



RESEARCH PAPER



Lignocellulosic Biosorbents from *Vitis vinifera* Agro-Waste for Cd(II) Removal: Isotherm and Kinetic Modelling in an LCA Framework

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Abstract

Cadmium contamination of water presents significant ecological and health risks and frequently necessitates expensive and energy-intensive remediation methods. This study investigated the biosorption of Cd(II) ions from wastewater utilizing agricultural waste-derived biosorbents, specifically vine shoots, grape stalks, and exhausted grape pomace. The results indicate that grape stalks exhibited the highest maximum adsorption capacity ($q_{\max}=77.52$ mg/g) and removal efficiency (87.67%), followed closely by vine shoots ($q_{\max}=40.82$ mg/g, 93.86% removal) and exhausted grape pomace ($q_{\max}=26.67$ mg/g, 73.02% removal). Kinetic studies revealed that the biosorption process adhered to a pseudo second-order model, suggesting chemisorption, while isothermic studies indicated that the Langmuir model best described the adsorption behavior. A Life Cycle Assessment demonstrated that grape stalks are the most sustainable option, with lower energy consumption (17.55 kJ) and shorter processing times (10 min) than other biosorbents. The findings underscore the potential of using winery waste as an effective and environmentally sustainable solution for heavy metal removal, promoting sustainable practices within the wine industry, and contributing to circular economic principles.

Highlights

- Vine shoots and grape stalks achieve 99.94% and 96.95% Cd(II) removal, respectively.
- Exhausted grape pomace reaches up to 79.29% removal, with lower costs and energy.
- Langmuir isotherm shows max adsorption: 77.52 mg/g for grape stalks.
- Adsorption kinetics follow pseudo-second order, indicating chemisorption.
- LCA shows grape stalks as the most sustainable, minimizing environmental impact.

Keywords Biosorption · Winery waste management · Life cycle assessment · Cadmium removal · Adsorption kinetics · Isotherm modeling

Introduction

In Europe, agriculture contributes 700 million of the over one billion tons of waste produced annually (Toop et al. 2017). Grapes, with an annual yield of over 60 million metric tons, are among the world's most significant fruit crops (Bustamante et al. 2008). In this context, the wine industry is calling for recycling initiatives to utilize its waste as raw materials for value-added products. This aims to enhance sustainability within one of agriculture's primary sectors. Spain, ranked as the third-largest wine producer, achieved an average of 50.9 million hectoliters of wine during the 2023/2024 campaign (Ministry of Agriculture, Fisheries and Food, 2024). According to Spigno et al., (2017), vine shoots, a non-woody lignocellulosic agricultural residue,

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yield approximately 1.7 tons/hectare annually in Spain. During the winemaking process, grape pomace, grape stalks, and wine lees are the primary solid by-products. Grape stalks represent approximately 15% of total solid waste and around 4% of processed grape weight (Silva et al. 2021). Grape pomace, which can reach 60% of solid waste by weight, represents 20–25% of grape input (Ferri et al. 2020). European Council Regulation mandates that lees and grape pomace be sent to alcohol distilleries for conversion into exhausted grape pomace (*Council Regulation (EC) No 479/2008*, 2008).

Addressing heavy metal pollution in aqueous solutions is equally crucial. Heavy metals, highly toxic due to their non-biodegradability, mobility, and persistence, enter the environment primarily through human activities, posing serious health risks to humans, soil, and aquatic life (Elahi et al. 2020; El-Gaayda et al. 2021). Cadmium (Cd(II)) is the second most widely used heavy metal in industries (Yaashikaa et al. 2024). It is found in diverse sources, including batteries, plastics, metal coatings, pigments, electroplating or metallurgical industries, insecticides, chemicals, and nuclear fission plants (Priya et al. 2022). Numerous technologies, such as nanofiltration, ion-exchange, and chemical precipitation, have been developed to remove pollutants from contaminated water. However, these conventional methods have several drawbacks, such as low removal efficiency, high energy consumption and high operational costs, production of hazardous sludge, and disposal issues (Wang et al. 2022). Biological and agricultural materials have emerged as viable solutions in the search for readily available, low-cost biosorbents (Girijan and Kumar 2019). Biosorption offers many benefits, including low cost and significantly increased heavy metal removal efficacy in aqueous solutions (Bano et al. 2018). Furthermore, biosorption does not produce toxic sludge by-products, and the biosorbents can be regenerated for reuse. Biosorbents can be derived from algae, microbial, and agricultural waste-based materials. Using dead biomass, such as agricultural waste, instead of living biomass has several advantages. One key benefit is the removal of the need for growth media and nutrients, which reduces overall operating expenses (Barros et al. 2020). Agricultural residues, mainly consisting of lignin, cellulose, or hemicellulose, possess high adsorption capacities and diverse functional groups that enable metal complexation (Zamora-Ledezma et al. 2021). Winery waste contains lignocellulosic compounds with significant active functional groups, rendering them valuable as biosorbents for removing metals from aqueous solutions (Sánchez-Ponce et al. 2023). Additionally, these biosorbents can be easily modified to enhance their sorption capacities and their adaptability for industrial-scale applications (Castro et al. 2021).

Furthermore, this study supports the United Nations Sustainable Development Goals (SDGs) by promoting sustainable industry and infrastructure (SDG 9), clean water and sanitation (SDG 6), aquatic life protection (SDG 14), responsible consumption and production (SDG 12), and addressing climate action (SDG 13) (see Fig. 1). This research is novel in its approach by comparing the biosorption efficiency of three different unmodified winery wastes (vine shoots, grape stalks, and exhausted grape pomace) in removing Cd(II) from water, while simultaneously incorporating life cycle assessment (LCA) to evaluate the environmental impacts of these processes. This dual focus on comparative effectiveness and sustainability remains largely unexplored in current literature, distinguishing this study from other works in the field. When compared to other research that use agricultural residues for Cd(II) removal, many have demonstrated the potential of biosorption without addressing environmental impact or comparing multiple biosorbents. For instance, Moubarik and Grimi, (2015), studied olive stones and sugar cane bagasse as biosorbents for Cd(II) removal, but did not incorporate a systematic comparison or LCA. Studies specifically addressing winery residues for Cd(II) biosorption are limited and frequently involve additional processing. For example, Sardella et al., (2015), converted viticultural waste into activated carbon to remove cadmium and lead, requiring additional processing steps. By using unmodified winery waste, this study reduces processing costs and avoids chemical inputs, providing a cost-effective alternative. This research also distinguishes itself by addressing the lack of studies using winery residues for Cd(II) removal, as most existing research focuses on other heavy metals, such as chromium. For instance, Sánchez-Ponce et al., (2023) used a by-product from the sherry wine industry to remove Cr(VI). Applying LCA to biosorption with natural, waste-derived biosorbents further enhances the novelty of this work. Few studies, such as those by Garcia et al., (2022) or Filote et al., (2021), have applied LCA to heavy metal biosorption, typically focusing on nanomaterials or microbial sorbents.

Materials and Methods

Biosorbents Characterization

In this research, vine shoots, grape stalks, and exhausted grape pomace were used as biosorbent materials for the removal of cadmium(II) from wastewater. The vine shoots and grape stalks were supplied by a winery (Cárdenas, La Rioja, Spain).

After the harvest, generally from September to early October, grape stalks are generated as a significant by-product of

Fig. 1 Contribution of the bio-sorption process using winery agro-waste to the SDGs. The process achieves up to 99.95% Cd(II) removal from wastewater (SDG 6), reuses agricultural residues without commercial value (SDG 12), reduces energy consumption to as low as 17.55 kJ in optimized scenarios (SDG 13), prevents heavy metal discharge into aquatic ecosystems (SDG 14), and promotes sustainable innovation in industrial wastewater treatment (SDG 9)



the winemaking industry. Grape stalks refer to the stems of the grapes that are separated during the destemming process, a common technique used before grape pressing in winemaking. This process involves the use of a specialized machine, the destemmer, which forces the grapes to pass through rotating blades, thereby separating the grapes from their stalks. The separated stems, known as grape stalks, constitute a considerable waste product in wine production. Following the harvest, pruning occurs, usually between December and January. During this phase, vine shoots, essential for the growth and production of grapes, become waste. Pruning the vine shoots helps control the excessive growth of the vine and improves the quality of the next harvest. Although initially vital, once they have served their purpose, vine shoots become a by-product of viticulture.

On the other hand, the alcohol distillation industry uses winery waste as a primary raw material. Specifically, grape pomace, the main solid organic waste produced during the pressing and fermentation stages, is used. Grape pomace mainly consists of peels and seeds (Beres et al. 2017). According to European Council Regulation (Council Regulation (EC) No 479/2008, 2008) lees and grape pomace

must be transferred to alcohol distilleries in order to be transformed into exhausted grape pomace. After the initial winemaking process, this residual grape pomace serves as a crucial input for alcohol distillation. During distillation, this pomace produces alcohol, leaving exhausted grape pomace as the final waste product. Figure 2 illustrates the generation of these three winery wastes, which are used as biosorbents in this study.

To prepare these residues for use as biosorbents in the cadmium removal process from water, they were cleaned 3 times with deionized water and then sun-dried for 48 h. Subsequently, they were ground and sieved to achieve particle sizes less than or equal to 160 μm .

