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Removal of metals from water of Yarinacocha Lagoon with activated carbon from cocoa pod husks[1]

Remoção de metais da água da Lagoa Yarinacocha com carvão ativado da casca da vagem de cacau

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HIGHLIGHTS:

Organic residues from cocoa constitute a potential alternative for their conversion into activated carbon.
The microporous nature of activated carbon provides efficiency in the removal of metals from water.
Carbonization of residues at low temperatures and activation at high temperatures increase the yield of activated carbon.

ABSTRACT: The problem addressed is the contamination of the Yarinacocha Lagoon water by heavy metals and poor use of agricultural residues. It was manufactured activated carbon from cocoa (*Theobroma cacao* L.) pod husks and determined its adsorbent effect in removing polluting metals from the waters of the Yarinacocha Lagoon. The response surface methodology was applied with factorial designs 3^3 and 3^2, with three replicates to optimize obtaining the adsorbent and measure its effectiveness in metal removal. The modeling of the pyrolysis process resulted in 17.27 g of activated carbon from 295.72 g of dry pod husks, optimal with the following optimal parameters: 150 °C as activation temperature, 450 °C as carbonization temperature, and 2.5 hours as modification time. This resulted in effective removal of pollutant metals (aluminum: 91.43%, copper: 75%, iron: 58.33% and zinc: 58.33%), from waters samples demonstrating that it is possible to manufacture activated carbon from cocoa pod husks, with an adsorbent potential to remove metals from the waters of the Yarinacocha Lagoon.

Key words: *Theobroma cacao* L., adsorbent, response surface methodology, carbonization, pyrolysis

RESUMO: O problema tratado é a contaminação da água da Lagoa Yarinacocha por metais pesados e o uso inadequado de resíduos agrícolas. Foi fabricado carvão ativado a partir de cascas de cacau (*Theobroma cacao* L.) e foi determinado seu efeito adsorvente na remoção de metais poluentes das águas da Lagoa Yarinacocha. A metodologia da superfície de resposta foi aplicada com experimentos fatoriais 3^3 e 3^2, com três repetições para otimizar a obtenção do adsorvente e medir sua eficácia na remoção de metais. A modelagem do processo de pirólise, resultou em 17,27 g de carvão ativado a partir de 295,72 g de cascas de cápsulas secas, ótimo com os seguintes parâmetros ótimos: 150 °C como temperatura de ativação, 450 °C como temperatura de carbonização, e 2,5 horas como tempo de modificação. Isto resultou na remoção efetiva de metais poluentes (alumínio: 91,43%, cobre: 75%, ferro: 58,33% e zinco: 58,33%), de amostras de água demonstrando que é possível fabricar carvão ativado a partir de cascas de casca de cacau, com um potencial adsorvente para remover metais das águas da Lagoa Yarinacocha.

Palavras-chave: *Theobroma cacao* L., adsorvente, metodologia de superfície de resposta, carbonização, pirólise

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Introduction

The growing problem of water contamination from wells, streams, rivers, lakes and lagoons awaken the interest of scientists to investigate economically, socially and environmentally feasible technological solutions. Lignocellulosic residues can be converted into an effective adsorbent by transforming them by pyrolysis (Torres et al., 2021) into activated carbons with high adsorption efficiency because of their effect on their microstructure (Kozyatnyk et al., 2021). According to Liu et al. (2021), its fibers, which contain cellulose, hemicellulose, and lignin, are comprised of C (41%), O (52%), and H (6%). Activation by pyrolysis allows carbon atoms to be, reorganized and, form carbon structures that increase the C/O and C/H ratios of activated carbon (Liu et al., 2021). Iamsaard et al. (2022) managed to remove nickel, zinc, and copper from wastewater samples using activated carbon from pineapple leaves (*Ananas comosus*), thus demonstrating the usefulness of pyrolysis activation.

