

Portable Filter Prototype for Hg²⁺ Removal with Bamboo fiber, Carbon Nanotubes and Activated Carbon

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Abstract

The uncontrolled use and dumping of mercury waste (Hg²⁺), a highly toxic metal ion, generated by industrial processes, mining, among others, have contaminated both surface and subway water sources, becoming a risk to human health due to the need for direct consumption of water from these sources, in addition to the damage caused to the general population, particularly the Army soldiers who perform activities in these areas. The purpose of this research is to design a prototype of a portable filter adaptable to the canteens of the Colombian National Army, which allows an efficient removal of mercury ions in aqueous solutions, for which adsorption isotherms in liquid phase will be used as a methodological resource of analysis. The result obtained is an adsorbent that combines the functional characteristics of three materials (the structure and flexibility of bamboo fibers, the micro porosity of carbon nanotubes and the porosity of activated carbon), mechanically mixed in a 1:7:2 mass ratio (FB, CA, CNT); with which, at 16.0 °C, at a pressure of 0.747 bar and a contact time of 60 minutes, an adsorption capacity of 35 mmol g⁻¹ was observed against a synthetic Hg²⁺ solution.

Keywords:

Mercury ions, Portable filter, Adsorption, Bamboo fibers, Carbon nanotubes, Activated carbon, Water pollution.

Introduction

Mercury is an element of high atomic mass ($A_r = 200.59 \text{ g mol}^{-1}$), with a density of 13534 kg m^{-3} , whose presence in aqueous effluents basically consists of Hg^{2+} , Hg^+ , Hg_2^{2+} and methylmercury (CH_3Hg^+) ions, all of which are extremely toxic and the latter with high mobility through food chains (Cadavid-Muñoz & Arango-Ruiz, 2021; Gafner-Rojas, 2018). Mercury species reach the earth's crust and bodies due to industrial activities, informal mining operations, and burning of fossil fuels (oil, coal, and natural gas) (Díaz Arriaga, 2015). In addition, mercury is characterized by a relatively high vapor pressure ($2.46 \times 10^{-3} \text{ bar}$ at $25 \text{ }^\circ\text{C}$). The presence of ionic mercury species in aqueous effluents represents a real threat to human health and ecosystems due to the high capacity for bioaccumulation; exposure to mercury can cause neurological, renal, and cardiovascular damage; in addition, it represents a high risk for pregnant women and children (Cadavid-Muñoz & Arango-Ruiz, 2021).

In Colombia, many water bodies are particularly affected by the presence of various pollutants, including toxic metal ions (e.g., ionic species of mercury), which increase health risks for those who consume untreated water and especially for the "floating" population, which, among other actors, includes members of the security and defense forces in large areas

of the territory (Vargas Licona & Marrugo Negrete, 2019). In adverse and very demanding conditions, this population is exposed to intense days where it is not always possible to have safe sources of hydration from the organoleptic (color, smell, taste), biological (pathogenic organisms) and chemical perspectives (substances harmful diseases) (Cadavid-Muñoz & Arango-Ruiz, 2021).

Some characteristics of water, such as color, turbidity, the presence of pathogenic microorganisms, and the possible existence of toxins and other forms of organic matter, can be overcome through simple filtration and heat treatment operations; however, a persistent threat is represented by chemical species such as some transition metal ions that, even in small concentrations, they represent a high risk to life (Chen et al., 2014). In Colombia, artisanal gold mining is one of the main causes of mercury (Hg) contamination. Various studies have shown that at least 24.7% of volunteers present symptoms of poisoning by this metal. In addition, rivers such as the Cotuhe and Putumayo have high levels of mercury, which further aggravates the environmental and health impact associated with this activity (Alcalá-Orozco et al., 2019). Faced with this problem, the development of portable and efficient technologies that can provide safe drinking water in the area of operations is crucial.

From the perspective of water treatment, various techniques have been explored

for the removal of mercury ions; It is presumed, depending on the concentration, that conventional primary treatments suppress the vast majority of pollutants (colloids, organic matter and numerous ionic species). However, additional operations, including chemical precipitation, ion exchange, membrane filtration, and adsorption, are required to ensure that toxic ions of certain metals (mercury, cadmium, lead, and chromium) do not exceed demanding safety limits.