Additionally, an elemental analysis of the three biosorbents was carried out in accordance with ASTM standard (ASTM, Standard D5373-16 2016). To ensure consistency and reliability, the experiments were performed in triplicate, maintaining a relative deviation within $\pm 0.13\%$. The average values obtained were used for analysis, as presented in Table 1. Elemental analysis revealed that among the raw materials used for biosorption, vine shoots contained the highest carbon percentage, followed by grape stalks and

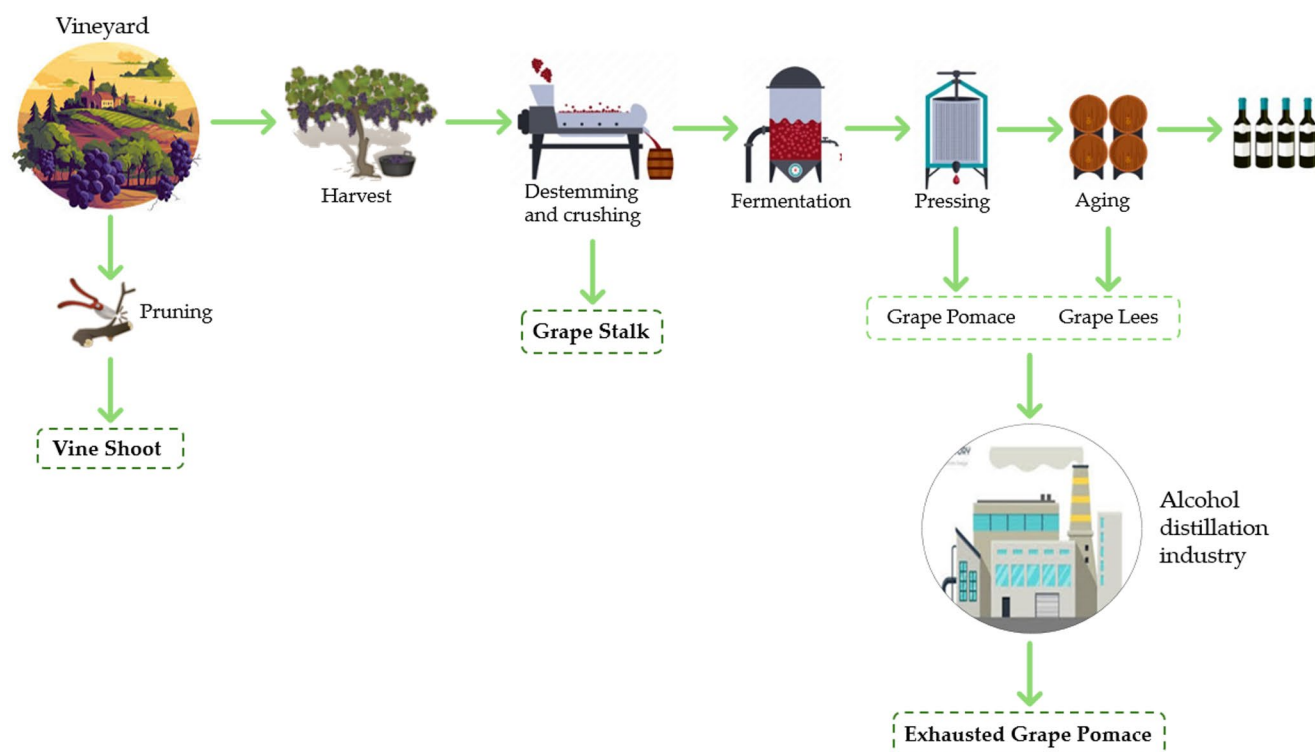


Fig. 2 Process of generating winery waste used as biosorbent

Table 1 Elemental characterization of biosorbents

Element	Unit	Biosorbent		
		Vine shoot	Grape stalk	Exhausted grape pomace
Carbon	%	45.61	44.71	20.08
Hydrogen	%	5.88	5.51	2.07
Nitrogen	%	0.46	1.34	1.92
Oxygen	%	48.05	48.45	75.93

then exhausted grape pomace. Specifically, vine shoots were composed of 45.61% carbon and 48.05% oxygen, while grape stalks had 44.71% carbon and 48.45% oxygen. These values align with typical characteristics of lignocellulosic materials. To confirm the lignin content, the TAPPI T222 om-6 method was applied, measuring Klason lignin. The lignin content was calculated as the average of three separate sets of determinations, revealing lignocellulosic contents of 36.5% in vine shoots and 47.8% in grape stalks. In contrast, exhausted grape pomace was found to contain only 20.08% carbon and 75.93% oxygen, indicating it is not primarily lignocellulosic. According to Spinei and Oroian, (2021), the lignocellulosic content in exhausted grape pomace ranges between 16.8% and 24.2%.

Design of Experiments

This research employed Response Surface Methodology (RSM), a powerful experimental approach for analyzing the relationships between controlled factors and their

corresponding responses under defined criteria (Ba Mohammed et al. 2020). RSM combines experimental design and regression analysis to optimize processes, reducing the number of necessary trials and improving operational efficiency (Gasemloo et al. 2019). In this study, RSM was combined with the Box-Behnken design (BBD) to identify the optimal conditions for cadmium adsorption using vine shoots, grape stalks, and exhausted grape pomace.

RSM based in BBD is a powerful tool widely applied in biosorption studies to optimize key operational parameters, enabling the determination of conditions that maximize removal capacity. Design-Expert v.23.1.0 software (Stat-Ease Inc., Minneapolis, USA) was employed to fit a quadratic model to the experimental data and identify the parameters yielding the highest response. The Design of Experiments (DoE) technique was employed to improve process efficiency by making simultaneous adjustments to all relevant parameters.

For cadmium biosorption studies, 25 experiments were conducted, focusing on four key input parameters: (1) initial Cd(II) concentration (C_0), (2) solution pH (pH), (3) stirring time ($Time$), and (4) doses of vine shoot, grape stalk, or exhausted grape pomace (Bio_Dose). The initial Cd(II) concentrations (0.5, 50, and 100 mg/L) were selected based on regulatory relevance and experimental design requirements. The minimum value of 0.5 mg/L corresponds to the regulatory limit established in Spain for Cd(II) discharge into

Table 2 Input parameters and their ranges

Input	Notation	Magnitude	Levels		
			-1	0	1
Initial Cd(II) concentration	C_0	mg/L	0.5	50	100
Solution pH	pH	–	2	4	8
Stirring time	$Time$	minute	10	95	180
Biosorbent dose	Bio_Dose	g	0.1	1.55	3

public wastewater treatment systems. The maximum concentration of 100 mg/L reflects conditions found in highly contaminated industrial effluents. The intermediate concentration of 50 mg/L was set as the central value required by the BBD employed in this study, which is essential for constructing a second-order polynomial model in the framework of RSM. The stirring speed was consistently maintained at 300 rpm. Table 2 outlines these input parameters, detailing their respective ranges and values used in the experiments.

To prepare the experimental water samples contaminated with cadmium, $CdCl_2 \cdot 2.5H_2O$ and distilled water were used. After the biosorption process, the remaining Cd(II) ions in the solution were then analyzed using an atomic absorption spectrophotometer (PinAAcle 500; PerkinElmer Inc., Waltham, USA). The efficiency of Cd(II) removal was determined by measuring absorbance, which was used to calculate the final Cd(II) concentration (C_1) for each experiment. The removal efficiency ($\%Rem$) was calculated using Eq. (1) to compare the final concentration (C_1) with the initial one (C_0).

$$\%Rem = \frac{C_0 - C_1}{C_0} \cdot 100 \quad (1)$$

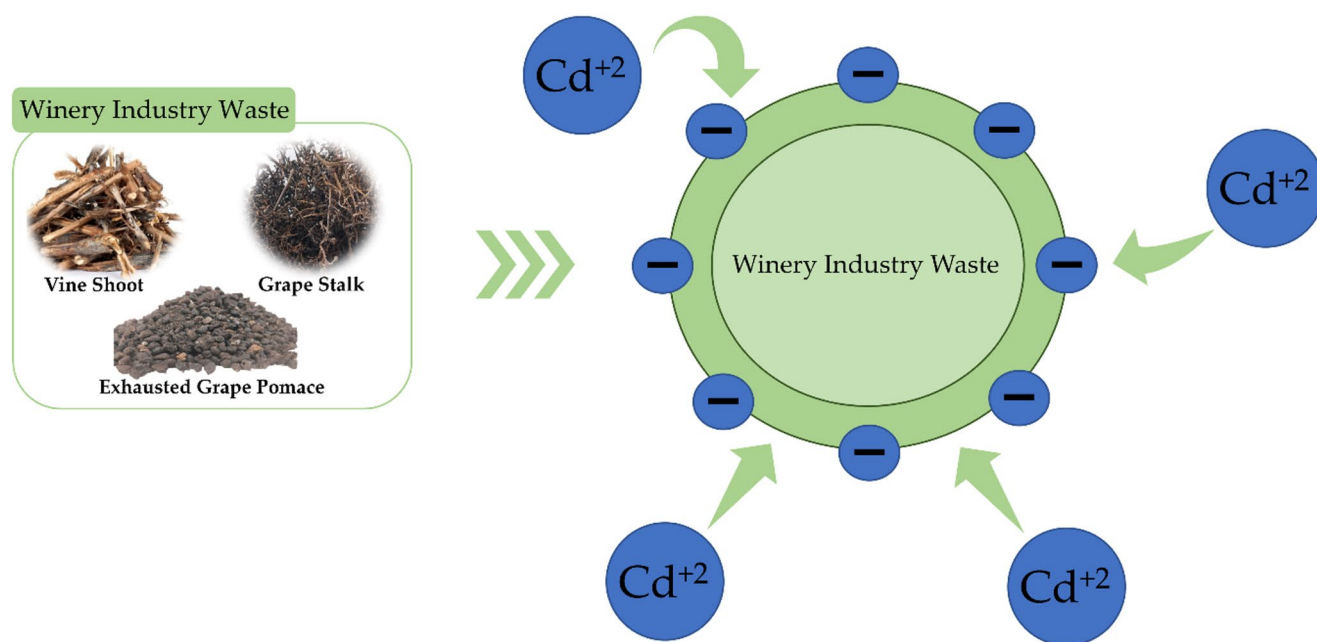
All the reagents used in this study were of analytical grade and provided by Merck & Company.

