SYNTHESIS OF BIOCARBON FROM WASTE OF PHASEOLUS VULGARIS L. TO OBTAIN A SYSTEM OF WATER FILTRATION ENHANCED WITH SILVER NANOPARTICLES.

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DOI: https://doi.org/10.22267/huellas.251121.25

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Fecha de recepción: 20 de febrero de 2024

Abstract.

The effective management of solid waste is paramount to safeguarding our environment and ensuring a sustainable future. Globally, the agricultural sector significantly contributes to soil and water pollution, despite its substantial contribution to the global Gross Domestic Product (GDP), particularly in developing countries like those in the Equator. Agricultural waste generated post-production is often inadequately managed and underutilized during processing and product collection. This study focuses on obtaining activated carbon from bean pod residues (Phaseolus Vulgaris L.) and its application for water purification. To obtain activated carbon, bean pods were collected, and subjected to carbonization and chemical activation with phosphoric acid (H3PO4) at 700°C, followed by washing. Samples were characterized using Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD). A preliminary gravimetric analysis was conducted to assess the technical feasibility of the process. Furthermore, the properties of activated carbon were enhanced by integrating silver nanoparticles (NPs-Ag), thereby increasing its effectiveness in contaminant removal and bactericidal activity. This enhanced activated carbon was employed in water microbiological analysis filters, resulting in a pH reduction from 8.2 to 7.45 and a 70% decrease in microorganisms. The efficiency of activated carbon extraction reached 63.83%; however, process optimization was identified as necessary. Notably, the most promising results in water purification were observed with the incorporation of silver nanoparticles. These findings support the viability of a sustainable solution to address agricultural waste management challenges and improve access to clean water in vulnerable communities.

Keywords: bean, activated carbon, solid waste, sustainability, environmental, filtration, water.

Resumen.

La gestión efectiva de los desechos sólidos es fundamental para proteger nuestro entorno y asegurar un futuro sostenible. A nivel mundial, el sector agrícola contribuye significativamente a la contaminación del suelo y el agua, a pesar de su importante aporte al Producto Interno Bruto (PIB) global, especialmente en países en desarrollo como Ecuador.

SÍNTESIS DE BIOCARBÓN A PARTIR DE RESIDUOS DE PHASEOLUS VULGARIS L. PARA UN SISTEMA DE FILTRACIÓN DE AGUA POTENCIADO CON NANOPARTÍCULAS DE PLATA.

Los desechos agrícolas generados después de la producción no se gestionan adecuadamente ni se aprovechan eficientemente durante el procesamiento y la recolección de los productos. Este estudio se enfoca en la obtención de carbón activado a partir de residuos de las vainas y ramas pequeñas del frijol (Phaseolus Vulgaris L.) y su aplicación para purificación de agua. Para obtener carbón activado, se recolectaron vainas de frijol, las cuales fueron sometidas a carbonización mediante un proceso de pirolisis y activación química con ácido fosfórico (H3PO4) a 700°C, seguido de un proceso de lavado. Las muestras fueron caracterizadas mediante Espectroscopía de Infrarrojo por Transformada de Fourier (FTIR) y Difracción de Rayos X (XRD). Se realizó un análisis gravimétrico preliminar para evaluar la viabilidad técnica del proceso. Además, se mejoraron las propiedades del carbón activado integrando nanopartículas de plata (NPs-Ag), aumentando así su eficacia en la eliminación de contaminantes y su capacidad bactericida. Este carbón activado mejorado se utilizó en filtros para análisis microbiológico del agua, mostrando una reducción del pH de 8.2 a 7.45 y una disminución del 70% de microorganismos. La eficiencia de obtención del carbón activado alcanzó el 63.83%; sin embargo, se identificó la necesidad de optimizar el proceso. Los resultados del presente trabajo muestran la factibilidad de obtención de carbón activado a partir de los residuos del frejol, mostrando su utilidad para la purificación de agua, especialmente al incorporar nanopartículas de plata. Estos hallazgos muestran un posible camino sostenible para enfrentar los desafíos de gestión de residuos agrícolas y mejorar el acceso al agua pura en comunidades vulnerables.

Palabras Clave: frejol, carbón activado, desechos sólidos, sostenibilidad, ambiente, filtración, agua.

I. INTRODUCTION.

Within the framework of the 2030 agenda and its sustainable development goals, organic solid waste management is crucial for the health of the planet and community well-being. Population growth has increased agricultural production and, with it, the generation of organic waste, aggravating soil and water pollution (De León Duarte, 2022).