Adsorption is a widely used technique for the removal of contaminants from water, standing out for its effectiveness and high efficiency-cost ratio. In particular, materials such as activated carbon, clays, zeolites and molecular sieves, carbon nanotubes and other carbonaceous materials, as well as certain biomass derivatives (bamboo fibers), have shown great potential in the removal of ionic species present in aqueous effluents, due to surface area effects and ion exchange that give them adsorbent properties and the ability to act in aqueous media.

The study of the adsorbent properties of activated carbon is of great interest from the basic and applied perspectives. Fundamentally, the effect of raw materials, carbonization agents and activation methodologies on texture properties (surface area, porosity, pore size distribution and roughness) and surface chemistry is studied.

To obtain materials with reproducible and recoverable properties against circularity precepts. These efforts are reflected in numerous research and commercial applications, many of them focused on the removal of metal ions (*e.g.* ionic mercury species) present in synthetic and natural aqueous media (Céspedes *et al.*, 2007)

By recognizing some properties of carbon nanotubes such as high mechanical resistance, capillarity and electrical behavior, it is presumed that these characteristics contribute to improving the adsorbent properties of the mixture that contains them. However, the most common morphology of these materials, as well as activated carbon, is finely divided particles; In addition, the incorporation of bamboo fibres in the adsorbent material involves the intervention of other additives (*e.g.* clays) which, without prejudice to the adsorption capacity, allow the configuration, in practical terms, of an adjustable filter-type removal device.

In this sense, there is the possibility that activated carbon, carbon nanotubes, bamboo fibers and configuration additive, contribute synergistically to the adsorption of mercury ionic species (Alguacil *et al.*, 2014). At constant temperature and a given initial ion concentration (C_i), the quantification of the adsorption capacity (mg g^{-1} or mmol g^{-1}) requires the final concentration (C_f), obtained, under equilibrium conditions; these data are represented by isotherms of

adsorption that in principle may correspond to a type I isotherm (Langmuir), in the IUPAC classification or the Freundlich type (Céspedes *et al.*, 2007)

Adsorption capacity (N_0) is a parameter that indicates that, at a given input concentration (C_0) of the adsorbent (ionic mercury species), a certain amount of adsorbent will become saturated after a finite time; i.e., there is a "service time" (t) for a given mass (volume due to density) of the adsorbent. In practice, this translates into knowing the thickness (Z) that an adsorbent column through which the effluent is mobilized at a certain linear velocity (u) *must have*. The so-called Bohart and Adams model is an empirical relationship that allows correlating the flow rate (u), the concentration at the inlet of the adsorbent (C_0), the concentration at the exit of the column (C_e), the thickness of the bed (Z) and the service time of the adsorbent (t). (Rojas *et al.*, 2012).

The purpose of this work is to examine the adsorption capacity of a solid mixture of activated carbon, carbon nanotubes, bamboo fibers and clay, against synthetic solutions of Hg²⁺ ion, with a view to obtaining parameters that allow the design of an adjustable filter for portable water tanks.

Experimental part

The present study is organized in phases that correspond to: Evaluation of the adsorption efficiency of Hg²⁺ in aqueous solutions by batches, design of the organization

of the adsorbent material for the filter based on the analysis of the results of the behavior of the adsorbents and a column study simulating in continuous flow conditions that leads to identify the behavior of the adsorbents in contact with the solution to establish the breakdown curve of the adsorbents as a function of the mercury solution. Each of these phases was designed sequentially, ensuring a logical transition from the preparation of the materials to the design of the final prototype, with the aim of maximizing the efficiency of the adsorption process and its applicability.

The first phase starts with the description of the adsorbents, which include bamboo fibers (FB), carbon nanotubes (CNTs) and activated carbon (CA). With the following characteristics; the activated carbon used in this study is an Orbe-type® granular activated carbon (GAC), as for the bamboo fibers, "bamboo Dynasty" category 1 yarn was used, made from 100% pure bamboo rayon, known for its natural adsorption properties and high mechanical resistance. For the carbon nanotubes, walled carbon nanotubes were selected multiple (MWCNTs), supplied by the company FX NANO, which have a length of between 10 and 30 microns and a purity of more than 95%. It is important to note that no structural or chemical modification is made to the materials, preserving their original shape and properties in order to evaluate their performance in their native state.

