0.56 mmol.L^{-1}. Contact time was varied to analyze the process behavior over time at the dye's natural pH (6.5 ± 0.2) with continuous stirring, using an orbital shaker (LCD® – HS-AJ-0660-PRO), at 200RPM. The experimental tests were conducted at temperatures ranging from 10°C to 40°C, inclusive. The data were fitted to pseudo-first-order (Lagergren, 1898) (equation 5) and pseudo-second-order (Ho & Mckay, 1999) (equation 6) kinetic models, as described in Table 3, to determine the predominant adsorption process mechanism.

Models	Equation
Pseudo – 1st order	$\frac{dq_t}{dt} = k_1 \left(q_e - q_t \right)$ (5)
Pseudo – 2nd order	$\frac{dq_t}{dt} = k_2 \left(q_e - q_t \right)^2$ (6)

Table 3. Equations of the pseudo-first-order and pseudo-second-order kinetic models.

Where q_t is the adsorption capacity of the adsorbate in mmol.g^{-1}, t is the time in minutes, and K_1 and K_2 are the pseudo first and second order rates, respectively.

Results and discussion

Characterization of adsorbent materials:

Point of zero charge (PZC): The pH difference (ΔpH) between the final pH (pH_f) and the initial pH (pH_i) was plotted against the pH_i values. The PZC corresponded to the point at which the curve intersected the zero axis of the abscissa in Fig. 2. This resulted in values of 5.85 (red) for Tr and 3.92 (blue) for CAT. The working pH for all tests was 6.5 ± 0.2, higher than the PZCs of both materials. Therefore, both Tr and CAT were negatively charged under the conditions of this study. As VC dye is cationic, both adsorbent materials have the potential to adsorb it. This is because the surface of the solids will have a positive charge at pH values below the PZC and a negative charge at higher pH values. Therefore, adsorbents with low PZC values are compatible with cationic dyes, while those with high PZC values are more suitable for capturing anionic dyes.

It should be noted that the relatively low PZC values obtained for both materials were to be expected, given their lignocellulosic nature and the predominance of negatively charged functional groups on their surface, such as hydroxyl, carboxyl and phenolic groups. As pointed out by other authors (Ochoa Torres et al., 2026), this type of material has a predominantly

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acidic surface, which translates into low PZC values. The activation with phosphoric acid process increases the surface acidity of the CAT, consequently lowering its pHpzc compared to the precursor material (Raji et al., 2023). Furthermore, the pKa of the CV is approximately 0.8. This implies that at the pH values tested in this study (pH 6.5), the dye is fully ionized in its cationic (positive) form, while the CAT surface, having a low pHpzc, is strongly deprotonated, favoring CV adsorption. Similar behavior has been reported for other lignocellulosic adsorbents, such as banana peel, with PZC in comparable ranges (Piol et al., 2021), as well as for different activated carbons obtained from biomass (Guilhen et al., 2022).

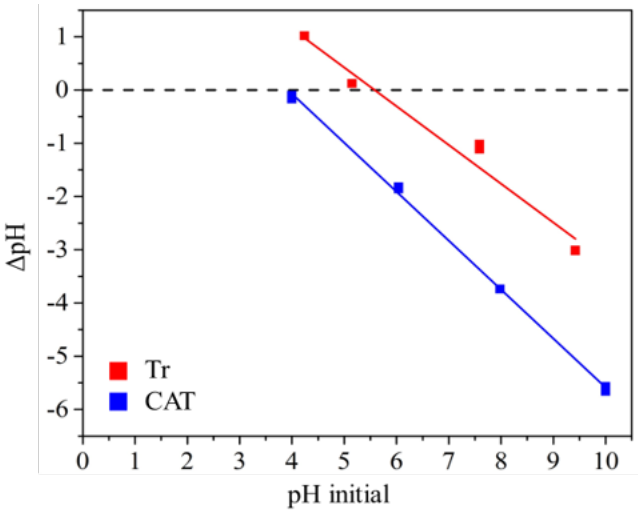

Fig. 2. Determination of the point of zero charge (pHpzc) of *Moringa oleífera* trunk residue (Tr) and activated carbon (CAT).