Historically, waste disposal was not problematic due to low population density and vast areas of natural purification. However, modernization has increased the quantity and complexity of waste, making it difficult to manage. Overpopulation and consumerism have led to a massive accumulation of waste, creating an urgent need for effective alternatives for its management (Aylwin Ríos, 2017).

Among the main conventional methods of agricultural waste management are uncontrolled burning and disposal of waste in open dumps, which continue to cause serious problems of contamination, disease, and proliferation of vectors (Reina Orosco, 2015). Waste incineration, for example, produces toxic and carcinogenic substances, such as dioxins and furans. In addition, the decomposition of organic matter generates greenhouse gases, such as methane and carbon dioxide. It contributes to the presence of contaminants in the soil surface and groundwater sources (Vega Alonso, 2019).

The agricultural sector, which represents 3.6% of the world's Gross Domestic Product (GDP), is a major generator of organic solid waste (Salgado Ortiz, 2020). In Latin America and the Caribbean, production reaches approximately 430 thousand tons per day (Porras & González, 2016a). In Equator, the situation is similar, with about 7840 tons per day of organic waste (Salgado Ortiz, 2020).

It is essential to revitalize agricultural waste through reuse to preserve resources and minimize environmental impact. Implementing sustainable practices in agricultural sectors, such as bean production, is vital for environmental balance and future well-being.

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Beans, Phaseolus vulgaris L., are an important crop in the Equator, especially in rural areas. Countries such as India, Brazil, Mexico, the United States, and Uganda account for 57.7% of global bean production and consumption (Torres Navarrete et al., 2013). In the Equator, about 35,000 hectares of beans are cultivated, being the Sierra Norte the main producer (Torres Navarrete et al., 2013). However, bean production generates solid waste that, if not properly managed, causes environmental problems (Mota Muñoz & Espinoza Rosales, 2019; Porras & González, 2016).

Activated carbon (AC) is used in various industries due to its adsorbent properties in liquids and gases, such as gas purification, gold extraction, and water treatment, and is produced from various carbonaceous precursors, including agricultural waste (Evwierhoma et al., 2018).

The procurement methods include a carbonization process followed by an activation process. Carbonization is a decomposition process by pyrolysis at high temperatures, in the absence of oxygen, with a temperature range of about 400°C to 600°C for about one hour (Pastor et al.,1999). Such activation can be performed by physical activation (PA) or chemical activation (CCA). In PA, the sample is prepared by washing and drying at high temperatures in an oven, followed by carbonization in an inert atmosphere and activation with water vapor (H_2O), carbon dioxide (CO_2), or air at high temperatures and pressure (Ukanwa et al., 2019; Demiral & Demiral, 2018). On the other hand, in AQ, a similar preparation process is followed, but with the addition of the impregnation of activating agents such as phosphoric acid (H_3PO_4), zinc chloride ($ZnCl_2$), or potassium hydroxide (KOH), prior to carbonization at high temperatures (Kra et al., 2019).

Among the most effective methods, impregnation with agents such as $ZnCl_2$ or KOH are found, followed by carbonization in a nitrogen atmosphere and washing with acid solutions and distilled water to remove impurities (Demiral & Demiral, 2018). Preliminary studies conducted by the Nigerian Journal of Technology showed that the most suitable method to obtain activated carbon from bean pods is the chemical method and that the best activating agent is ortho-phosphoric acid (H_3PO_4), the conditions used were carbonization at 500°C and activation at 700°C (Evwierhoma et al., 2018).

In short, activated carbon is a porous material obtained through carbonization and activation of organic materials, mainly of plant origin. This procedure is carried out with the aim of achieving high porosity and extensive surface area. Its water filtration capacity is notorious thanks to its amorphous structure.

Its capacity for water filtration is notorious thanks to its amorphous structure and its large surface area with pores of various sizes. It is widely used in wastewater treatment and other industrial processes (Nartey & Zhao, 2014). However, adding additional value to it, such as silver nanoparticles, enhances the effectiveness of water filtration systems; by offering to remove pathogenic microorganisms present in water. (Poornima Parvathi et al., 2020; Ghaedi et al., 2012). This study contributes to the development of sustainable technologies for water treatment.