The objective of this initial phase is to obtain a clear characterization of each of the selected adsorbents with respect to their individual mercury ion adsorption (Hg²⁺) capacity, which will allow a direct comparison of their efficiency under controlled conditions. Once this preliminary assessment is completed, the materials are combined in different proportions to examine their joint efficacy in adsorption of mercury from aqueous solutions. In order to establish synergies between the three materials and identify the proportions that best perform for the filter design and develop a more efficient adsorption strategy.

To carry out the evaluation, a "stock" solution of HgCl₂ is prepared at a concentration of 100 ppm in type 1 distilled water, which acts as the base solution. From this stock solution, different aqueous solutions of Hg²⁺ are made with initial concentrations ranging from 1 ppm to 8 ppm. These tests are performed under controlled conditions of ambient temperature, maintained at 16°C, and a neutral pH of 7. It is essential to emphasize that pH and temperature directly influence the behavior of the adsorbents, so they remain constant throughout the experimental process.

Calculations for the preparation of solutions are performed using the following equation (1):

$$V_1 = \frac{C_2 \cdot V_2}{C_1} \quad (1)$$

Where, C₂ represents the desired concentration of mercury in the final solution, while C₁ corresponds to the concentration of the stock solution, which is 100 ppm of HgCl₂. V₂ refers to the volume of the sample, in this case 10 ml, which will be the volume selected to be put in contact with the adsorbent material. This calculation allows the mercury concentrations in the different solutions to be used in the adsorption tests to be precisely adjusted.

Adsorption tests are carried out in batches, following a rigorous procedure that involves exposing each adsorbent material to HgCl₂ solutions. For each test, 0.1 grams of the adsorbent material (either bamboo fibres, activated carbon or carbon nanotubes) is used in combination with 10 ml of the prepared solution of Hg²⁺.

To achieve a greater interaction of the solution with the adsorbents, continuous movements are generated, allowing the adsorbent material to come into direct contact with the contaminant, for a minimum period of one hour and a maximum of 8 days.

Once the contact time is completed, a 2 ml sample of the treated solution is collected, which is transferred to microcentrifuge tubes for further analysis. This procedure is essential to quantify the amount of Hg²⁺ remaining in the solution after interaction with the adsorbents. The use of microcentrifuge tubes allows for precise handling of small volumes, reducing the risk of

contamination and Facilitating the Subsequent analysis.

Parameter	Value
Concentration	Variable (1-8 ppm)
pH	7
Temperature	16°C
Contact Time	1 hour - 8 days

Table 1. Initial constant parameters

During the second phase of the study, the

samples obtained after the adsorption process are analyzed in order to determine the residual concentration of Hg²⁺ in the solutions. To do this, the analytical technique of atomic absorption spectrophotometry (AAS) is used. The equipment used in this study is controlled by SpectrAA software. To ensure the accuracy and reliability of the results, the equipment is calibrated to make three replicates for each sample and for the standards used in the calibration. The equipment is organized to establish a measurement time of 5 seconds and a pre-reading delay of equal duration, ensuring that the analysis conditions are constant and reproducible in all tests.

The next stage of the procedure involves the preparation of the

calibration Using Standards of mercury with known concentrations.

These Patterns Allow set a

Precise relationship between the measured absorbance

and the concentration of Hg²⁺ in the treated samples. The calibration curve is obtained by measuring the absorbance of the standards and,

Subsequently, adjusting the data to a calibration line, whose correlation coefficient (R) serves as an indicator of the reliability of the model. From the equation of the line, the concentration of mercury in the experimental samples can be calculated, allowing a quantitative evaluation of the adsorption efficiency of each material. Using the following equation (2):

$$y = mx + b \quad (2)$$

Subsequently, the adsorption efficiency is calculated, the concentration of Hg²⁺ measured in the treated samples is compared with the previously generated calibration curve, which allows the residual concentration of Hg²⁺ in the solutions to be accurately determined after the adsorption process. This value represents the amount of mercury that has not been captured by the adsorbent and therefore remains in the aqueous phase.

The concentration of Hg²⁺ in each sample is obtained and with this data, the adsorption efficiency (%), which indicates the percentage of mercury removed by the adsorbent material in relation to the initial concentration of Hg²⁺ in the solution, is calculated. The calculation is made using the following formula (3):

$$E\% = \left(\frac{C - C_f}{C} \right) \times 100 \quad (3)$$

where C is the initial concentration of Hg²⁺

and it is the final concentration.

C
f

Next, an adsorption isotherm is calculated using the Langmuir model, in order to determine the maximum adsorption capacity of each of the materials, as well as to evaluate their behavior under controlled conditions.

