

Cocoa husk bioadsorption: A sustainable solution for lubricant wastewater treatment

Bioadsorción de cáscara de cacao: Una solución sostenible para el tratamiento de aguas residuales lubricantes

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ABSTRACT

*This research evaluated the effectiveness of cocoa husk (*Theobroma cacao* L.) as a bioadsorbent for removing heavy metals from an auto service lubricant wastewater. To produce flour particles with sizes of 850 μm and 1 mm, cocoa husks were dehydrated, ground and sieved. Pb and Al levels in the effluent were initially 0.017 ppm and 1.016 ppm, correspondingly. Four different treatments, each with a different particle diameter (850 μm and 1 mm) and dosage of cocoa husk (10 and 15 g), were evaluated using a Completely Randomized Design (CRD) with a 2² factorial arrangement. These tests were carried out with a pH of 4 and a 6-hour exposure period. The ability of each treatment to extract Pb and Al from the wastewater was remarkable. The greatest removal effectiveness, however, was shown by treatment T2, which used 15 g of 850 μm cocoa husk flour and reduced the concentration of Pb by 94.12% and the concentration of Al by 46.49%. These results indicate cocoa husk flour's significant potential as a bioadsorbent for the efficient treatment of wastewater polluted with heavy metals.*

Keywords: Bioadsorption, heavy metal removal, wastewater, cocoa husk flour.

RESUMEN

Esta investigación evaluó la efectividad de la cáscara de cacao (*Theobroma cacao* L.) como bioadsorbente para la eliminación de metales pesados de aguas residuales de lubricantes de autoservicio. Para producir partículas de harina con tamaños de 850 μm y 1 mm, se deshidrataron, molieron y tamizaron cáscaras de cacao. Los niveles de Pb y Al en el efluente fueron inicialmente de 0,017 ppm y 1,016 ppm, respectivamente. Se evaluaron cuatro tratamientos diferentes, cada uno con diferente diámetro de partícula (850 μm y 1 mm) y dosis de cáscara de cacao (10 y 15 g), mediante un Diseño Completamente Aleatorio (DCA) con arreglo factorial 2². Estas pruebas se realizaron con un pH de 4 y un período de exposición de 6 horas. La capacidad de cada tratamiento para extraer Pb y Al de las aguas residuales fue notable. Sin embargo, la mayor efectividad de eliminación la mostró el tratamiento T2, que utilizó 15 g de harina de cáscara de cacao de 850 μm y redujo la concentración de Pb en un 94,12% y la concentración de Al en un 46,49%. Estos resultados indican el importante potencial de la harina de cáscara de cacao como bioadsorbente para el tratamiento eficiente de aguas residuales contaminadas con metales pesados.

Palabras clave: Bioadsorción, eliminación de metales pesados, aguas residuales, harina de cáscara de cacao.

INTRODUCTION

Wastewater generated by lubrication plants poses a severe environmental threat due to its high concentration of heavy metals, including lead (Pb), cadmium (Cd), chromium (Cr) and zinc (Zn) (Díaz et al., 2018); (Guilcamaigua et al., 2019). These metals originate from the extensive use of lubricants in the automotive industry and pose significant risks to both human health and ecosystems (Sánchez, 2018). Moreover, the presence of oily waste compounds the issue by reducing oxygen levels in water bodies and intensifying overall water pollution (Manzanarez, 2022). The absence of effective treatment systems in Ecuadorian lubrication plants has led to the contamination of urban sewage networks, aggravating the country's environmental challenges (García et al., 2019) (Guilcamaigua et al., 2019) (Guilcamaigua et al., 2019) (Guilcamaigua et al., 2019).

The proliferation of lubrication establishments in Manabí has exacerbated the issue, with wastewater contaminating both urban and rural water bodies (Torres & Reyes, 2021). This problem is endemic to Ecuador, where inadequate wastewater treatment infrastructure and practices fail to address pollutants from industrial sources like vehicle lubrication. The consequent disregard for environmental regulations has led to a surge in diseases linked to exposure to these toxic effluents (Robalino, 2021).