Biosorption Process and Life Cycle Assessment

Biosorption is a physicochemical process that uses biomaterials, or biosorbents, for the removal of pollutants from aqueous solutions. In this process, ions from a liquid phase bind to the surface of a solid phase, such as biomass. Biosorption involves multiple mechanisms, including adsorption, ion exchange, surface complexation, chelation, precipitation, redox reactions, and electrostatic interactions (Syeda et al. 2022). Biosorbents derived from agricultural waste, such as vine shoot, grape stalk, and exhausted grape pomace, are particularly promising due to their low cost and abundance. These materials are rich in lignocellulose, which provides numerous active sites for the binding of heavy metals (Sánchez-Ponce et al. 2023). Unlike living biomass, dead biomass from agricultural residues requires no growth media or nutrients, which reduces operational costs (Yaashikaa et al. 2024). The process complies with green chemistry principles, making it a sustainable and attractive alternative for mitigating environmental pollution (Anastopoulos et al. 2019).

Figure 3 shows the biosorption mechanism occurring on the surface of the biosorbents used in this research (vine shoot, grape stalk and exhausted grape pomace).

Finally, a Life Cycle Assessment (LCA) was conducted to quantify the environmental impacts of the biosorption process used in this study by measuring the energy, materials, and pollutants involved (Haque et al. 2023). Reusing

**Fig. 3** Biosorption process using winery wastes

agricultural resources can create inter-industrial connections and increase the value of byproducts. Therefore, LCA measures how agricultural waste affects the environment and transforms it into byproducts with added value (Vaneck-haute et al. 2018). For instance, recent studies have demonstrated that biosorption using algae biosorbents can achieve significant cadmium removal (Fawzy et al. 2022). By integrating LCA into the biosorption process, researchers can assess not only the effectiveness of cadmium removal but also the overall environmental benefits, such as reduced chemical usage and lower energy consumption compared to conventional treatment methods (Sabando-Fraile et al. 2024).

The LCA was conducted following the guidelines outlined in ISO Standards 14,040/14044 (ISO 2006). Figure 4 details the four steps followed to perform the LCA: (1) defining the goal and scope of the study, (2) conducting a Life Cycle Inventory (LCI) analysis, (3) performing a Life Cycle Impact Assessment (LCIA), and (4) interpreting the results. When applied to the biosorption process for cadmium removal from wastewater, LCA can provide insights into the sustainability and efficiency of using biological materials as sorbents.

The initial phase of the LCA involves defining the goal and scope, which must be clearly outlined. This study aims to compare the environmental impacts of using three biosorbents (vine shoots, grape stalks, and exhausted grape

pomace) in the Cd(II) biosorption process. The functional unit selected is the treatment of 1 L of Cd(II)-contaminated distilled water. The results obtained from the three biosorbent scenarios were then analyzed and compared.

The scope of the LCA is limited to the biosorption process using these three biosorbents, conducted from a "gate-to-gate" perspective. This study examines resource use and environmental impacts from emissions during the biosorption process. Scaled-up inventory data were used to identify elements that could have the most significant environmental impacts. Laboratory-prepared wastewater samples were used for the experiments, accounting for final and initial cadmium concentrations, the dose of biosorbent, and the energy consumed during these biosorption processes. Vine shoots, grape stalks, and exhausted grape pomace, as agricultural residues with minimal commercial value, were considered raw materials without intrinsic worth, meaning allocation factors were not applied. Energy consumption of the stirrer used during these biosorption processes was measured and included in the analysis. Additionally, the energy required for grinding and sieving was also considered in the Life Cycle Inventory (LCI). LCI was conducted using SimaPro v.9.2.0.2, with background data sourced from the Ecoinvent 3 database. Material and energy inputs were derived from laboratory experiments and equipment specifications. The system boundaries were defined as gate-to-gate, encompassing all laboratory-scale operations directly related to

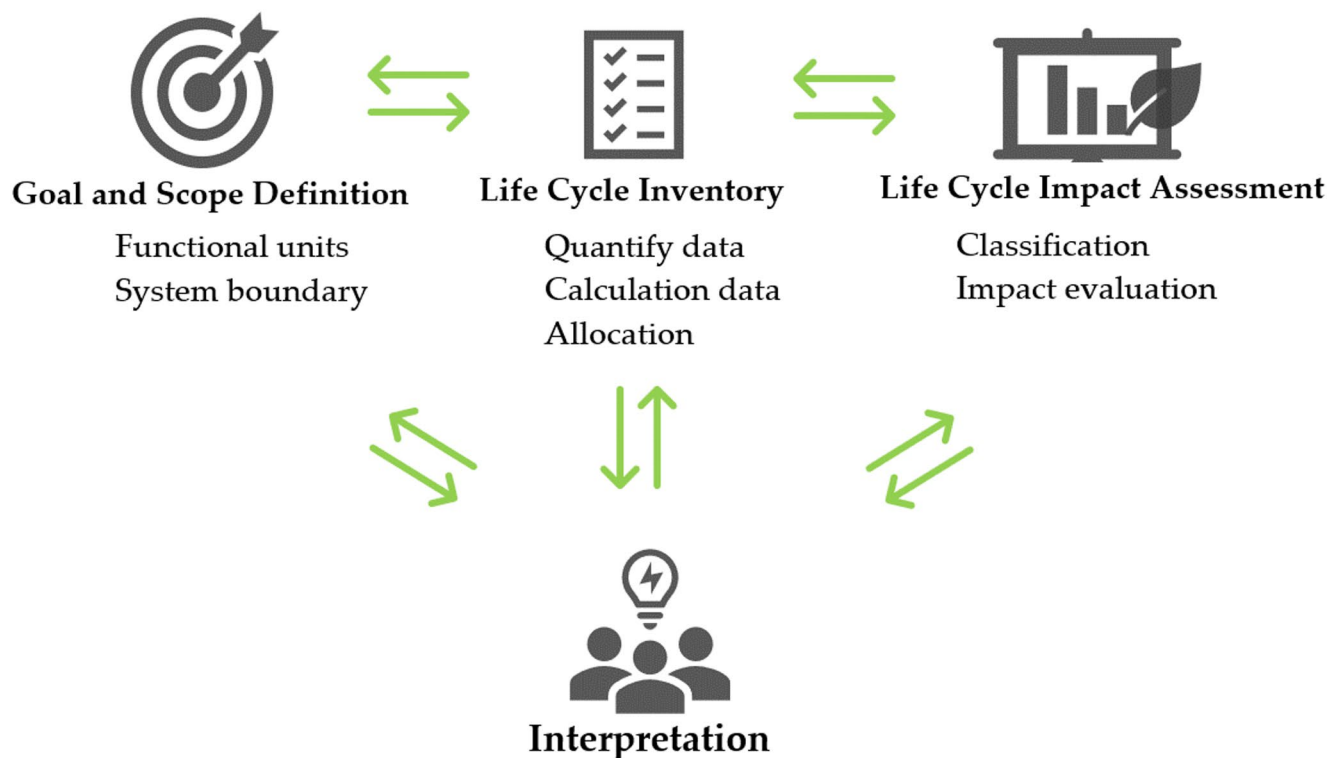


Fig. 4 Life cycle assessments key steps

the biosorption process, including biosorbent preparation (grinding and sieving), biosorption experiments (stirring), and preparation of Cd(II) solutions. Table 3 summarizes all input and output flows considered in the inventory, clearly distinguishing included and excluded elements along with their respective justifications.

Two main approaches can be employed: midpoint and endpoint analyses. While the midpoint approach models environmental impacts into specific damage categories, the endpoint approach measures broader environmental burdens on ecosystems, human health, and resources (Sangma and Chalie-u 2023). In this study, only the midpoint approach was applied, using the CML-IA baseline to evaluate environmental impacts. The midpoint approach was chosen for its greater transparency and reduced uncertainty compared to endpoint indicators. Midpoint indicators, which focus on early stages of the cause-effect chain, are commonly preferred in wastewater studies (Khanali et al. 2022). Unlike endpoint indicators, which condense impacts across several categories but add complexity and ambiguity, midpoint indicators offer a clearer and more reliable assessment of environmental impacts (Reap et al. 2008). The CML-IA midpoint method was selected for the Life Cycle Impact Assessment (LCIA) as it is widely recommended for evaluating environmental impacts in wastewater treatment scenarios due to its reduced uncertainty and higher interpretability (Corominas et al. 2020). All eleven midpoint categories defined in the CML-IA baseline were included in this study: global warming, ozone layer depletion, human

toxicity, freshwater aquatic ecotoxicity, marine aquatic ecotoxicity, terrestrial ecotoxicity, photochemical oxidation, acidification, eutrophication, and abiotic resource depletion (fossil and mineral). These categories were chosen because they comprehensively represent the environmental burdens potentially associated with biosorption processes, especially those involving energy use, material consumption, and interaction with aquatic ecosystems. Their inclusion ensures a holistic environmental profile of each biosorbent and enables robust comparison under the principles of sustainability and circular resource use.