Cocoa production in the Ucayali region of Peru has increased because, in the last years. Between January and March 2021, the export of 2,163 tons of cocoa grains were exported (MIDAGRI, 2021), generating 8.652 tons of pod husks, which contributed to water contamination with solid waste. The Ministry of Environment (MINAM, 2013) has reported increasing contamination by iron, nitrates, lead, aluminum, mercury, and other heavy metals in groundwater and surface water. The objective of this study was to manufacture activated carbon from cocoa pod husks (*Theobroma cacao* L.) and determine its ability to absorb and remove metals present in water samples from the Yarinacocha Lagoon, Ucayali, Peru.

Materials and Methods

Activated carbon was manufactured in the General Chemistry Laboratory of the National University of Ucayali -UNU, Pucallpa City, Ucayali Region, Peru, (coordinates 8° 24' 48" S, 74° 34' 8" W; altitude 154 m above sea level (SENAMHI, 2019). Adsorption tests were conducted in the BIOVITAL laboratory in Huánuco City, Peru, (coordinates 9° 55' 50.2" S, 76° 14' 5" W; altitude: 1,894 m above sea level (Gobierno Regional de Huánuco, 2015), between October 2015 and March 2016.

The biological material included cocoa pods of clone CCN51 (Figure 1), purchased from the Pucallpa Wholesale Market, located at km 6, Mz. B2 Lot. 1b Municipal urban enabling.

The manufacture of activated carbon followed the flow chart established by Tiegam et al. (2021), with modifications as follows: cobs were chopped, seeds and mucilage were removed, selected, and classified. Approximately 300 g were chopped into 5 mm pieces and thermally modified at 150, 200 and 250 °C for 1.5, 2.0 and 2.5 hours, before being carbonized at 450, 500 and 550 °C for one hour in a stainless steel reactor and muffle furnace. Charcoal was ground to particles of 180 µm, and activated charcoal was washed with distilled H_2O until the black color was removed. It was then dried at 150 °C for one hour, packed in a hermetically sealed Ziploc bag and stored until later use. Response parameters are shown in Table 1.

Figure 1. Cocoa fruits, clone CCN51, used in this study

Table 1. Coding, levels and values of the evaluated factors for the activated carbon preparation

Parameters	Unit	Code	-1	0	1
Thermal activation temperature	°C	X_1	150	200	250
Carbonization temperature	°C	X_2	450	500	550
Modification time	h	X_3	1.5	2.0	2.5

The response variable (Y_1) is the amount of activated carbon, generating Eq. 1:

$$\begin{aligned} Y_1 &= B_0 + B_1X_1 + B_2X_2 + B_3X_3 + \\ &+ B_{11}X_1^2 + B_{12}X_1X_2 + B_{13}X_1X_3 + \\ &+ B_{22}X_2^2 + B_{23}X_2X_3 + B_{33}X_3^2 \end{aligned} \quad (1)$$

where:

Y_1 - response variables (amount of activated carbon);
B_0 - coefficient of the constant;
B_1, B_2 and B_3 - linear coefficients;
B_{12}, B_{13} and B_{23} - binary interaction coefficients;
B_{11}, B_{22} and B_{33} - coefficients quadratic; and,
X_1, X_2, and X_3 - coded values of the activated carbon preparation variables.

Sampling of water from the Yarinacocha lagoon was carried out 50 m from Puerto Callao (Figure 2), in glass bottles with hermetic lids, which were covered with aluminum foil and immediately stored in a thermal container with ice to keep them refrigerated.

Adsorption tests were carried out using the atomic absorption spectroscopy technique, recommended by Baird et

Figure 2. Sampling of water in the Yarinacocha Lagoon, in front of Puerto Callao-Ucayali-Peru

Table 2. Coding, levels and values of the evaluated factors for metal removal from water samples with activated carbon

Factors	Unit	Code	-1	0	1
Activated carbon quantity	g	X_1	1	3	6
Shaking time	min	X_2	30	60	90

al. (2017). Water samples of 200 mL each were taken from the Yarinacocha Lagoon. The water had initially aluminum (Al^{+3}), copper (Cu^{+2}), iron (Fe^{+2}), and zinc (Zn^{+2}) concentrations of 0.035, 0.04, 0.36 and 0,0024 respectively mg L^{-1} of respectively; 1, 3, and 6 g of washed and dried activated carbon was added, then filtered. Samples were kept refrigerated in hermetically sealed and coded glass jars. The removal of metals Al^{+3}, Cu^{+2}, Fe^{+2} and Zn^{+2} in the water samples was measured by flame atomic a constructed absorption, using a UNICAM-989 spectrophotometer, for which a calibration curve was made with a minimum of four concentrations and a reagent blank in the linear range shown for each element (Table 2).