Once the adsorption behavior of the materials separately and the batch removal conditions have been evaluated, the most efficient design is determined from the data obtained. To achieve this goal, it is essential to study the combined behavior of the adsorbents, keeping constant the experimental parameters that influence the efficiency of the adsorption process. These parameters include the initial concentration of Hg²⁺ in the solution, the pH, the temperature of the system, and the contact time between the adsorbents and the mercury solution, as detailed in the table below:

	FB	CA	CNT
MZ 1	5%	60%	35%
MZ 2	5%	70%	25%
MZ 3	5%	80%	15%
MZ 4	10%	70%	20%

Table 2. Parameters Behavior Set

Once this has been done, a new adsorption isotherm is calculated to identify how the materials in synergy retain the mercury molecules.

Study in column

This study is divided into 3 phases, washing the material, preparing the solution and measuring the volumetric flow

The activated charcoal was washed with distilled water to remove any parts that may flow with the solution stream. It was then dried at 105 °C for 5 h.

The conditions for filling the column are detailed in Table 3, where the proportions of the different materials used in the bed are specified. In this case, the column bed is composed of carbon nanotubes (CNT), bamboo fibers (FB) and activated carbon (CA), with a total height of 5.0 cm and an internal diameter of 1.0 cm. The percentages of filling of the column are as follows: 10% of FB, 25% of CNT and 70% of AC.

Material	Height (cm)	Weight (g)	Percentage of fill with respect to the total height (%)
FB	1,25	0,0547	10
CNT	0,25	0,0417	20
AC	3,5	2,1971	70

Table 3. Column filling conditions.

The experimental setup (Figure 1) consisted of a Pyrex® glass column with a height of 25 cm and an internal diameter of 10 mm, two glass vessels to contain the Hg²⁺ solution before entering and after the column, and a peristaltic pump.



Figure 2. Illustration of the assembly to establish the breakout curve.

The experiments were conducted with an initial concentration of 0.8 mg dm⁻³ Hg²⁺, a bed height of 5 cm, and a flow rate of 0.97 cm³ min⁻¹. At regular intervals, aliquots of the fluid were taken at the exit of the bed using a syringe with a hypodermic needle. These samples were centrifuged and the concentration of Hg²⁺ was measured, the representation of the assembly is shown in Figure 2.



Figure 2. Illustration of the assembly to establish the breakout curve.

As for the volumetric flow, the methodology used consisted of measuring the mass of water that was collected in one minute after passing through the peristaltic pump. The results obtained were as follows:

- Body of water: 0.9668 g
- Water density at 23 °C: 0.99762 g cm⁻³
- Volumetric flow: 0.97 cm³ min⁻¹.

This volumetric flow value corresponds to the entry of the column, since, when crossing the bed, the flow loses linear speed and decreases.

Results and discussion

Atomic absorption spectrophotometry was used to determine the residual concentration of Hg²⁺ in the samples after adsorption assays. The calibration curve obtained (Figure 3) showed a linear relationship with a correlation coefficient greater than 0.99, allowing a precise quantification of Hg²⁺ ions.

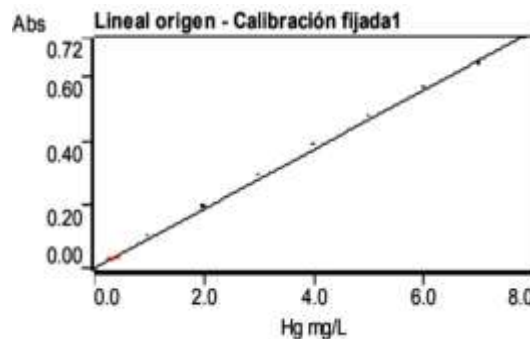


Figure 3. Calibration curve

Batch adsorption assays revealed significant differences in the ability to

adsorption of Hg²⁺ between the materials evaluated: bamboo fibers (FB), carbon nanotubes (CNTs) and activated carbon (CA). By analyzing the graph of adsorption isotherms with 1 hour of contact (Figure 4), the Hg²⁺ removal capacity of each material is established.