Kinetic Study

Kinetic studies in each biosorption process provide crucial data on the adsorption rate, the performance of the biosorbents, and the mechanisms of mass transfer (Wang and Guo 2020). Understanding the adsorption kinetics is crucial for designing efficient biosorption systems, as it allows for the prediction of how quickly Cd(II) ions will bond to the surface of the biosorbents (Syeda et al. 2022). By applying pseudo first order (Eq. (1)) and pseudo second order (Eq. (2)) kinetic models, this research evaluates the rate at which adsorption occurs and highlights the effectiveness of each biosorbent, essential for optimizing the design and operation of the biosorption processes.

$$\ln(q_e - q_t) = \ln(q_e) - k_1 \cdot t \quad (2)$$

Table 3 Life cycle inventory inputs and outputs, with inclusion criteria and justifications

Item	Unit	Material/process	Included/excluded	Justification
Input (resources)	L	Water (used for metal solution preparation)	Included	Essential for solution preparation; modeled as laboratory-grade water
Input (chemicals)	mg	CdCl ₂ ·2.5H ₂ O (cadmium reagent)	Included	Reagent source for biosorption experiments
Input (materials)	g	Biosorbent dose	Included	Comparative functional element across biosorbents
Input (electricity)	kJ	Electricity for stirring, grinding, and sieving	Included	Primary energy input, measured per experiment
Output	mg	Final Cd(II) concentration in solution	Included	Used to determine removal efficiency
Output (Cd(II) removed)	mg	Cd(II) retained by the biosorbent	Included	Indicates pollutant removal
Emissions to water	mg	Residual Cd(II) in treated water	Included	Considered relevant emission from the system
Infrastructure	–	Laboratory glassware, beakers, sensors	Excluded	Assumed reusable and negligible in impact for all cases
Transport	–	Reagents and materials to laboratory	Excluded	Uniform sourcing for all scenarios, negligible at laboratory scale
Waste treatment	–	Post-experiment liquid handling and biosorbent disposal	Excluded	Out of gate-to-gate scope, same handling assumed for all scenarios
Drying process	–	Ambient drying of biosorbents	Excluded	No active energy input, performed at room temperature

$$\frac{t}{q_t} = \frac{1}{k_2 \cdot q_e^2} + \frac{t}{q_e} \quad (3) \quad \log(q_e) = \frac{1}{n} \cdot \log(C_e) + \log(k_F) \quad (6)$$

where q_t and q_e (mg/g) represent the quantities of Cd(II) ions adsorbed at time t (min) and equilibrium, respectively. The pseudo first and pseudo second order adsorption rate constants are denoted by k_1 (1/min) and k_2 (g/mg·min), respectively.

Isothermic Study

Isothermic studies were applied to each biosorption process to better understand the interaction mechanisms between Cd(II) ions (the adsorbate) and the biosorbent surfaces (vine shoots, grape stalks, and exhausted grape pomace) (Fathollahi et al. 2021). The Langmuir and Freundlich isotherm models were used to evaluate the adsorption behavior. The Langmuir isotherm assumes adsorption occurs in a monolayer on a homogenous surface, describing the relationship between the adsorbed metal ions and the available binding sites (Syeda et al. 2022). The equation representing this model is given by Eq. (3):

$$\frac{1}{q_e} = \frac{1}{C_e \cdot k_L \cdot q_{\max}} + \frac{1}{q_{\max}} \quad (4)$$

where C_e is the concentration of Cd(II) in the solution at equilibrium (mg/L), q_e represents the amount of Cd(II) adsorbed onto the biosorbent at equilibrium (mg/g), $q_{\max}$ refers to the maximum monolayer adsorption capacity of the biosorbent (mg/g), and k_L is the Langmuir constant (L/mg), which reflects the affinity between the Cd(II) ions and the biosorbent surface.

Using the Langmuir isotherm equation, a dimensionless constant known as the separation factor (R_L) can be calculated as follows (Eq. (4)):

$$RL = \frac{1}{1 + k_L \cdot I_{\text{Con}}} \quad (5)$$

An R_L value between 0 and 1 suggests favorable biosorption, while a value greater than 1 indicates that the biosorption is less effective. If the R_L equals 1, it means a linear biosorption process, and a value of 0 implies that the biosorption is irreversible (Gherraby et al. 2024).

The Freundlich isotherm suggests a multilayer adsorption process on heterogeneous surfaces, providing insights into the surface diversity of the biosorbent (Syeda et al. 2022). The equation used in this model is expressed as given in the following equation (Eq. 5):

where q_e represents the amount Cd(II) adsorbed onto the biosorbent at equilibrium (mg/g), and C_e is the concentration of Cd(II) in the solution at equilibrium (mg/L). The constants k_F and n are Freundlich-specific parameters: k_F (mg/g)(L/mg)^{1/n} is the Freundlich constant that indicates the adsorption capacity of the biosorbent, and n is a dimensionless constant that reflects the adsorption intensity or favorability. A higher n value suggests more favorable adsorption, with n typically being greater than 1 for favorable adsorption conditions.

FTIR Characterization of Biosorbents

Fourier Transform Infrared (FTIR) analysis was performed to identify the chemical structures and determine the functional groups present on the surface of the biosorbents, which may influence their ability to adsorb Cd(II) ions. The measurements were conducted using a PerkinElmer Spectrum Two FTIR spectrometer (PerkinElmer, USA) within the wavenumber range of 4000–500 cm⁻¹. For each biosorbent sample, four individual scans were taken, and the resulting spectra were averaged to ensure the accuracy and representativeness of the data, providing a comprehensive overview of the functional groups involved in the biosorption process.

SEM-EDS

To understand the mechanism of heavy metal adsorption in biosorbents, the surface morphology and elemental composition of the biosorbent has been analyzed using SEM-EDS, which provides detailed information on the distribution and potential interactions of surface functional groups.

Results and Discussion

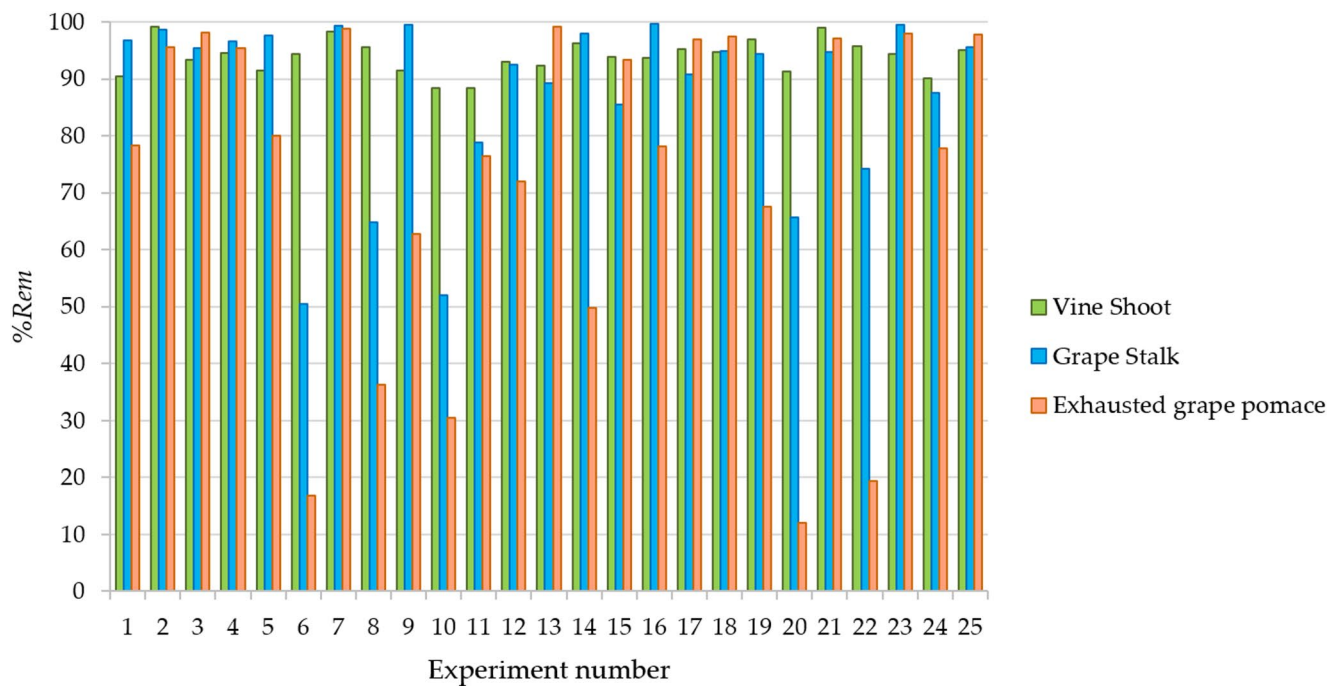
Experimental Results

Table 4 presents the values of the input parameters and the corresponding results obtained for the outputs. As previously explained, the output variable investigated was the percentage removal of Cd(II) ions from water during the biosorption process using vine shoots (%Rem_VS), grape stalks (%Rem_GS), or exhausted grape pomace (%Rem_EGP).

Figure 5 compares the percentage of Cd(II) removal achieved for each of the three biosorbents. The results indicate that vine shoots are the most effective and stable biosorbent, with an average removal rate of 93.86%. Grape

Table 4 Results and design of experiments

Experiment number	Input parameters				Output variables		
	<i>C₀</i>	<i>pH</i>	<i>Bio_Dose</i>	<i>Time</i>	<i>%Rem_{VS}</i>	<i>%Rem_{GS}</i>	<i>%Rem_{EGP}</i>
	mg/L	–	g	min	%	%	%
1	0.5	2.0	1.55	95	90.40	96.69	78.37
2	100	8.0	1.55	95	99.13	98.59	95.59
3	50	2.0	3.00	95	93.40	95.43	98.21
4	50	5.0	3.00	10	94.51	96.55	95.47
5	0.5	5.0	1.55	10	91.41	97.61	79.99
6	100	5.0	0.10	95	94.32	50.40	16.76
7	50	8.0	1.55	180	98.35	99.24	98.86
8	50	5.0	0.10	180	95.52	64.81	36.28
9	0.5	8.0	1.55	95	91.40	99.52	62.79
10	50	2.0	0.10	95	88.33	52.06	30.40
11	0.5	5.0	0.10	95	88.41	78.84	76.36
12	50	2.0	1.55	10	93.04	92.54	71.94
13	50	8.0	3.00	95	92.25	89.16	99.20
14	50	8.0	1.55	10	96.25	97.89	49.82
15	100	2.0	1.55	95	93.84	85.43	93.38
16	0.5	5.0	1.55	180	93.70	99.71	78.11
17	50	2.0	1.55	180	95.29	90.81	96.89
18	50	5.0	3.00	180	94.65	94.87	97.46
19	100	5.0	1.55	10	96.91	94.44	67.47
20	50	5.0	0.10	10	91.29	65.74	11.94
21	100	5.0	1.55	180	98.98	94.65	97.10
22	50	8.0	0.10	95	95.74	74.26	19.44
23	50	5.0	1.55	95	94.30	99.54	98.02
24	0.5	5.0	3.00	95	90.10	87.51	77.84
25	100	5.0	3.00	95	94.97	95.60	97.87

**Fig. 5** Comparison of removal percentages depending on the biosorbent

stalks follow with 87.67%, while exhausted grape pomace has the lowest removal rate at 73.02%. Notably, trials 6, 8, 10, 20, and 22 showed significant drops in removal percentage. These experiments share the common characteristic that the biosorbent dose (*Bio_Dose*) used was the lowest, at 0.1 g. This observation suggests that using this specific amount of biosorbent results in a notable decrease in contaminant removal efficiency, highlighting the crucial importance of the biosorbent dosage in the process. The explanation for this observation relates to the principle of adsorption: a higher amount of biosorbent increases the availability of adsorption sites to capture Cd(II) ions, resulting in a higher removal percentage (Waqar et al. 2023).