Images obtained by SEM of the raw *Moringa oleífera* trunk (Fig. 3a and 3b) show an irregular and heterogeneous surface with fibrous structures characteristic of lignocellulosic material. At low magnification (62x), a compact morphology with areas of rough texture can be seen. At higher magnification (372x), finer details of the surface structure can be observed, with unevenly distributed cavities and depressions, though there is no well-defined porosity.

The images of activated carbon (Figure 3c and d) obtained at 400x and 100x magnification show a significant transformation in the surface structure of the material compared to the raw log. The surface of the activated carbon is much rougher and more fractured, with multiple cavities, cracks, and pores apparent. These pores are distributed heterogeneously and vary in size, suggesting a significant increase in the material's specific surface area. This characteristic is key to its efficiency as an adsorbent as it provides more active sites for interacting with contaminant molecules.

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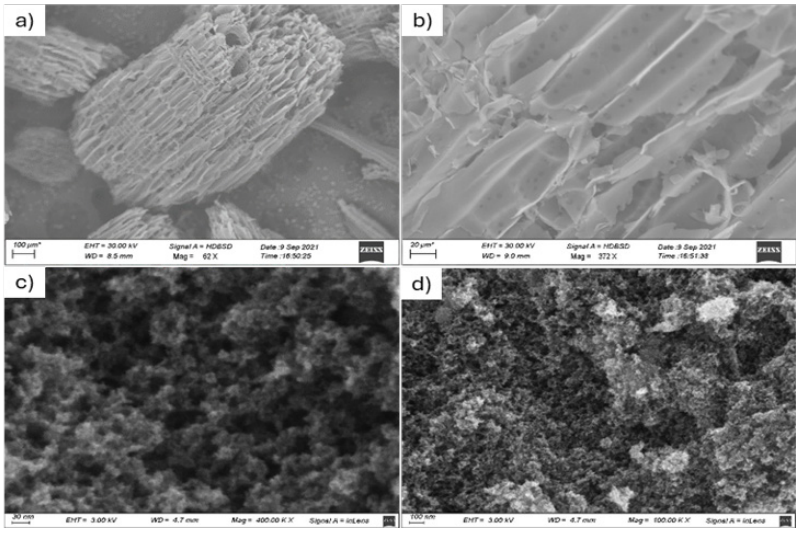

Fig. 3. SEM micrographs of *Moringa oleifera* trunk residue (Tr) at magnifications of a) 62X and b) 372X, and activated carbon (CAT) at magnifications of c) 400X and d) 100X.

Adsorption dosage and equilibrium study:

Fig. 4 shows the mass dosage curves of the adsorbent as a function of percentage removal and the adsorption capacity of VC per gram of material (q_e). Figure 4a corresponds to the Tr material and Figure 4b to the CAT material.

For Tr, increasing the dosage leads to an increase in removal, from 56% to 97%, accompanied by a decrease in q_e, from 0.20 to 0.06 mmol.g^{-1}. In contrast, removal in CAT exceeds 99% for all analyzed masses, while qe decreases from 0.35 to 0.06 mmol.g^{-1}. This indicates greater adsorption efficiency compared to Tr.

The analysis indicates that removal was never less than 90% in CAT. Therefore, masses of less than 0.1000 g are suggested for isothermal tests; however, very low values could increase experimental errors, which is why 0.1000 g was selected for subsequent tests. For Tr, 80% removal is achieved with 0.2000 g, considered the optimal mass for future studies. However, 0.1000 g was used in order to standardize the mass of adsorbent.

It is important to note that the process of obtaining CAT from Tr had a yield of 40%, meaning that 40.00 g of CAT were obtained from every 100.0 g of Tr.