The response variable ($Y_{Al, Cu, Fe, Zn}$), represents the removal of metals, generating Eq. 2:

$$\begin{aligned} Y_{Al,Cu,Fe,Zn} &= B_0 + B_1X_1 + B_2X_2 + \\ &+ B_{11}X_1^2 + B_{12}X_1X_2 + B_{22}X_2^2 \end{aligned} \quad (2)$$

where:

$Y_{Al,Cu,Fe,Zn}$ - response variables (removal of Al^{+3}, Cu^{+2}, Fe^{+2} and Zn^{+2});

B_0 - constant coefficient;

B_1 and B_2 - linear coefficients;

B_{12} - binary interaction coefficient;

B_{11} and B_{22} - quadratic coefficients; and,

X_1 and X_2 - coded values of the variables responsible for metal removal.

To obtain activated carbon, a 3^3 factorial design with three replicates was utilized, and, to evaluate the adsorbent effectiveness of activated carbon, in the removal of metals from the water samples from the Yarinacocha Lagoon, a factorial design of 3^2 with three replicates was used.

The data obtained were subjected to statistical analysis using response surface methodology with Statgraphics CENTURION XIX software, version 19.1.2 (StatPoint 2020).

Results and Discussion

The analysis of variance of the amount of activated carbon as a function of the thermal activation temperature, carbonization temperature and modification time is shown in Table 3.

No significant effects of the evaluated factors were found.

The methodology indicated that the optimal amount of activated carbon was 17.27 g, obtained at 150 °C activation temperature, 450 °C carbonization temperature, and with 2.5 hours of modification.

The use of plant residues and their conversion into activated carbon was investigated by Çeçen (2014), who attributed the adsorption of heavy metals to their ionic and polar nature, because activated carbon, by presenting acid oxides on its surface, become hydrophilic and polar, facilitating the adsorption of ionic and polar species, such as Fe^{+2}, Cu^{+2}, Zn^{+2} and Al^{+3}. Bhattacharjee et al. (2020), indicated that agricultural waste materials, such as fruit residues, have wide ranging use applications for the sequestration of dyes and heavy metals from wastewater. According to Lin et al. (2021), obtaining activated carbon is based on two key processes: carbonization of the raw material and activation of the carbonized product to achieve high performance. With respect to carbonization, the physical method used by Sakhiya et al. (2021) was performed at 700 °C by pyrolysis (thermal decomposition of a solid in an inert atmosphere or without oxygen), which differs from the 550 °C used in the present study. Bedane et al. (2019), who used an activation temperature of 200 °C, indicated that the moderate activation allowed for the formation of activated carbon with a very narrow micropore size distribution and a practically homogeneous microporous structure.

Table 3. Analysis of variance of the amount of activated carbon obtained

Source of variation	SS	DF	MS	F-value	p-value
A: Thermal activation temperature	6.956	1	6.956	0.58	0.4586^{ns}
B: Carbonization temperature	13.694	1	13.694	1.13	0.3021^{ns}
C: Modification time	51.376	1	51.376	4.25	0.0549^{ns}
AA	0.069	1	0.069	0.01	0.9407^{ns}
AB	33.333	1	33.333	2.76	0.1152^{ns}
AC	4.613	1	4.613	0.38	0.5450^{ns}
BB	35.268	1	35.268	2.92	0.1059^{ns}
BC	36.820	1	36.820	3.04	0.0990^{ns}
CC	31.175	1	31.175	2.58	0.1268^{ns}
Error	205.579	17	12.093		
CV (%)	34.9				

^{ns} - Not significant; SS - Sum of squares; DF - Degrees of freedom; MS - Mean square; CV - Coefficient of variation

The statistical analysis of the data regarding the adsorbent effect of activated carbon in the removal of Al^{+3}, Cu^{+2}, Fe^{+2} and Zn^{+2}, present in the water samples as well and their significance, are shown in Table 4.