Conversely, reducing the biosorbent dose limits the number of available adsorption sites, leading to quicker saturation of the biosorbent and, consequently, a decrease in the contaminant removal percentage.

To validate the performance of the quadratic models developed for the Cd(II) biosorption process, a correlation analysis was conducted between the experimental and predicted removal efficiencies for each biosorbent (Fig. 6). In all three cases, the data points are closely aligned along the diagonal line, indicating excellent agreement between the predicted and actual values, and confirming the strong predictive accuracy of the models. The corresponding coefficients of determination (R^2) are shown in Table 5. The

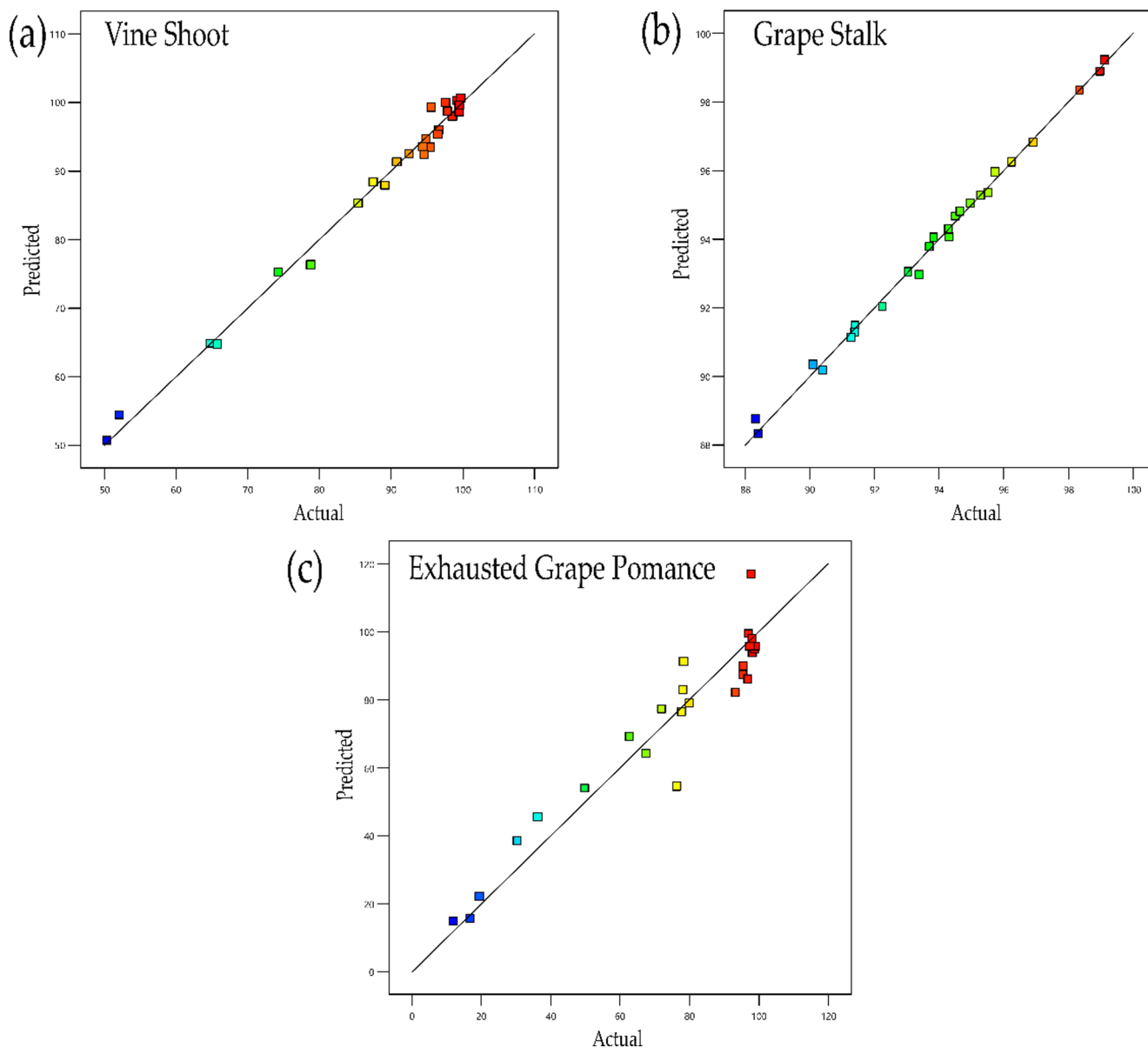


Fig. 6 Correlation between actual and predicted Cd(II) removal efficiencies for each biosorbent: (a) vine shoot, (b) grape stalk, and (c) exhausted grape pomace

Table 5 Determination coefficients (R^2) of the quadratic models for Cd(II) biosorption using each biosorbent

R^2	Biosorbent		
	Vine shoot	Grape stalk	Exhausted grape pomace
R^2	0.9960	0.9902	0.9159

highest R^2 was observed for vine shoot (0.9960), followed by grape stalk (0.9902), both indicating excellent model fits. Although slightly lower, the exhausted grape pomace model also showed a good correlation with an R^2 of 0.9159, confirming its reliability. These results demonstrate that the quadratic models developed through RSM are statistically robust and well suited to describe the Cd(II) removal behavior for all biosorbents studied.

Optimization and Life Cycle Assessment

To evaluate the biosorption efficiency of using vine shoots, grape stalks, and exhausted grape pomace, this study aimed to determine the optimal conditions for removing Cd(II). Two optimization scenarios were analyzed to assess the effects of varying input parameters on removal efficiency and resource use. The first scenario, detailed in Table 6, focused just on achieving maximum Cd(II) removal. On the other hand, the second scenario, shown in Table 7, aims not only to maximize Cd(II) removal but also to optimize cost-effectiveness and reduce resource usage by minimizing the input parameters E_Cons , $Time$ and Bio_Dose . A comparison of the results from the two scenarios reveals notable differences in E_Cons , $Time$, and Cd(II) removal efficiencies ($\%Rem$). In Optimization 1, high removal rates were achieved, with each biosorbent reaching close to 99.95% efficiency. However, this scenario required higher E_Cons ,

especially for vine shoots and exhausted grape pomace (298.4 and 285.95 kJ, respectively), and involved prolonged $Time$ values. In contrast, Optimization 2 minimized energy use to 17.55 kJ for all biosorbents and shortened $Time$ to just 10 min. Notably, the optimal Bio_Dose remained relatively consistent across both scenarios, with values clustering between 1 and 1.6 g, suggesting that a dose within this range is generally effective for Cd(II) removal in both high-efficiency and low-energy contexts. While Cd(II) removal efficiencies were high across both scenarios, the second optimization resulted in slightly reduced performance for exhausted grape pomace, with a $\%Rem$ of 79.29. This decrease in efficiency can likely be attributed to the lower pH of 2.5 used, as acidic conditions can introduce more hydrogen ions that compete with Cd(II) ions for binding sites, reducing the material's adsorption capacity (Rezaee and Ahmady-Asbchin 2023). This result highlights the importance of maintaining a near-neutral to slightly alkaline pH , as observed in the other cases (pH 7–8), to maximize cadmium removal.

Using winery industry waste to remove Cd(II) from wastewater offers significant benefits and supports the circular economy. However, this biosorption process may also pose environmental challenges. To assess these potential impacts, an LCA was conducted. LCA is a tool used for assessing the environmental impacts of products (Othoniel et al. 2019). The objective of this LCA was to compare the environmental impacts associated with two optimization scenarios. The assessment was performed from a gate-to-gate perspective, using SimaPro software.

The midpoint indicator assessment yielded eleven impact categories related to the environment, human health, and resources, as depicted in Figs. 7 and 8. These figures

Table 6 Results for optimization 1

	Unit	Goal	Min	Max	Optimization 1		
					Vine shoot	Grape stalk	Exhausted grape pomace
C_0	mg/l	In Range	0.5	100	70	40	90
pH	–	In Range	2	8	8	8	7
$Time$	min	In Range	10	180	170	12	163
Bio_Dose	g	In Range	0.1	3	1.16	1.66	1.36
$\%Rem$	%	max	11.94	99.99	99.94	99.95	99.96
E_Cons	kJ	In Range	20.15	318.5	301	23.21	288.55

Table 7 Results for optimization 2

	Unit	Goal	Min	Max	Optimization 2		
					Vine shoot	Grape stalk	Exhausted grape pomace
C_0	mg/l	In Range	0.5	100	100	0.5	0.5
pH	–	In Range	2	8	8	6	2.5
$Time$	min	min	10	180	10	10	10
Bio_Dose	g	min	0.1	3	1.11	1.01	1.25
$\%Rem$	%	max	11.94	99.99	99.94	96.95	79.29
E_Cons	kJ	min	20.15	318.5	20.15	20.15	20.15