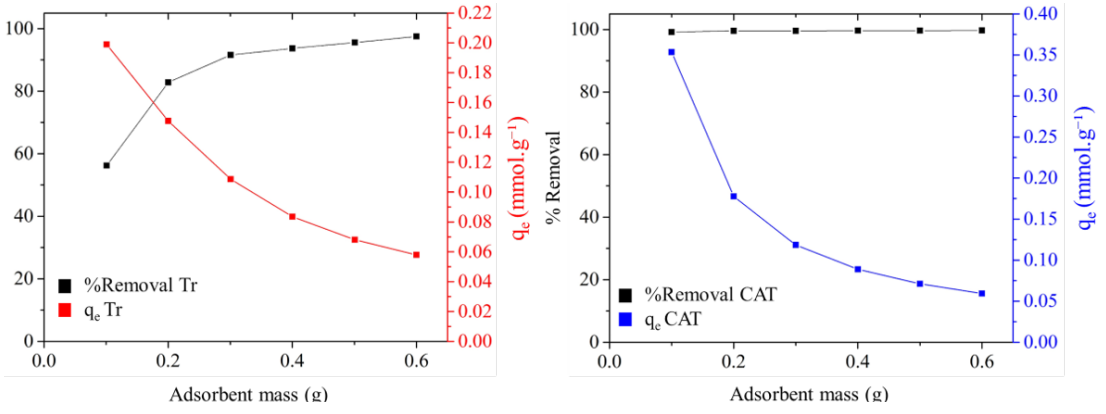

Fig. 4. Effect of adsorbent dosage on the removal of crystal violet (CV): a) raw *Moringa oleifera* trunk residue (Tr) and b) activated carbon (CAT).

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Fig. 5 shows the equilibrium adsorption capacity (q_e) versus the VC concentration (C_e) at equilibrium for material Tr, together with the Langmuir (red line) and Freundlich (blue line) models fits at the four evaluated temperatures. The dotted lines indicate the 95% confidence intervals for each model. Fig. 6 shows the same graphs for the CAT adsorbent material.