There was a correlation for the removal of Cu^{+2}, Fe^{+2} and Zn^{+2} with the amount of activated carbon used, and all metals, were correlated with shaking time, at $p \leq 0.05$.

The coefficients of determination (R^2) of the models varied from 0.6782 to 0.9557 (Table 5), which is a good fit considering that the tests were conducted at the laboratory. The type of experimental design allowed us to observe the influence of the independent variables on the removal of the metals present in the water samples of Yarinacocha Lagoon.

Table 4. Summary of the analyses of variance for the removal of Al^{+3}, Cu^{+2}, Fe^{+2} and Zn^{+2}

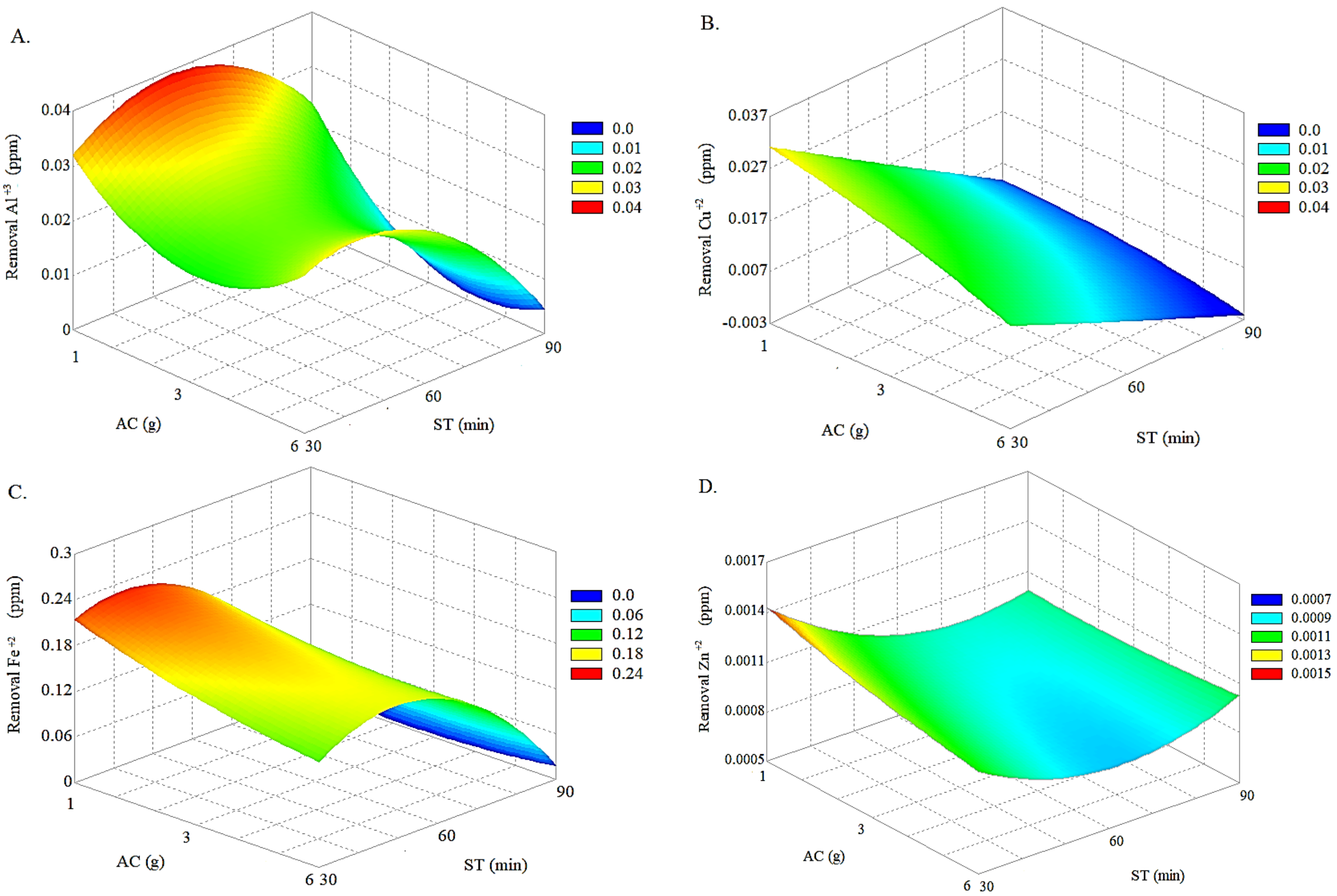
Sources of variation	p-value			
	Al^{+3}	Cu^{+2}	Fe^{+2}	Zn^{+2}
A: Activated carbon quantity	0.2336^{ns}	1.0E-4*	0.0212*	0.1138^{ns}
B: Shaking time	1.3E-15*	1.0E-10*	1.0E-10*	1.6E-3*
AA	4.3E-3*	0.9672^{ns}	0.2804^{ns}	0.6179^{ns}
AB	0.0679^{ns}	0.1418^{ns}	4.4E-3*	0.0372*
BB	1.0E-4*	0.9176^{ns}	1.0E-10*	5.0E-4*
CV (%)	28.22	41.76	24.57	44.32