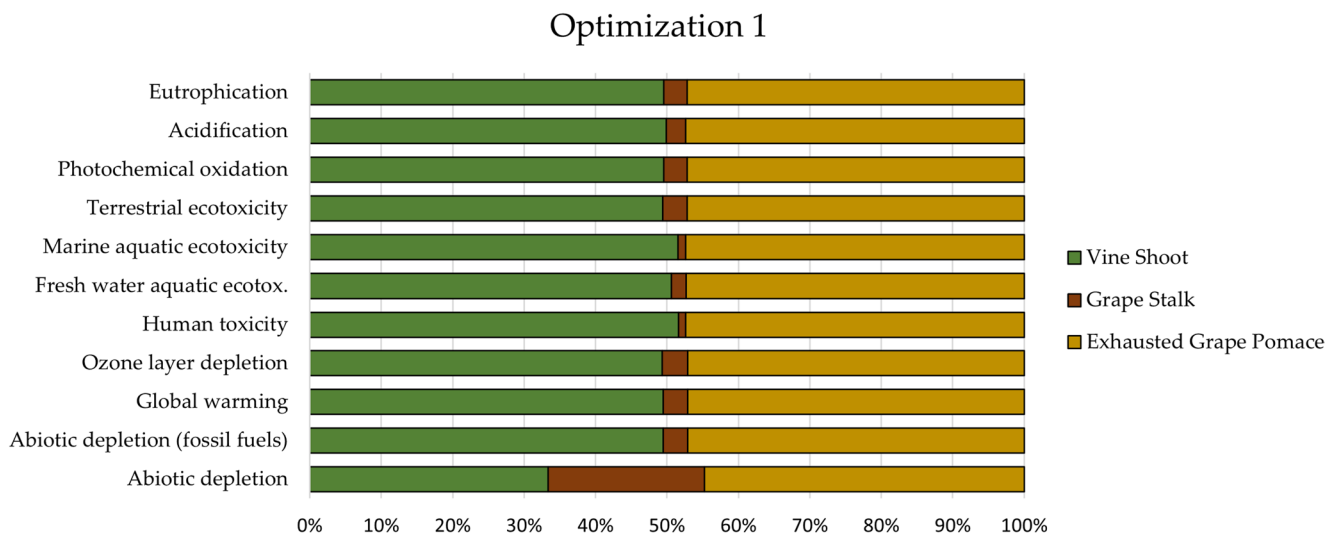


Fig. 7 Comparison of results for LCA with midpoint indicator for the first optimization scenario

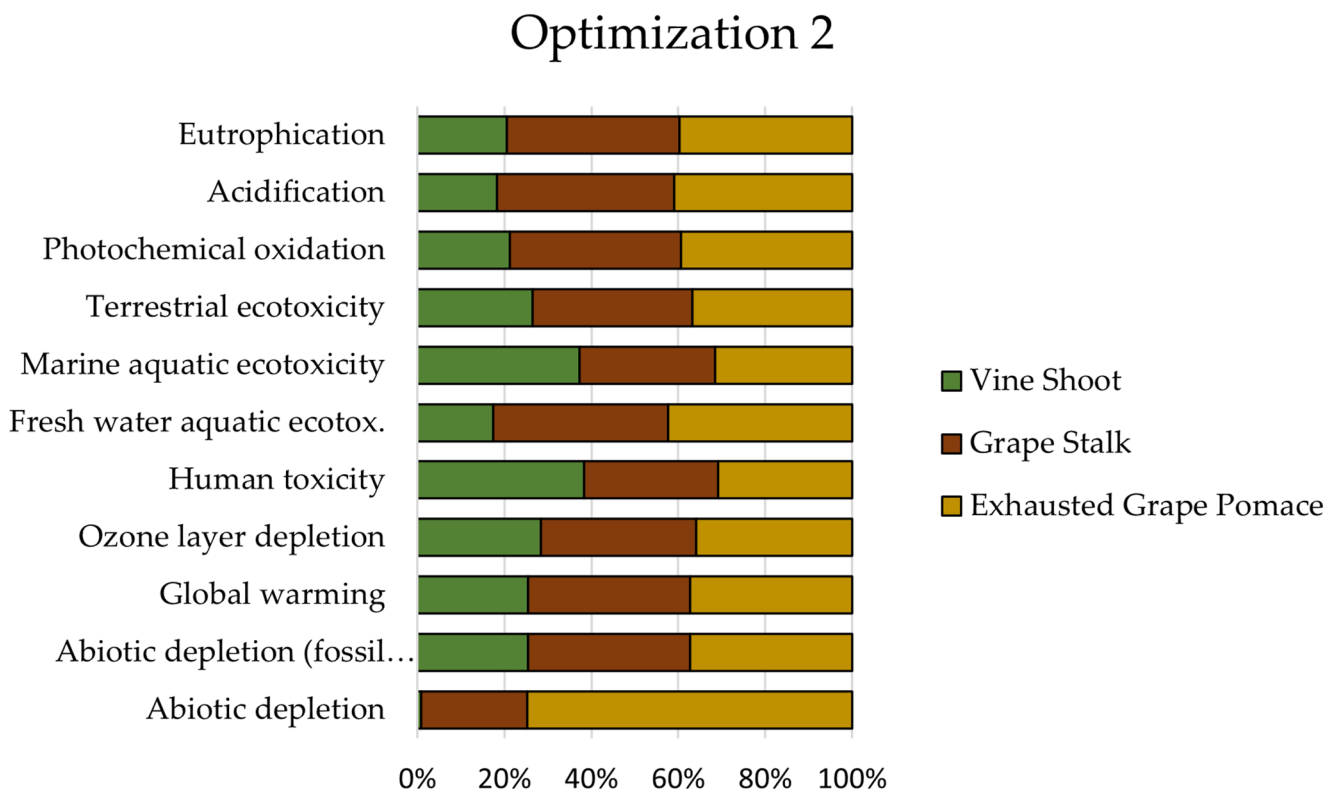


Fig. 8 Comparison of results for LCA with midpoint indicator for the second optimization scenario

illustrate the LCA results for midpoint indicators, highlighting the environmental impacts of using vine shoots, grape stalks, and exhausted grape pomace in Optimization 1 and Optimization 2, respectively. In Optimization 1 (see Fig. 7), exhausted grape pomace exhibits the highest environmental impact across most categories, including eutrophication, acidification, photochemical oxidation, and abiotic depletion. This substantial impact is likely due to the higher energy

consumption requirements, particularly for exhausted grape pomace and vine shoots, in this scenario. In contrast, grape stalks and vine shoots show lower impacts across most categories, with grape stalks performing particularly well in human toxicity, global warming, and abiotic depletion. In Optimization 2 (see Fig. 8), environmental impacts are generally reduced for all biosorbents, particularly for exhausted grape pomace. This improvement likely stems from reduced

Time and *E_Cons* set in Optimization 2. Grape stalks also exhibit minimal impacts, particularly in categories such as global warming and photochemical oxidation, reinforcing as the most sustainable choice across both scenarios.

Sensitivity Analysis

Sensitivity analysis is a methodological approach used to simulate and assess how variations in input parameters affect the outputs of a mathematical or computational model. It is particularly useful during model development to evaluate the degree of influence that each input has on the final results (Wang et al. 2023). In this study, a sensitivity analysis was carried out to verify the robustness of the life cycle assessment (LCA) results under plausible variations in energy demand.

Specifically, a 10% decrease in total energy consumption was simulated, representing potential fluctuations in laboratory-scale requirements, especially during the grinding, sieving, and stirring stages. The impact categories most affected by this energy reduction were abiotic depletion (fossil fuels), global warming, human toxicity, freshwater aquatic ecotoxicity, and marine aquatic ecotoxicity. This is consistent with the fact that electricity consumption is commonly associated with the use of fossil fuels and the release of greenhouse gases (CO_2 , NO_x), as well as secondary emissions with toxic potential. As such, these categories are proportionally more sensitive to changes in energy input compared to others such as acidification or eutrophication, which are influenced by different environmental mechanisms. Despite the reductions observed, the relative environmental performance among the biosorbents remained consistent. As shown in Fig. 9, although the

absolute values of environmental impacts decreased slightly with lower energy input, the comparative trends and ranking between biosorbents were preserved. This confirms that the environmental conclusions derived from the LCA are not significantly altered by moderate variations in energy assumptions, indicating that the model is robust and suitable for evaluating the environmental implications of these biosorption processes.

Kinetic Study

A kinetic analysis was conducted to assess the Cd(II) biosorption behavior of the three biosorbents. The analysis used the pseudo first order and pseudo second order models. The equations for these models are presented in Eqs. (2) and (3), respectively. These models help describe the adsorption rates and provide insight into the underlying biosorption mechanisms (Kavisri et al. 2023). The experiments were conducted at a *pH* of 8, a *C₀* of 100 mg/L, a *Bio_Dose* of 2.14 g, and *Time* (10–180 min).