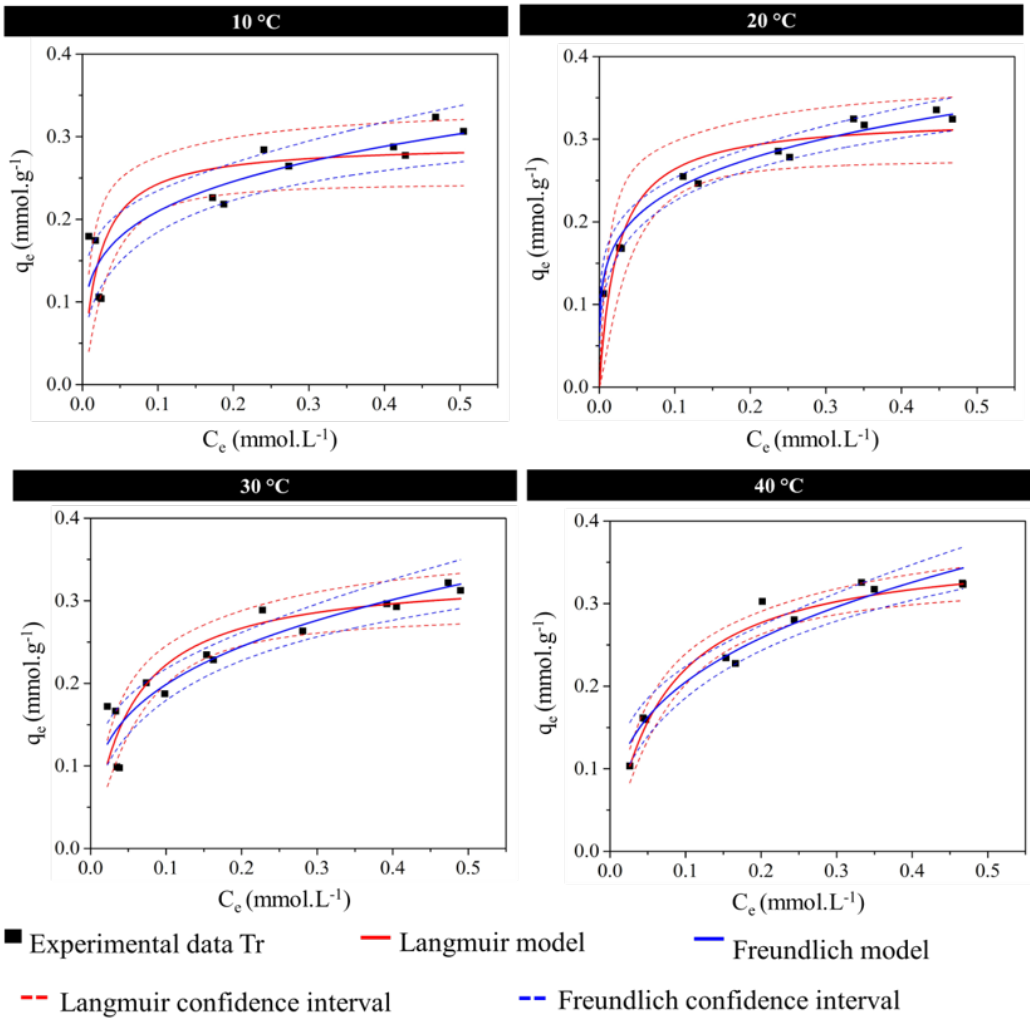

Fig. 5. Adsorption isotherm fitting of crystal violet (CV) onto raw *Moringa oleífera* trunk (Tr) at different temperatures.

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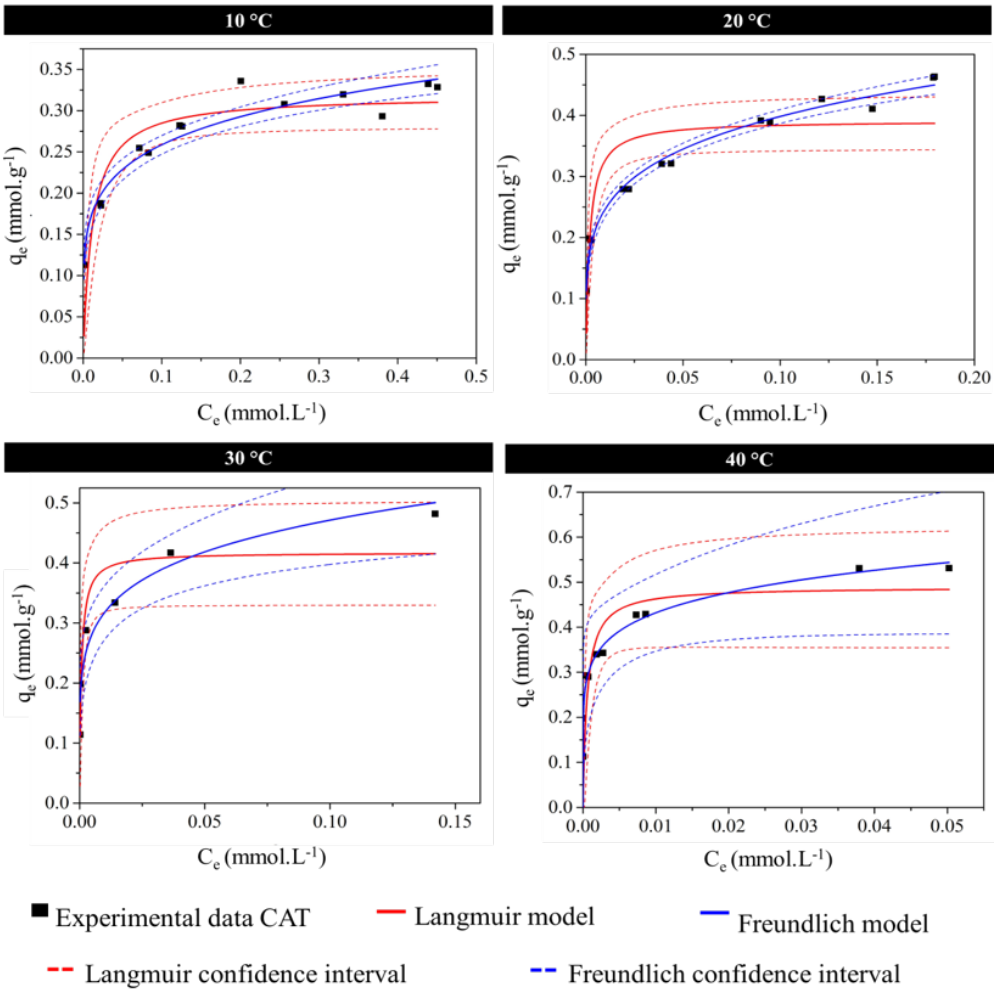