^{ns}, and * - Not significant, and significant respectively, $p \leq 0.05$, by the F test; CV - Coefficient of variation

Table 5. Determination coefficients (R^2) and fitted models

Metal removed	R^2	Equation	
Al^{+3}	0.8085	$\hat{y}_{Al^{+3}} = 0.003936 - 0.007058^{ns}X_1 + 0.00133^{*}X_2 + 0.001278^{*}X_1^2 - 0.000012^{*}X_2^2$	(3)
Cu^{+2}	0.9020	$\hat{y}_{Cu^{+2}} = 0.0456503 - 0.0032462^{*}X_1 - 0.0004705^{*}X_2$	(4)
Fe^{+2}	0.9557	$\hat{y}_{Fe^{+2}} = 0.061239 - 0.029772^{*}X_1 + 0.008049^{*}X_2 + 0.000243^{*}X_1X_2 - 0.000095^{*}X_2^2$	(5)
Zn^{+2}	0.6782	$\hat{y}_{Zn^{+2}} = 0.00232152 - 0.0001169^{ns}X_1 - 0.00003643^{*}X_2 + 0.0000011^{*}X_1X_2 + 0.00000002^{*}X_2^2$	(6)

^{ns} and * - Not significant, and significant respectively, $p \leq 0.05$, by the F test; R^2 - Coefficient of determination



ST - Shaking time; AC - Activated carbon

Figure 3. Removal by activated carbon action of Al^{+3} (A), Cu^{+2} (B), Fe^{+2} (C), Zn^{+2} (D) in function of shaking time and activated carbon quantity

The adjusted model determined an optimal removal value of 0.032 ppm for Al^{+3}, with 1.0 g of activated carbon for a stirring time of 52.10 min (Eq. 3); similarly it determined an optimal removal value of 0.03 ppm for Cu^{+2}, with 1.0 g of activated carbon for a stirring time of 30.0 min (Eq. 4); 0.21 ppm for Fe^{+2}, with 1.0 g of activated carbon for a stirring time of 43.48 min (Eq. 5); and, 0.0014 ppm of Zn^{+2}, with 1.0 g of activated carbon for a stirring time of 30.0 min (Eq. 6), (Figure 3).

Table 6 shows the adsorbent effect of activated carbon, in the removal of metals from water samples, whose values resulted from the optimization of the models, carried out under ideal conditions, which established the use of 1 g of activated carbon, to remove each metals.