Cocoa husk, a readily available agricultural byproduct in Ecuador due to its robust cocoa industry, offers a promising and cost-effective solution for mitigating heavy metal pollution in wastewater. Previous studies have demonstrated the efficacy of cocoa husk in removing metals such as Pb, cadmium, nickel, and zinc (Pérez et al., 2020); (Macías, 2021). Moreover, its inherent properties, including thermal stability and corrosion resistance, coupled with its economic accessibility, make it a suitable candidate for industrial applications like activated carbon production (Marín & López, 2019).

Cocoa husk bioadsorption emerges as a sustainable solution for treating lubricant wastewater, directly aligning with the Sustainable Development Goals (SDG) 6: clean water and sanitation. By effectively

removing harmful pollutants, this approach safeguards water resources, enhances public health, and promotes responsible consumption and production (SDG 12). Moreover, it drives innovation in wastewater treatment technologies, contributing to SDG 9: Industry, Innovation and Infrastructure. This research underscores the potential of bio-based solutions in addressing pressing environmental challenges and fostering sustainable development. Given these advantages, this research aims to evaluate the effectiveness of cocoa husk (*Theobroma cacao* L.) as a bioadsorbent for removing heavy metals from lubricant wastewater.

METHODOLOGY

Wastewater samples

Composite wastewater samples were collected from an auto service center in Calceta, Manabí, Ecuador, following the (Instituto Ecuatoriano de Normalidad [INEN], 2013) standard. Daily samples were obtained over six days in sterilized 4 L polypropylene bottles, resulting in a 24 L composite sample. A 13 L subsample was subsequently extracted for analysis. The concentrations of Pb and aluminum (Al), the target heavy metals for this study, were determined using Inductively Coupled Plasma (ICP) spectrometry (López & Pincay, 2020).

Biomass processing

Cocoa husks were collected and processed following the methodology of (Pérez et al., 2020). The husks were initially washed with potable water to remove impurities and the endocarp was subsequently removed to reduce mucilage content (Tapia, 2015). After cutting the husks into approximately 5 mm pieces and rinsing with distilled water, they were dehydrated at 70 °C for 24 hours. The dried husks were then ground using a Corona mill and sieved to obtain 200 g of cocoa flour with particle sizes of 1 mm and 850 µm, respectively, resulting in a total of 400 g of processed cocoa husk.

Experimental design

A Completely Randomized Design (CRD) with a 2x2 factorial arrangement was applied to analyze the impacts of two components, each having two levels. This design produced four separate treatments, each of which was reproduced three

times, for a total of twelve experimental units (table 1). The key experimental parameters were regulated as follows: 1) the pH of the solution was maintained at 4, based on research showing that metals adhere better in acidic settings (Sánchez, 2018); and 2) the sample agitation period was 6 minutes at 100 rpm, followed by a 6-hour resting contact time (Banchon et al., 2017).

Table 1. Experimental design applied.

Treatments	Nomenclature	Particle size	Husk quantity (g)
T1	A1B1	850 um	10
T2	A1B2	850 um	15
T3	A2B1	1 mm	10
T4	A2B2	1 mm	15

Bioadsorption efficiency

The percentage of contaminant removal was determined using the methodology outlined by (Rodríguez-Arellano et al., 2021) and calculated according to equation 1.

$$\%removal = \frac{vip - vfp}{vip} \times 100$$

Where:

vip = Initial value of the heavy metal

vfp = Final value of the heavy metal

After gathering data, InfoStat software was used to perform analysis of variance (ANOVA) in order to identify statistically significant differences between the treatment means, which were represented in bar graphs.

RESULTS AND DISCUSSION

High levels of Pb (0.017 mg L⁻¹) and Al (1.016 mg L⁻¹) were found in untreated effluent from an auto service center, both of which were beyond set legal limits in Ecuador. These results are in line with other investigations that show substantial heavy metal pollution in industrial waste water, including those conducted by Castillo (2015) and Aguas (2022). Aquatic ecosystems are seriously threatened by these contaminants' persistence and the ineffective removal capabilities of traditional wastewater treatment facilities, as Quintana & Jaimes (2019) point out.