Fig. 6. Adsorption isotherm fitting of crystal violet (CV) onto activated carbon from *Moringa oleífera* trunk (CAT) at different temperatures.

Table 4 shows the parameters obtained by fitting the Langmuir and Freundlich models to the experimental data for the adsorption of VC on Tr and CAT at different temperatures.

Analysis of the correlation coefficients (R^2) and homocedasticity shows that, for Tr, the Freundlich model offers a better fit at low temperatures, while at 30 and 40 °C, both models have similar behaviour. In the Freundlich model, the Kf parameter increases with temperature, indicating greater adsorption capacity, while the n parameter decreases, reflecting lower process intensity. Similarly, in the Langmuir model, the Q_m parameter increases with temperature, which supports the endothermic nature of the process. Meanwhile, b decreases, which can be interpreted as a reduction in the affinity of VC for the Tr surface at higher temperatures. These results align with those reported by (Alghamdi et al., 2024) on manganese removal using *Moringa oleífera* seeds, where both models exhibited comparable fits, as well as with the findings of (Abbas et al., 2021;

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Tenev et al., 2019), who observed a superior fit to the Freundlich model for VC removal using peanut shells. However, it was found that the Langmuir model better described the adsorption of cationic dyes structurally similar to VC onto lignocellulosic materials from industrial waste. For the CAT material, the Freundlich model provided the best fit at all temperatures, as it produced higher R^2 values. In this case, both Kf and n increase with temperature, indicating a more efficient process at higher temperatures. Similarly, in the Langmuir model, the Q_m and b parameters also increased with temperature, supporting the hypothesis of an endothermic process. The value of n obtained at 30<°C was greater than 5, representing highly favorable adsorption and exceeding values reported for other adsorbents, such as peanut shells (n = 2.14; (Abbas et al., 2021)) and zeolite (n = 1.50; Sarabadan et al., 2019). This value is comparable to that of Merck activated carbon (n = 5.00; Sarabadan et al., 2019). Similarly, Raji et al. (2023) reported that *Moringa oleífera* -derived activated carbon best fit the Langmuir model, demonstrating that the nature of the material determines the model that most accurately describes adsorption.

Overall, the results suggest that the adsorption of VC onto Tr and CAT is favored at high temperatures, and while both models can describe the phenomenon, the Freundlich model provides the best fit, particularly for CAT. This indicates heterogeneity in the adsorbing surface and greater retention capacity under high thermal conditions.

Materia	Temperature (°C)	Langmuir model			Freundlich model		
		Q_m (mmolg^{-1})	b (Lmmol^{-1})	R^2	K_f	n	R^2
Tr	10	0.29	49.20	0.61	0.36	4.35	0.80
	20	0.33	41.71	0.73	0.39	4.78	0.94
	30	0.33	18.39	0.81	0.40	3.33	0.86
	40	0.37	14.65	0.94	0.44	2.99	0.93
CAT	10	0.32	85.27	0.74	0.39	5.60	0.95
	20	0.39	504.06	0.77	0.64	4.80	0.98
	30	0.44	726.21	0.81	0.69	5.90	0.81
	40	0.48	1818.46	0.89	0.77	7.64	0.92

Table 4. Summary of adsorption isotherm model parameters for crystal violet (CV) removal using raw trunk residue (Tr) and activated carbon (CAT).