Table 8 presents the kinetic results, showing differences between the two models. The pseudo first order model provided moderate correlation coefficients (R^2 values) of 0.9660 for vine shoots, 0.9562 for grape stalks, and 0.9601 for exhausted grape pomace. The predicted equilibrium adsorption capacity q_e values were low and k_1 rate constants ranged from 0.0384 to 0.0557 min^{-1} . In contrast, the pseudo second order model demonstrated an excellent fit, with R^2 values of 0.9999 for both vine shoots and grape stalks, and 0.9954 for exhausted grape pomace. This model also yielded higher equilibrium adsorption capacities. The rate constants k_2 varied, with grape stalks showing the highest value at 1.9369 $\text{g/mg} \cdot \text{min}$, suggesting faster adsorption

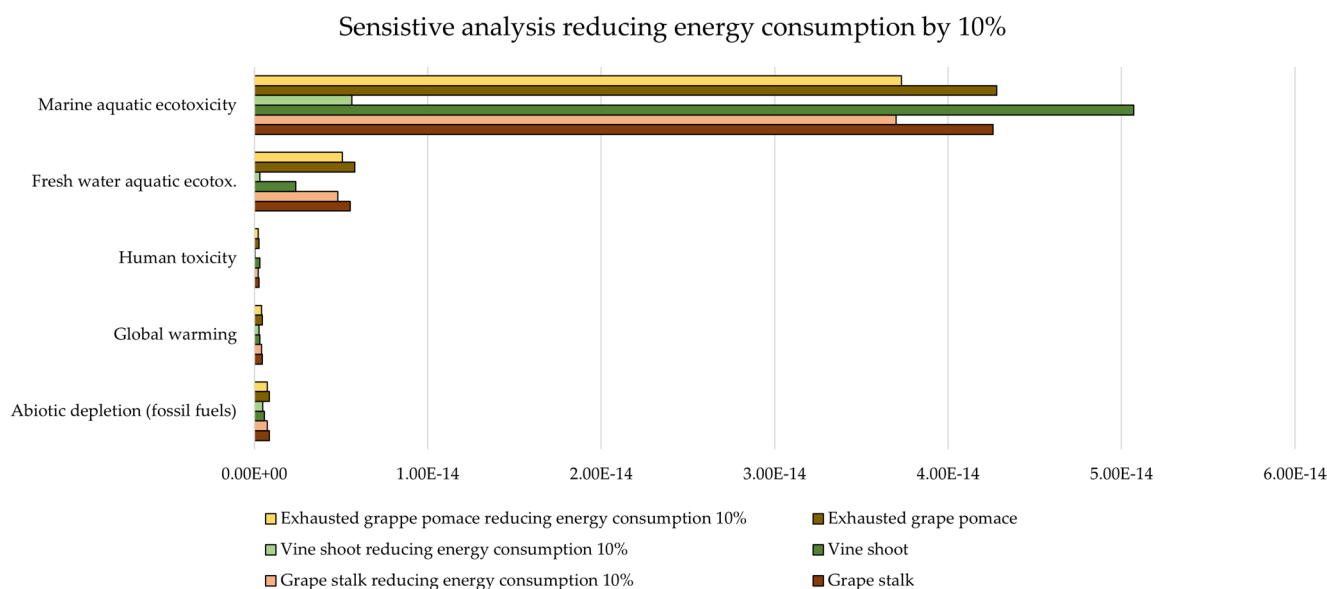


Fig. 9 Environmental impact comparison under standard and reduced energy input conditions (–10%) for the most sensitive midpoint categories

Table 8 Sorption kinetic comparison

Model	Parameter	Biosorbent		
		Vine shoot	Grape stalk	Exhausted grape pomace
Pseudo-first order	R^2	0.9660	0.9562	0.9601
	q_e (mg/g)	0.0952	0.0590	2.2123
	k_1 (min^{-1})	0.0557	0.0428	0.0384
Pseudo-second order	R^2	0.9999	0.9999	0.9954
	q_e (mg/g)	4.7036	4.8117	4.8551
	k_2 (g/mg min)	1.4000	1.9369	0.0337

Table 9 Sorption isotherm comparison

Model	Parameter	Biosorbent		
		Vine shoot	Grape stalk	Exhausted grape pomace
Langmuir	R^2	0.9982	0.9999	0.9987
	$q_{\max}$ (mg/g)	40.8163	77.5194	26.6667
	k_L (L/mg)	0.2132	0.0689	0.0109
Freundlich	R^2	0.9899	0.9986	0.9951
	n	0.6343	1.3526	1.2410
	k_F (mg/g)(L/mg) $^{1/n}$	10.2944	5.9429	2.3534

kinetics for this biosorbent. Overall, the pseudo-second-order model offered a substantially better fit for all biosorbents, suggesting that Cd(II) biosorption is likely controlled by chemisorption, involving electron sharing or exchange between the biosorbent surface and Cd(II) ions (Ho 2006).

Isothermic Study

The isothermic behavior of Cd(II) biosorption onto vine shoots, grape stalks and exhausted grape pomace was analyzed using the Langmuir and Freundlich models, represented by Eqs. (4) and (6), respectively. These models were applied to characterize and compare each biosorbent's adsorption capacity. In this case, the experiments were performed at a pH of 8, with C_0 ranging from 0.5 to 100 mg/L. A Bio_Dose of 0.1 g and a $Time$ of 75 min were maintained to ensure equilibrium conditions.

Table 9 summarizes the results of the isothermic study, with the key parameters derived from both models. For the Langmuir model, high R^2 values were observed for vine shoots, grape stalks, and exhausted grape pomace, indicating a strong fit. The maximum adsorption capacities ($q_{\max}$) aligned with this finding, with grape stalks showing the highest capacity at 77.5194 mg/g, followed by vine shoots at 40.8163 mg/g and exhausted grape pomace at 26.6667 mg/g. The Freundlich model also fit the data well but showed slightly lower R^2 values compared to the Langmuir model. Additionally, the Freundlich parameter n , which indicates adsorption intensity, was highest for grape stalks (1.3526), suggesting favorable Cd(II) biosorption

Table 10 Comparison of $q_{\max}$ values for Cd(II) biosorption using different agricultural wastes

$q_{\max}$ (mg/g)	Biosorbent	References
14.22	Alkaline-treated coconut shell powder	(Tcheka et al. 2024)
54.00	Grape bagasse	(Farinella et al. 2007; Niculescu and Ionete 2023)
21.83	Pomelo peel	(Kwikima et al. 2021)
1.09	De-oiled palm kernel waste	(Hebbani et al. 2021)
70.40	Raw aloe vera wastes	(Noli et al. 2019)
42.10	Pineapple peel	(Syeda et al. 2022)
33.20	Modified sugarcane bagasse	(Liu et al. 2020)
19.96	Potato peel	(Huang et al. 2020)

conditions. While both models provided a satisfactory fit for all biosorbents, the Langmuir model showed a stronger correlation overall, suggesting it is better suited for describing the Cd(II) biosorption process. Finally, using Eq. (5) and C_0 from 0.5 to 100 mg/L, the Langmuir isotherm (R_L) varied for the different biosorbents studied. For vine shoots, R_L ranged from 0.9036 to 0.0480, while for grape stalks, it ranged from 0.9667 to 0.1267. Exhausted grape pomace displayed R_L values between 0.9945 and 0.4772. These results suggest that the Langmuir isotherm provides a suitable fit for the Cd(II) biosorption process for each biosorbent, as the R_L values were within the range of 0 to 1. This finding underscores the applicability of the Langmuir model in describing the monolayer biosorption of Cd(II) ions.

Table 10 summarizes the $q_{\max}$ values for Cd(II) biosorption using various agricultural waste-derived biosorbents from other researchers.

FTIR Characterization and Comparison

FTIR analysis allows for the identification of different functional groups in the analyzed lignocellulosic materials: grape stalks, vine shoots, and exhausted grape pomace. The comparison of the spectra obtained before the Cd(II) biosorption process reveals significant differences in the intensity and position of the characteristic bands, providing information about the chemical structure and potential adsorptive capacity of each biomass (see Fig. 10).

The bands in the 3600–3200 cm^{-1} region, associated with the stretching vibrations of hydroxyl (–OH) groups from cellulose, hemicellulose, and lignin (Cruz-Lopes et al. 2022), appear with greater intensity in the spectrum of vine shoots, indicating a higher content of these compounds. In the case of grape stalks, the band is moderate, whereas in exhausted grape pomace, its intensity is lower, suggesting a reduction in available hydroxyl groups due to prior fermentation and distillation processes.

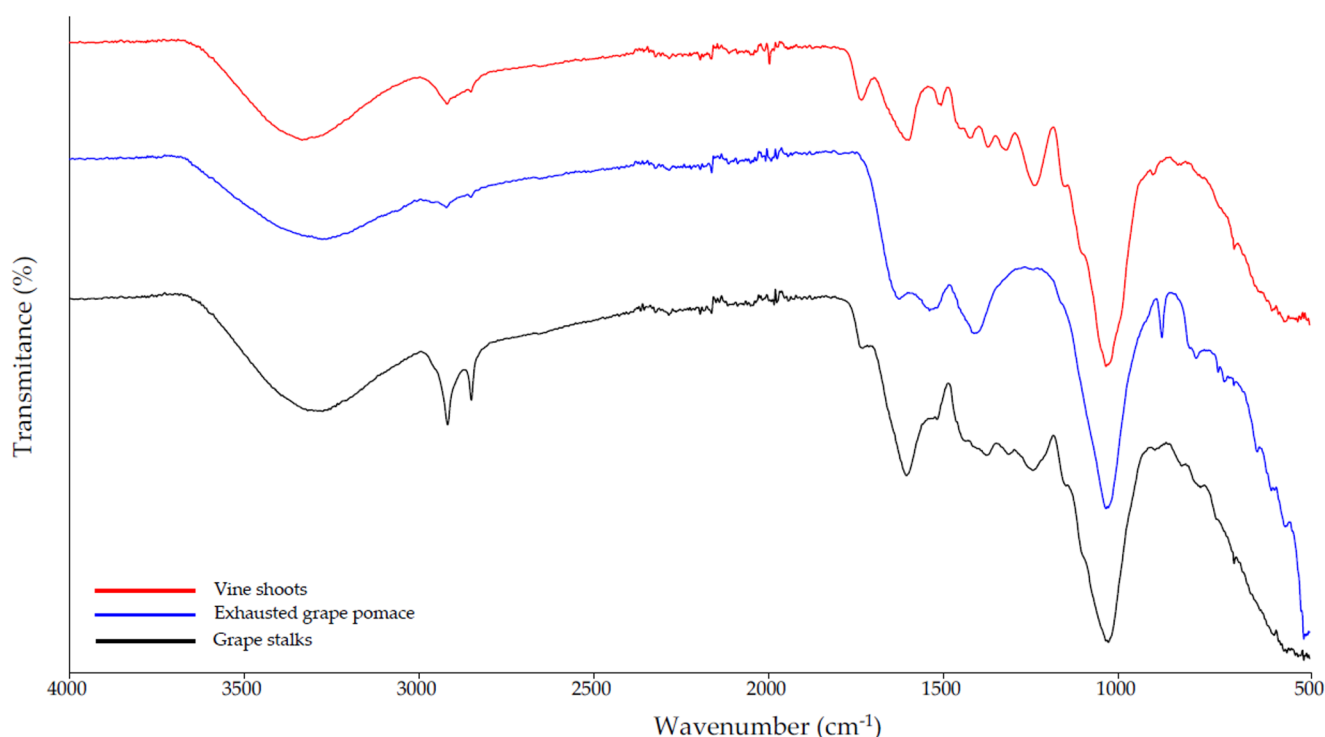


Fig. 10 FTIR spectra for vine shoots (red line), exhausted grape pomace (blue line), and grape stalks (black line) before Cd(II) biosorption process

In the 3000–2800 cm^{-1} region, where bands corresponding to aliphatic C–H bonds are observed (Volli et al. 2021), the vine shoot spectrum exhibits greater intensity compared to the other materials, indicating a higher proportion of aliphatic structures. On the other hand, in the 1750–1600 cm^{-1} region, related to the stretching vibrations of carbonyl (C=O) groups present in esters and carboxylic acids (Plöhn et al. 2021), the bands are more intense in grape stalks and vine shoots, whereas in exhausted grape pomace, they appear with lower intensity, suggesting a reduction in the content of carbonyl compounds.