According to Popoola et al. (2022), activated carbon from *Citrullus lanatus* peel, has proven to be an effective adsorbent in the removal of Pb^{+2}, Zn^{+2}, Fe^{+2} and Cu^{+2} in water, due to its presence of metal oxides, active functional groups, and large surface area. Alslaibi et al. (2014) experimented with adsorption of the heavy metals Fe^{+2}, Pb^{+2} y Cu^{+2} present in wastewater with activated carbon made from olive stone (*Olea europaea*) with high efficiency (99.32% Fe^{+2}, 98.83% Pb^{+2} and 98.55% Cu^{+2} removal). Other contaminants such as Cd have been removed from water using activated carbon made from the digestion residue of corn straw silage (*Zea mays*) (Tao et al., 2019), and pine wood (*Pinus tabuliformis*) (Liu et al., 2016). Furthermore, Mahmoud et al. (2021) removed Cd^{+2} and Sm from water samples using activated carbon from artichoke (*Cynara scolymus*) leaf litter.

Table 6. Percentage of metal removal due to the effect of activated carbon

	Metals (ppm)			
	Al^{+3}	Cu^{+2}	Fe^{+2}	Zn^{+2}
Initial	0.035	0.04	0.36	0.0024
Removed	0.032	0.03	0.21	0.0014
% removal	91.4	75.0	58.3	58.3

Alslaibi et al. (2014), obtained activated carbon from olive wood (*Olea europaea*) by physical activation with CO_2, with which they removed the ion Cr^{+3} from water samples. Ahmad et al. (2018), obtained activated carbon from banana peels (*Musa paradisiaca*) through chemical activation, with which they decontaminated water samples with heavy metals such as Pb^{+2}, Cu^{+2}, and Cd^{+2}. Wu et al. (2021) elaborated on activated carbon from alkaline lignin residues with which they removed Pb^{+2} from wastewater samples. Similarly, Saeed et al. (2022) used rice husks (*Oriza sativa*) to manufacture biochar, with which they managed to remove 72% Cd^{+2} from water samples at the laboratory level.

According to Iamsaard et al. (2022), granular activated carbon is one of the most effective adsorbents and has significant potential to remove metals from domestic water and wastewater. The adsorption of metals is possible due to the high internal surface of activated carbon, which has the capacity to adsorb or retain substances on its surface by its micropores. Functional groups, such as carbonyl and hydroxyl, could behave as adsorption sites (Gonçalves Júnior et al., 2022). The metals present in the water samples of Yarinacocha Lagoon, were transported from the solution phase to the interior and surface of the activated carbon by means of electrostatic attraction and surface complexation mechanisms, which usually occur in the adsorption of metals (Shahrokhi-Shahraki et al., 2021). The diffusion was supported by the porosity characteristics of the activated carbon, since, according to Qiu et al. (2018), mesopores generate the channeling of adsorbates (heavy metals) towards the micropores, which are sites with high adsorption capacity. The adsorption power of activated carbons to remove heavy metals, lies in an ion exchange mechanism, expanding the understanding of the adsorption mechanism (Dong et al., 2018). According to Dong et al. (2016), it depends largely on the properties of activated carbons that involve pore size distribution, as well as pH (zero point charge: PZC), surface functional groups, the condition in which the experiment is developed, chemical adsorption, specific adsorption, and electrostatic effects. Tan et al. (2015) explained that there are many functional groups on the surface of activated carbon that mainly contain oxygen, e.g., carboxylate (ACOOH), and hydroxyl (AOH), which can interact strongly with heavy metals, such as electrostatic attraction, ion exchange, and surface complexation. Additionally, Khare et al. (2017), hypothesized that the metal adsorption process is due to an initial surface adsorption followed by intraparticle diffusion.

Conclusions

1. The conversion of dry cocoa pod husks into activated carbon is feasible through thermal activation, carbonization and modification time.

2. The optimal values of the evaluated factors to manufacturing activated carbon from dry cocoa pod husks were determined to be 150 °C activation temperature, 450 °C carbonization temperature, and 2.5 hours modification time, with which it was possible to remove 91.4% aluminum, 75% copper, 58.3% iron, and zinc 58.3%.

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