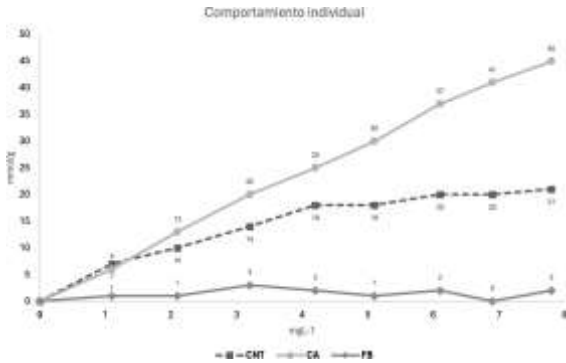


Figure 4. Hg²⁺ adsorption isotherms from aqueous solution with carbon nanotubes, activated carbon and bamboo fibers, within one hour of contact.

Bamboo fibers show a very limited capacity for Hg²⁺ adsorption, with an average efficiency of only 2%. The curve is almost flat, with values that are not

indicating that adsorption capacity is minimal throughout the entire concentration range. This low efficiency can be explained by the surface structure of bamboo fibers, which has a smaller specific surface area and fewer active sites available for mercury ion adsorption compared to the other materials.

Carbon nanotubes show significantly better behavior than bamboo fibers, with an adsorption curve gradually increasing to approximately 10 mmol-1g. Assays indicated an adsorption efficiency of 63% for CNTs in samples of 1 ppm Hg²⁺. Despite this

good performance, the curve stabilizes before reaching the level of removal achieved by activated carbon, indicating that although CNTs are effective, they do not have the highest adsorption capacity.

Activated carbon is the material with the best performance in terms of adsorption of Hg²⁺ in a time of one hour. The curve shows a constant and pronounced rise, reaching values close to 45 mmol g⁻¹, placing it as the most effective adsorbent. Batch adsorption tests show that granular activated carbon has an average efficiency of 60% at all concentrations.

For the extended contact time of 8 days, a change in the behavior of the adsorbents is evidenced (Figure 5), where bamboo fibers continue to show an extremely limited capacity for Hg²⁺ adsorption, with an average adsorption efficiency of only 2%, confirming that despite the prolonged contact time, Bamboo fibers remain the least effective adsorbent.

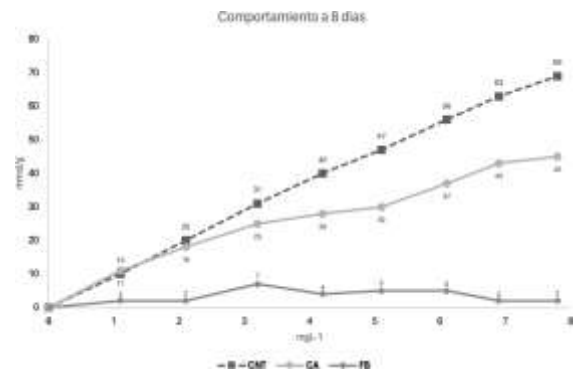


Figure 5. Hg²⁺ adsorption isotherms from aqueous solution with carbon nanotubes, activated carbon and bamboo fibers, in eight days of contact.

On the other hand, carbon nanotubes showed a significant improvement in adsorption as a function of time. In Figure 4, the results indicated that the CNTs initially adsorbed approximately 26% of the mercury present in an 8 ppm solution, however, after a period of 8 days, the adsorption capacity increases reaching 88%. This behavior indicates an adsorption kinetics that favors greater efficiency over time, which makes carbon nanotubes an efficient adsorbent in the removal of Hg²⁺.

In the case of activated carbon (AC), which had already shown a good performance in Figure 4, it was observed that its adsorption capacity reached 25 mmol g⁻¹ in the initial stages of the experiment, at a concentration of 4 ppm, indicating a high initial affinity towards the Hg²⁺ ions present in solution. With the prolongation of the contact time, significant increases in the adsorption capacity of the CA were recorded, reaching approximately 40 mmol g⁻¹ for the same concentration after a period of 8 days. This increase in adsorbent capacity not only reflects the material's stability over time, but also its potential to remove contaminants efficiently in long-term processes.

The fact that activated carbon reaches 40 mmol/g after 8 days is also indicative of its multi-stage adsorption capacity, where the initial adsorption can be followed by a slower diffusion process within the internal structures of the adsorbent, allowing the capture of more contaminant over time.

Activated carbon has also been shown to be less susceptible to rapid saturation, which is essential for use in continuous treatment systems.