The presence of lignin is evident in the 1600–1500 cm^{-1} region, corresponding to the vibrations of C=C bonds (Biswas et al. 2022). In this case, vine shoots exhibit the most intense band, indicating a higher lignin content in their structure compared to grape stalks and exhausted grape pomace.

In the 900–600 cm^{-1} region, corresponding to out-of-plane C–H vibrations (Biswas et al. 2022), vine shoots again present a more intense band than the other materials, suggesting greater structural stability due to their higher lignin content. In contrast, exhausted grape pomace exhibits less pronounced bands in this region, suggesting greater structural degradation.

SEM–EDS

The SEM–EDS images provide insight into the morphological changes of the three biosorbents before and after Cd(II) biosorption, demonstrating their effectiveness in capturing Cd(II) ions from wastewater. Figure 11a displays the surface morphology of the vine shoots (600 $\times$ magnification) before Cd(II) biosorption. It shows a structured, linear arrangement of woody fibers and parenchyma that typifies lignocellulosic biomass. Figure 11c, shows the grape stalks surface (800 $\times$ magnification). It has a similar fibrous texture, which suggest structural consistency in these biosorbents. In contrast, Fig. 11e, depicts the exhausted grape pomace (2000 $\times$ magnification), revealing a highly granular and porous surface. EDS mapping in Fig. 11b, d, and f shows the Cd(II) ions adsorbed by each biosorbent after the biosorption process, highlighted in blue. This confirms the presence and distribution of Cd(II) on the surfaces, validating the potential of these materials for metal ion retention. This SEM–EDS analysis thus underscores the effectiveness of these wine wastes as biosorbents for Cd(II) removal. Their morphological structure makes them suitable for wastewater treatment applications aimed at heavy metal remediation.

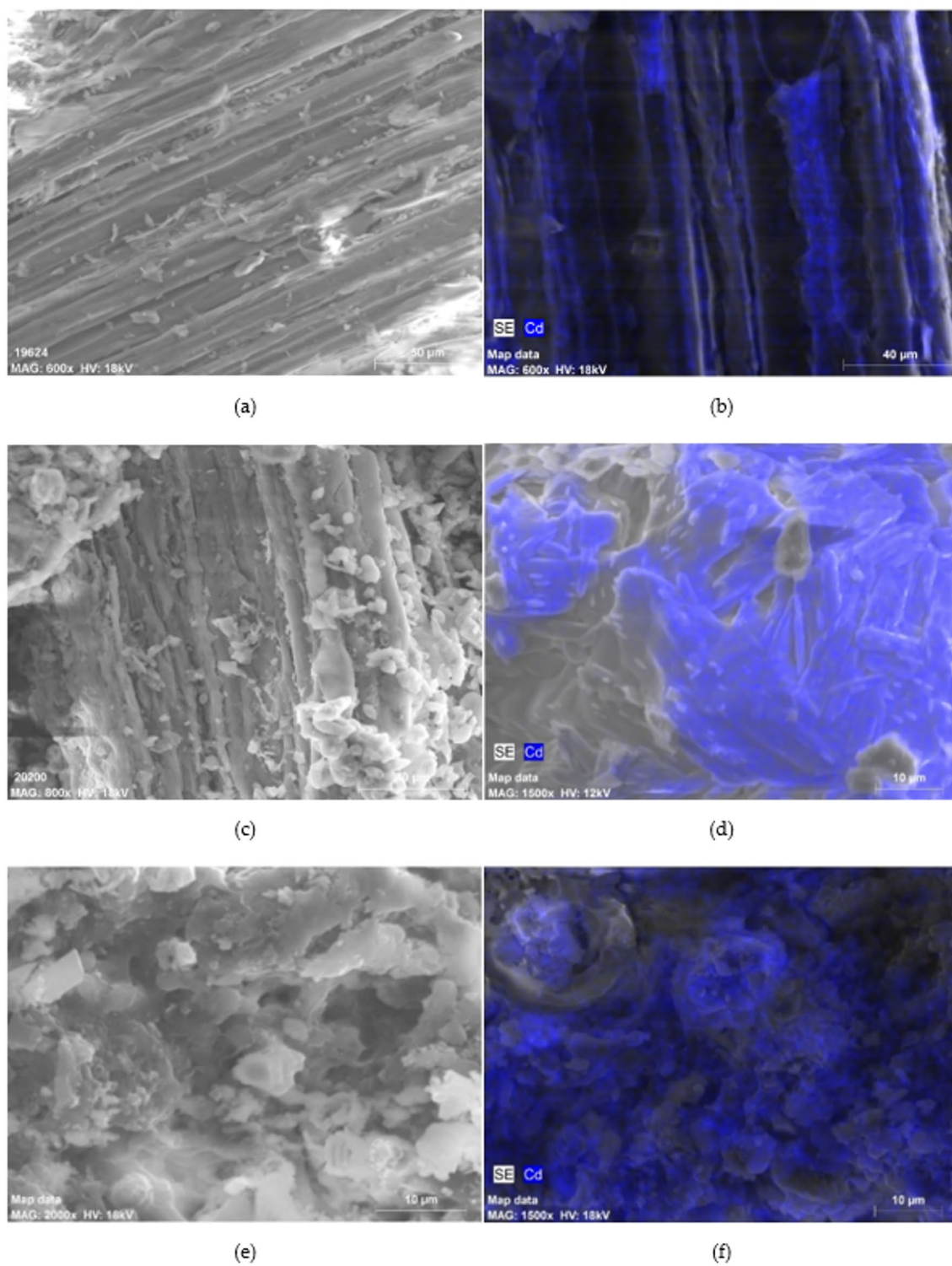


Fig. 11 SEM–EDS images of raw biomass (a) vine shoots, (c) grape stalks, and (e) exhausted grape pomace, and (b), (d), and (f) after Cd(II) biosorption process with Cd(II) ions highlighted in blue

Conclusions

This study evaluated the biosorption of Cd(II) using vine shoots, grape stalks, and exhausted grape pomace. Two optimization scenarios were analyzed: one focused on maximizing Cd(II) removal and the other aimed at maximizing removal while also reducing resource and energy use. In the first scenario, all biosorbents achieved very high removal efficiencies (around 99.95%). However, grape stalks were more resource-efficient, demonstrating lower energy consumption (E_{Cons}) compared to vine shoots and exhausted grape pomace. In the second scenario, exhausted grape pomace showed a decrease in Cd(II) removal efficiency ($\%Rem_{EGP}=79.29\%$) due to the lower pH (2.5), while grape stalks and vine shoots maintained higher removal efficiencies ($\%Rem_{GS}=96.95\%$ and $\%Rem_{VS}=99.94\%$). The kinetic study revealed that all three biosorbents followed the pseudo second order model, indicating that Cd(II) biosorption was primarily driven by chemisorption. Grape stalks exhibited the fastest adsorption kinetics, with the highest rate constant ($k_2=1.9369$ g/mg·min), suggesting the most efficient adsorption process among the three biosorbents. In the isothermic study, the Langmuir model best described the Cd(II) biosorption for all biosorbents, indicating monolayer adsorption on the biosorbent surfaces. Grape stalks also demonstrated the highest maximum adsorption capacity ($q_{max}=77.52$ mg/g), followed by vine shoots ($q_{max}=40.82$ mg/g) and exhausted grape pomace ($q_{max}=26.67$ mg/g). Considering the combined results of adsorption capacity, kinetic performance, and life cycle assessment, grape stalks proved to be the most effective biosorbent for Cd(II) removal. Their high efficiency, fast adsorption kinetics, and lower environmental impact under optimized conditions make them the most suitable and sustainable option for wastewater treatment among the three biosorbents evaluated. Future research will aim to validate the performance of these lignocellulosic biosorbents in real wastewater, where the presence of organic matter and competing ions may influence removal efficiency. Additionally, the reusability and long-term stability of the biosorbents will be assessed through multiple adsorption–desorption cycles, accompanied by structural and physicochemical characterization before and after regeneration. These investigations will be essential to confirm the practical viability of these biosorbents for real-world wastewater treatment applications.

Author Contributions Celia Sabando-Fraile: conceptualization; data curation; formal analysis; investigation; methodology; software; validation; visualization; roles/writing—original draft; writing—review and editing. Marina Corral-Bobadilla: conceptualization; data curation; formal analysis; funding acquisition; investigation; methodology; resources; supervision; validation; visualization; roles/writing—original draft; writing—review and editing. Susana Cabredo Pinillos:

formal analysis; data curation; methodology; writing—review and editing. Félix Gallarta González: formal analysis; data curation; methodology; writing—review and editing.

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Data Availability The datasets used during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of Interest The authors declare no competing interests.

Ethics Approval and Consent to Participate Not applicable.

Consent for Publication The manuscript is submitted with the consent of all authors.

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