The bioadsorption of Pb and Al was highly effective with treatment T2. It was successful in removing

Pb to levels that were non-detectable (ND) and 0.001 ppm. The three replicates (R1, R2 and R3) had clearance values for Al of 0.464 ppm, 0.529 ppm, and 0.638 ppm, in that order. These results are consistent with those of Soto (2019), who investigated the adsorption capacities of both chemically and unmodified cocoa husk for the removal of metals from mine effluent, observing starting values of 1.23 ppm for Al and 0.65 ppm for Pb. The outcomes of the experiment efficacy are attributed to the acidic atmosphere in which it was conducted. According to Duany et al. (2022) and Morales (2018), bioadsorption of heavy metals is more effective at low pH because of the high concentration of protons (H⁺), which allows metal ions to compete for binding sites on the bioadsorbent.

Due to its abundance of fibrous components, such as cellulose, hemicellulose, pectins, and lignin, cocoa husk is an efficient bioadsorbent (Eletta et al., 2020), as demonstrated by the high removal rates of Pb and Al in Treatment T2. It is well recognized that these components increase adsorption capacity (Maya, 2018). The results obtained with cocoa husk support its potential, even if Condor and Maza (2020) showed that a comparable material, coconut shell, was successful with Pb removal rates ranging from 94% to 96.40%. Cocoa husk offers itself as a practical, affordable, and extremely successful substitute for conventional techniques for the removal of heavy metals from wastewater because of its similar qualities.

As presented in table 2, the lowest residual Pb levels were consistently obtained by treatment T2, which began with a Pb concentration of 0.017 ppm. T2R2 reached 0 ppm, T2R1 at 0.001 ppm, and T2R3 at 0.002 ppm. However, as seen by the small decreases in replicates like T3R1 (0.015 ppm), T3R3 (0.015 ppm), and T4R1 (0.015 ppm), treatments T3 and T4 showed limited effectiveness in Pb elimination. The substantial variation in Pb removal effectiveness clearly indicates that T2's special treatment circumstances or features greatly improve its capacity for bioadsorption.

Table 2. Removal rate for Pb.

Treatment	Final concentration (ppm)	Individual removal rate (%)	Treatment removal rate (%)
T1 R1	0.000 (ND)	100	
T1 R2	0.003	82.35	91.18 (T1)
T1 R3	0.0015	91.18	
T2 R1	0.001	94.12	
T2 R2	0.000 (ND)	100	94.12 (T2)
T2 R3	0.002	88.24	
T3 R1	0.015	11.76	
T3 R2	0.007	58.82	27.45 (T3)
T3 R3	0.015	11.76	
T4 R1	0.015	11.76	
T4 R2	0.011	35.29	45.10 (T4)
T4 R3	0.002	88.24	

The analysis presented in table 3 shows no statistically significant differences in particle size or in the interaction between factors A and B. This finding is consistent with Daza et al. (2019), who note that p-values exceeding 0.05 suggest a lack of significant differences. However, a significant difference was observed for the cocoa husk factor, with a p-value of less than 0.05.

Table 3. Analysis of variance (Type III) of the interaction between factors A and B for Pb.

V.F.	CS	FD	MC	F	P-value
Model	0.00028	3	0.0001	5.599	0.02
	9		0		3
Particle size (A)	0.00000	1	0.0000	0.533	0.48
	9		1		6
Amount of cocoa husk (B)	0.00027	1	0.0002	15.99	0.00
	6		8	2	4
Factor A*Factor B	0.00000	1	0.0000	0.272	0.61
	5		1		6
Error	0.00013	8	0.0000		
	8		2		
Total	0.00042	11			
	7				

On the other hand, the lowest Al removal effectiveness was shown by treatments T4R4 (0.800 ppm), T4R2 (0.731 ppm) and T3R1 (0.722 ppm) (table 4). The significant performance difference indicates that certain remedies, particularly T2 and T3, are significantly more effective at reducing Al levels. This increased effectiveness is probably due to certain treatment circumstances or traits that boost their capacity to adsorb Al. Therefore, these results highlight how important it is to use efficient techniques for Al removal optimization.

Table 4. Removal rate for Al.