Adsorption kinetics study:

Fig. 7 and 8 show the adsorption curves of VC onto Tr and CAT vs time, at different temperatures. These curves have been adjusted to pseudo-first-order and pseudo-second-order kinetic models, with 95% confidence intervals. Equilibrium times in the VC–Tr system were close to 200 minutes at all evaluated temperatures, while the VC–CAT system required approximately 400 minutes, indicating a slower process.

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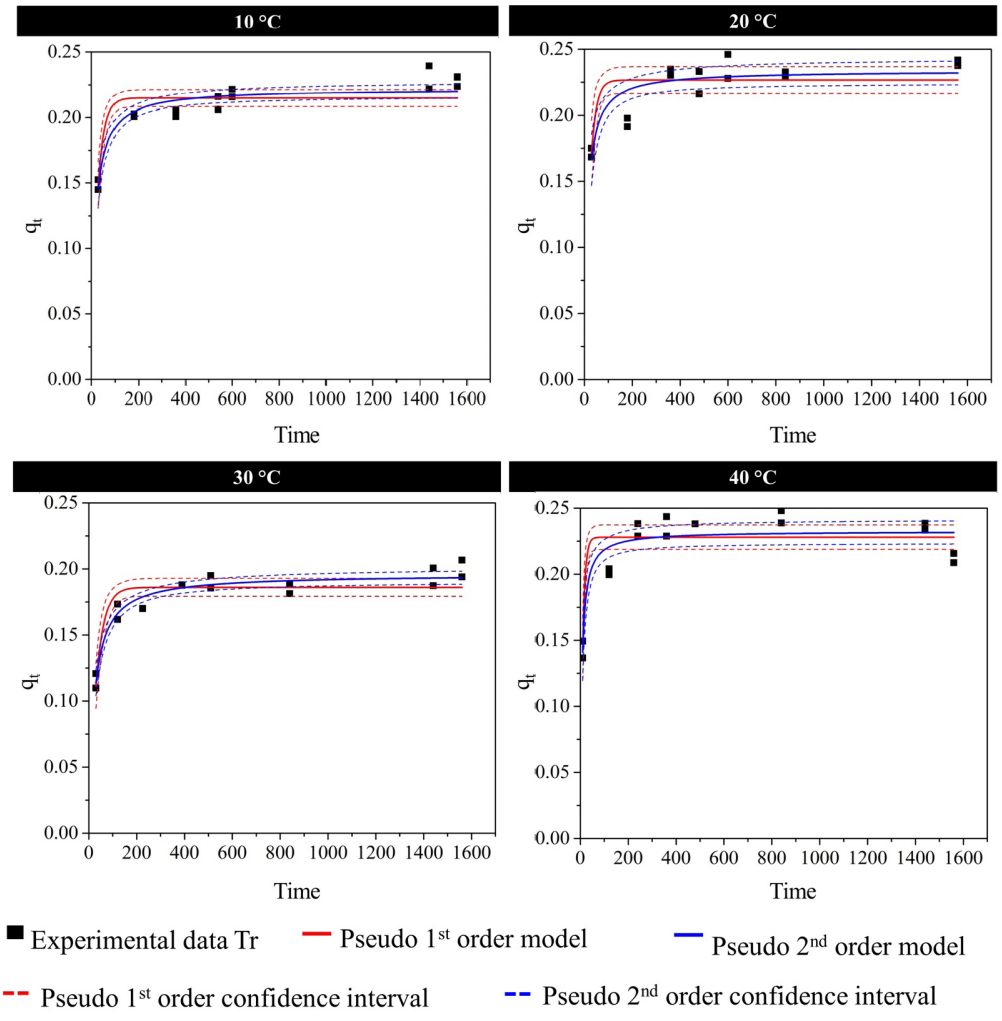

Fig. 7. Adsorption kinetics of crystal violet (CV) onto raw *Moringa oleífera* trunk residue (Tr) at different temperatures.