In the Hg²⁺ adsorption processes using adsorbent synergy, according to the data presented in Table 2, an adsorption isotherm is observed (Figure 6) that reflects the synergistic behavior when combined in specific proportions.

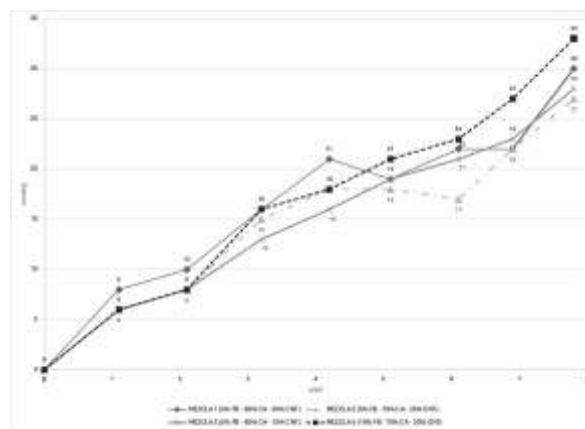


Figure 6. Comparison of adsorption isotherms of mixtures.

It is evident that materials mechanically mixed in a mass ratio of 1:7:2 (FB, CA, CNT) have the highest adsorption capacity along the different relative pressures, reaching a maximum of 33 mmol/g for an Hg²⁺ concentration of 8ppm during one hour of contact.

Figure 6 presents the general behavior of the mixtures, mixture 1 to 1/2 : 6 : 7/2 shows a less efficient behavior compared to MZ4, but superior to that of mixtures 2 and 3 in the initial stages of adsorption.

Mixture 3, with 80% CA and the lowest amount of CNT (15%), exhibits a more linear and progressive behavior in adsorption, indicating that activated carbon is definitely positioned as the predominant component in Hg²⁺ retention, since its capacity remains stable throughout the relative pressure range. Mix 2, with 70% CA and 25% CNT, shows an intermediate yield between blends 1 and 3. Their behavior suggests that a slightly higher proportion of CA improves adsorption capacity compared to MZ1, but does not reach the levels observed in MZ4.

Taking into account that a system with a stable and predictable behavior throughout the adsorption process must be prioritized, the design of the filter with mixture 4 is carried out based on the criteria of: efficiency, cost and availability of materials.

Filter Design

With the adsorption tests it is established that MZ4 is the most favorable composition, therefore the composition that is defined for the filter is: 10% bamboo fibers, 70% activated carbon and 20% carbon nanotubes, the latter are stabilized through a ceramic support, the mixture of clay + NTsC was calcined in a tube furnace using nitrogen atmosphere and maintaining a temperature of 700 °C for two hours. Powder samples were analyzed by XRD on a Bruker-AX D8-Advance diffractometer, with

$\alpha 1$ CuK radiation in the angular range 10–70 degrees 2θ at a scan speed of 2° min^{-1} . Subsequently, by X-ray diffraction (XRD), it is examined in powder for structural analysis. As can be seen, the XRD profile of the sample does not undergo any notable changes, a fact that highlights the conservation of the crystalline structure of the material.

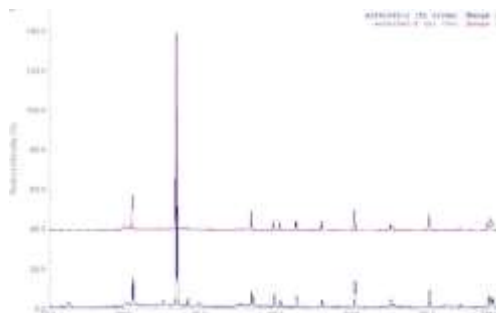


Figure 7. Diffractograms of the raw and calcined powder sample at 700 °C.

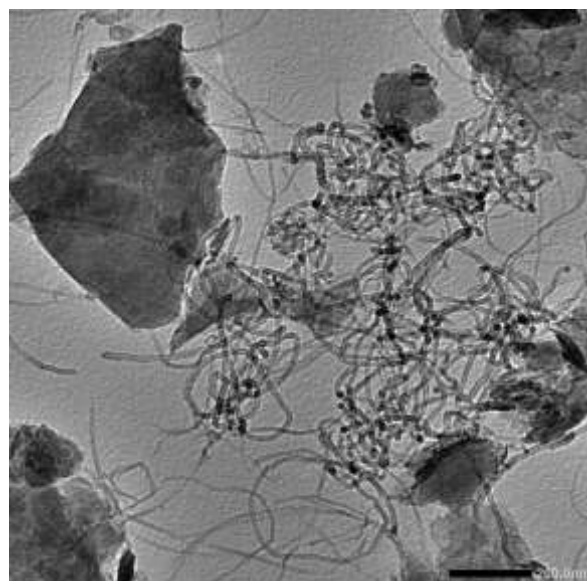


Figure 8. Micrograph images of the ceramic support and carbon nanotubes.