Treatment	Final concentration (ppm)	Individual removal rate (%)	Treatment removal rate (%)
T1 R1	0.639	37.11	
T1 R2	0.653	35.73	36.29 (T1)
T1 R3	0.65	36.02	
T2 R1	0.464	54.33	
T2 R2	0.529	47.93	46.49 (T2)
T2 R3	0.638	37.20	
T3 R1	0.722	28.94	
T3 R2	0.607	40.26	38.12 (T3)
T3 R3	0.557	45.18	
T4 R1	0.619	39.07	
T4 R2	0.731	28.05	29.46 (T4)
T4 R3	0.8	21.26	

There were no statistically significant variations between treatments for Al reduction, according to the multiple comparison test of means ($p > 0.05$) (table 5). This result is consistent with that of Daza et al. (2019). The very similar adsorption findings for aluminum across treatments, with final concentrations clustering around values like 0.639 ppm, 0.619 ppm, and 0.638 ppm, are responsible for the absence of considerable variance.

Table 5. Analysis of Variance (Type III) of the Interaction between Factors and B for Al.

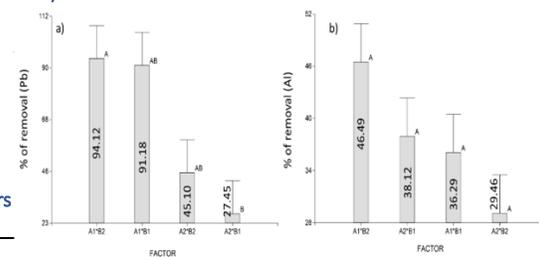
V.F.	CS	FD	MC	F	p-value
Modelo	0.0456	3	0.0152	2.6109	0.124
Particle size (A)	0.0002	1	0.0002	0.0316	0.863
Amount of cocoa husk (B)	0.0179	1	0.0179	3.0685	0.118
Factor A*Factor B	0.0276	1	0.0276	4.7325	0.061
Error	0.0466	8	0.0058		
Total	0.0922	11			

Figure 1 (a) illustrates that Pb removal effectiveness varies among treatments, following this pattern: T2 (94.12%) > T1 (91.18%) > T4 (45.10%) > T3 (27.45%). This finding aligns with the

research by Govea & Delgado (2019), which highlights that cocoa husk's high lignin and cellulose content, along with polar functional groups such as alcohols and aldehydes, contributes to its effectiveness. These functional groups act as electron pair donors, forming complexes with metal ions and thereby enhancing the adsorption process.

Regarding Al, figure 1 (b) shows that while no statistically significant differences are observed between treatments, T2 performs the best with a removal rate of 46.49%, followed by T3 (38.12%), T1 (36.29%), and T4 (29.46%). Although these results do not reveal significant variations in Al removal efficiency among the treatments, they suggest that T2 is the most effective option. The absence of significant differences in relation to sample size, as noted by (Daura et al., 2022), further supports the observed trend and indicates that the performance differences may not be solely attributable to statistical variability but could also reflect inherent treatment efficacy (Eletta et al., 2020).

Figure 1. Multiple testing of means between treatments: a) Pb; b) Al. Means with common letter are not significantly different ($p > 0.05$).



Microprecipitation and cation exchange are the main processes that propel the adsorption of metals like lead onto cocoa pod husk. These are made possible by the presence of essential components in the structure of the bioadsorbent, such as silicon (Si), potassium (K) and calcium (Ca) (Tejada-Tovar et al., 2022; Oggero et al., 2021). Treatment T2's increased amount of adsorbent material is responsible for its better performance. This higher dose extends the metal ion residence period and improves the availability of active adsorption sites. This eventually contributes to increased adsorption efficiency by facilitating more

intimate contact between the solid and liquid phases and more efficient ion diffusion (Rodríguez-Arellano et al., 2021).

CONCLUSIONS

This research highlights the impressive efficacy of bioadsorption using cocoa husk, especially under the T2 treatment conditions, which utilized 15 g of cocoa husk with a particle size of 850 µm. The process achieved a notable 94% reduction in Pb concentration and a 46.49% reduction in Al concentration from wastewater sourced from an auto service wastewater. These significant reductions underscore the potential of cocoa husk as an effective and sustainable method for heavy metal removal.

Cocoa husk not only offers a financially viable substitute for traditional treatment techniques but also promotes environmental sustainability. Optimizing the quantity and size of the bioadsorbent can improve performance, as seen by the high adsorption efficiency identified, especially with the T2 treatment settings. In line with the more general objectives of resource recovery and sustainable waste management, this strategy presents a viable way to control heavy metal contamination.

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