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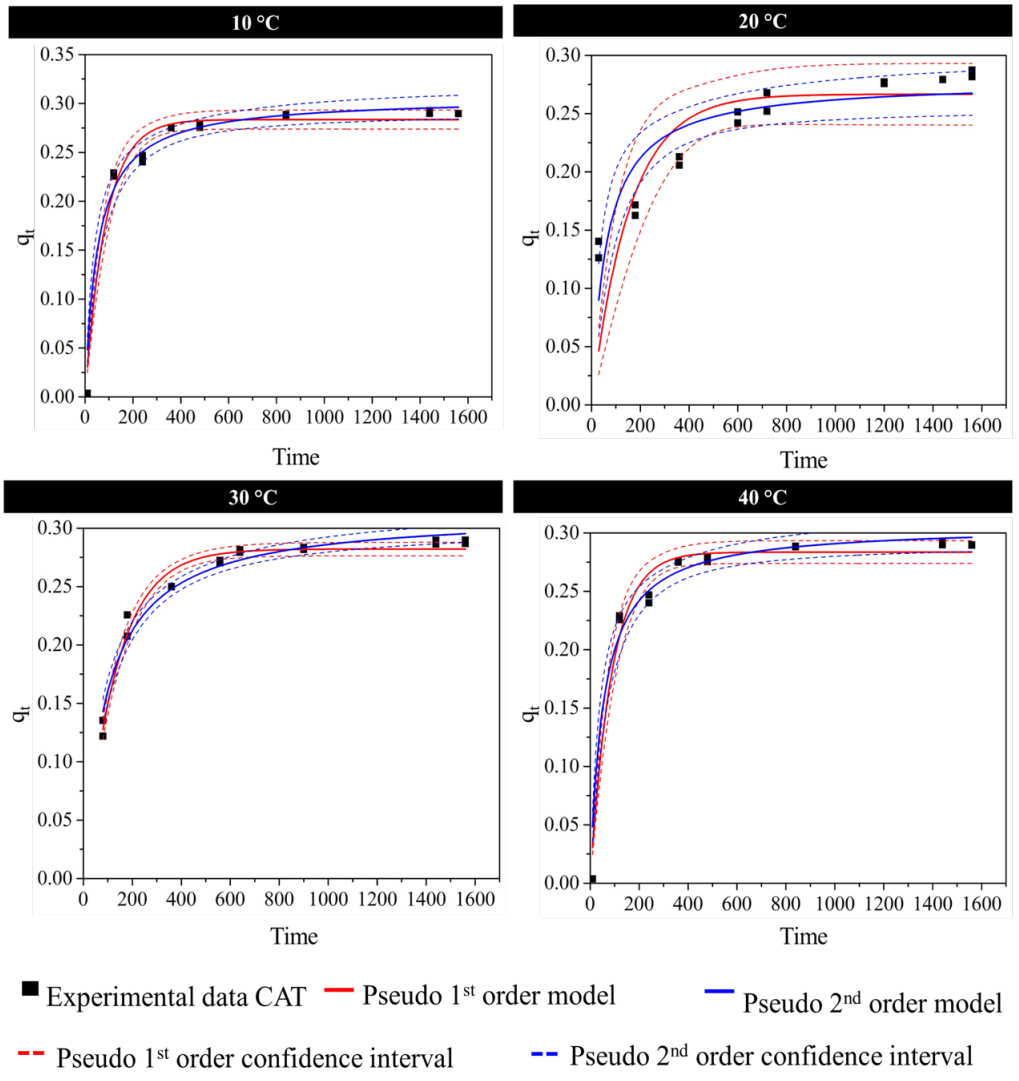

Fig. 8. Adsorption kinetics of crystal violet (CV) onto activated carbon from trunk residue (CAT) at different temperatures.