This allows us to establish that it is possible to support and stabilize carbon nanotubes in a ceramic structure, which would be taken to a 3D printing process, in order to generate a solid mesh-type or tubular structure simulating structures

porous structures that would be the structures of the filter to adapt the canteens.

Adsorption studies in a fixed-bed column, unlike batch experiments, do not work under equilibrium conditions as a feed solution continuously enters the column, where a permanent mass transfer process is established between a mobile phase containing the adsorbent and the solid phase of the adsorbent bed. In this research, the criterion of "breakdown time" was adopted as the period that elapses when the concentration of the adsorbable at the exit of the column reached 10% of the initial concentration, in addition, the bed is considered to be completely saturated when both concentrations are equal.

The amount adsorbed per gram of adsorbent (qb) at the breaking point was obtained from the following equation:

$$q = \frac{V}{100} \frac{C_0 - C}{m} \quad (4)$$

where t is the service time (min)
 10
 %

obtained when the concentration of Hg2+ at the exit of the column is 10% of the Initial concentration in mg dm⁻³, (C) is the

Volumetric flow (cm³ min⁻¹), (m) is the mass of adsorbent and the value of 1000 corresponds to a conversion factor.

The results of the removal of Hg2+ ions in the column are shown in Table 4 and the graphical behavior in Figure 7. The breakout time was 70 min and replacing this value, along with the others

Time (min)	C (dm ⁻³ mg)	C/C ₀
10	0,00	0,00
20	0,00	0,00
30	0,00	0,00
40	0,00	0,00
50	0,00	0,00
60	0,00	0,00
70	0,10	0,13
80	0,25	0,31
90	0,40	0,50
120	0,50	0,63
150	0,60	0,75
180	0,64	0,80
210	0,67	0,84
240	0,70	0,88
270	0,72	0,90
300	0,75	0,94
360	0,78	0,98
420	0,80	1,00
480	0,80	1,00

Table 4 Concentration of Hg2+ ions at the exit of the bed as a function of time and its relationship with concentration

initial.

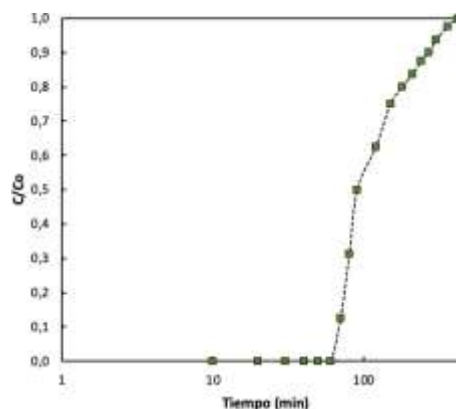


Figure 9. Rupture curve of Hg2+ ion removal in a bed of carbon nanotubes, bamboo fiber, and activated carbon.

Conclusions

It is evident that the adsorption of Hg²⁺ is favorable, since the materials mechanically mixed in a mass ratio 1:7:2 (FB, CA, CNT) have a high adsorption capacity, reaching a maximum of 33 mmol/g for a concentration of Hg²⁺ of 8 ppm.

The adaptation of a portable filter for the National Army's canteens can be achieved through two complementary approaches that guarantee efficiency in the removal of mercury from water sources. On the one hand, the use of a ceramic support transformed into filaments, molded by 3D printing in mesh or tubular structures, offers a solution adaptable to the characteristics of the canteens

The removal capacity per square centimeter of the adsorbent mixture developed in this work reaches its saturation level in a time of 70 minutes. Therefore, by increasing the volume of the filter, the adsorption capacity can be proportionally increased over time, optimizing its performance in practical applications.

The synergy between (FB, CA, CNT) favors the ability to remove mercury from aqueous sources, proving to be more efficient than individually.

Combining materials with ceramic material is an advanced design and development option for the adaptation of army canteens and reduce the risks of contamination due to consumption of mercury-contaminated water.

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