The parameters obtained from the fitting process (see Table 5) suggest that the pseudo-second-order model more accurately describes the adsorption kinetics in the case of Tr. The analysis of homoscedasticity suggests reliable and unbiased estimation of the pseudo-second order model. This behavior suggests that the kinetics are associated with both chemical interactions and the ratio of adsorbate molecules to available active sites. This result is consistent with reports in the literature on lignocellulosic adsorbents. For example, Abbas et al. (2021) observed a preferential fit to the pseudo-second-order model for VC in peanut shells, and Tenev et al. (2019) reported similar results for the removal of methylene blue and malachite green from waste in the cotton and tannin industries. For the VC–CAT system, the R2 values were closely matched at most temperatures, though a noticeable drop in fitting

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quality was observed at 20 °C ($R^2 \leq 0.76$). This high similarity in mathematical correlation at 10, 30, and 40 °C suggests that the adsorption rate on CAT is not governed by a single pure mechanism, but rather operates under a complex combination of surface chemical interactions and diffusion resistance within the porous matrix of the carbon.

In both systems, an increase in temperature was observed to result in a systematic increase in the pseudo-first-order (k_1) and pseudo-second-order (k_2) rate constants, thereby suggesting that adsorption is a thermally favored process. Conversely, it is noteworthy that the kinetic constants for Tr are higher than those for CAT, indicating that the adsorbate (VC) binds more rapidly to the surface of the material. While CAT may exhibit a higher adsorption capacity at equilibrium, Tr may prove more efficient for processes where contact time is limited, precisely because of its higher kinetic constants.

Adsorbent	Temperature (°C)	Pseudo - 1st order			Pseudo – 2nd order		
		q_e mmol g^{-1}	k_1 min^{-1}	R^2	q_e mmol g^{-1}	k_2 $g.mmol^{-1}.min^{-1}$	R^2
Tr	10	0.21	0.039	0.79	0.22	0.287	0.88
	20	0.23	0.047	0.60	0.23	0.351	0.74
	30	0.19	0.030	0.81	0.19	0.235	0.92
	40	0.23	0.098	0.78	0.23	0.660	0.83
CAT	10	0.26	0.002	0.92	0.31	0.009	0.94
	20	0.27	0.006	0.54	0.28	0.057	0.76
	30	0.28	0.007	0.97	0.31	0.033	0.97
	40	0.28	0.011	0.96	0.31	0.061	0.96

Table 5. Summary of kinetic model parameters for the adsorption process.

Conclusion

This study demonstrated the feasibility of using *Moringa oleifera* trunk waste as a cost-effective and sustainable adsorbent for the removal of crystal violet from aqueous solutions. The material was evaluated both in its raw form (Tr) and as activated carbon (CAT), and the results showed that both exhibit significant adsorption capacities. The results indicated that thermal activation effectively modified the surface properties of the material, generating a structure that enhances adsorption performance.

Future analyses characterizing the specific surface area and pore volume of these materials, along with thermodynamic studies, will allow to draw conclusions about the apparently different adsorption processes that occur on these materials. Overall, the findings support the hypothesis that *Moringa oleifera* waste, particularly in its activated form, can be used as a low-cost, efficient alternative adsorbent for the treatment of industrial effluents. This approach not only mitigates water pollution but also aligns with the principles of the circular economy, adding value to an agro-industrial by-product and transforming waste into a valuable resource.

Acknowledgements

To the University of Buenos Aires (UBACyT financing N° 20020220300235BA -20020190100323BA and PDE_43_2024) and to the company "El Moringuero S.A." from the agreement with FIUBA July 2022.

Revista Tecnología y Ciencia
DOI: https://doi.org/10.33414/rtyc.56.147-162.2026 - ISSN 1666-6933

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1-Administración del proyecto, 2-Adquisición de fondos, 3-Análisis formal, 4-Conceptualización, 5-Curaduría de datos, 6-Escritura - revisión y edición, 7-Investigación, 8-Metodología, 9-Recursos, 10-Redacción - borrador original, 11-Software, 12-Supervisión, 13-Validación, 14-Visualización.

El presente trabajo de investigación se realizo en el marco del Doctorado de Ingeniería, mención Tecnologías Químicas, Facultad Regional Buenos Aires, Universidad Tecnológica Nacional, Argentina.

This research was conducted as part of a PhD in Engineering with a specialization in Chemical Technologies at the Buenos Aires Regional Faculty of the National Technological University, Argentina.