This research proposes the reuse of bean pod remains to produce activated carbon (AC) and evaluate its effectiveness in water purification. In addition, it also seeks to improve its properties by incorporating silver nanoparticles. The objectives of the present work are the following: (1) to obtain AC from bean pods applicable to water filtration, (2) to characterize the AC by FTIR and XRD, (3) to evaluate the technical feasibility of the process by gravimetric analysis, (4) to evaluate the effectiveness of the filtrate in the presence of silver nanoparticles to the AC.

II. METHODOLOGY.

A. Preparation of activated carbon.

I. Materials.

The materials used were: 118.1 g of bean pods, a glass stirring rod, crucibles, 2 beakers of 100 ml, a spatula, filter paper, digital balance, oven, muffle, stove, pH meter MW150 MAX, mill, phosphoric acid, and distilled water. To characterize the activated carbon obtained, FTIR and XRD were used for characterization. FTIR was used to identify the functional groups on the surface of the carbon, while XRD was used to analyze its crystal structure.

II. Procedure.

The chemical method was employed for the production of activated carbon from beans, it is composed of carbonization and chemical activation stages (Ukanwa et al., 2019; Zięzio et al., 2020). For the latter stage, H_3PO_4 was selected as the activating agent based on previous research published by the Nigerian Journal of Technology (Evwierhoma et al, 2018). The procedure is presented in Figure 1.

Figure 1. *Metodología utilizada para la obtención de CA., 2024.*

Adjustments were made to the process, such as modifying the initial drying time of the samples and the integral use of the pod residues, without going through a previous screening process, in order to take maximum advantage of the available raw material. Also, the amount of activating agent was adjusted, opting for a ratio of 10 g in 20 ml of H_3PO_4, in contrast to the 5 g in 20.4 ml of H_3PO_4 used by Evwierhoma et al., 2018.

The bean residues were washed and dried at 105°C for 4 h and subsequently crushed to obtain 90.5 g of material. In the carbonization stage, the shredded material was treated in a muffle at 500°C for 1 h, producing charcoal. In the activation stage, 10 g of this charcoal was mixed with a solution of 20 ml of distilled water and 0.41 ml of H_3PO_4 and dried at 105°C for 24 hours. The resulting sample was activated in a muffle at 700°C for 30 minutes, followed by cooling for 12 hours. The sample was then washed with 0.1 N HCl solution, filtered, and dried again for 24 hours. Finally, the sample was washed with distilled water to a pH between 6 and 7 and dried again.

B. Incorporation of Silver NPs.

I. Materials.

To carry out the in situ synthesis of silver nanoparticles on activated carbon, the following materials and reagents were used: 10 g of commercial AC (Control) and 10 g of bean AC, magnetic stirrer, universal stand and tweezers, beakers, burette, spatula, 2 falcon tubes of 15 ml, 200 ml and 50 ml volumetric flask, glass stirring rod, hot plate, centrifuge, oven, sodium borohydride ($NaBH_4$), distilled water and silver nitrate (AgNO3). For the characterization of the material, a UV-visible spectrophotometer was used to determine the presence of the nanoparticles in the AC.

II. Procedure.

First of all, silver nanoparticles were synthesized, and two solutions were prepared: one of sodium borohydride and the other of silver nitrate. 0.03024 g of NaBH4 was dissolved in a 200-ml balloon and 0.0345 g of AgNO3 was dissolved in a 50-ml balloon. A drip system was set up to gradually add the NaBH4 to the AgNO3 solution in a beaker on a cooled stir plate. In a burette, 10 ml of NaBH4 was loaded, and stirring was activated at 300 rpm for 20 min. The burette stopcock was opened until the solution changed to colloidal yellow. This methodology is based on previous research on the synthesis of Ag-NPs (Sodha et al., 2015; Badi'ah et al., 2019).

Finally, the incorporation of silver nanoparticles was carried out by immersing 10 ml of silver nanoparticle solution per gram of activated carbon. After stirring, it was allowed to stand for 24 hours, and then the aqueous phase was separated from the solid by centrifugation at 1500 rpm for 30 minutes. The liquid phase was decanted, and the residue was completely dried in an oven at 50°C on filter paper. The presence of silver nanoparticles on the activated carbon was examined by UV-vis spectra analysis (El-Aassar et al., 2013).

C. Water filtration system and microbiological analysis.

III. Materials.

Precipitation beakers, spatulas, funnels, sand, cotton, and universal supports with tweezers, commercial and bean activated carbon, as well as variants with NPs-Ag, and water samples, were used.

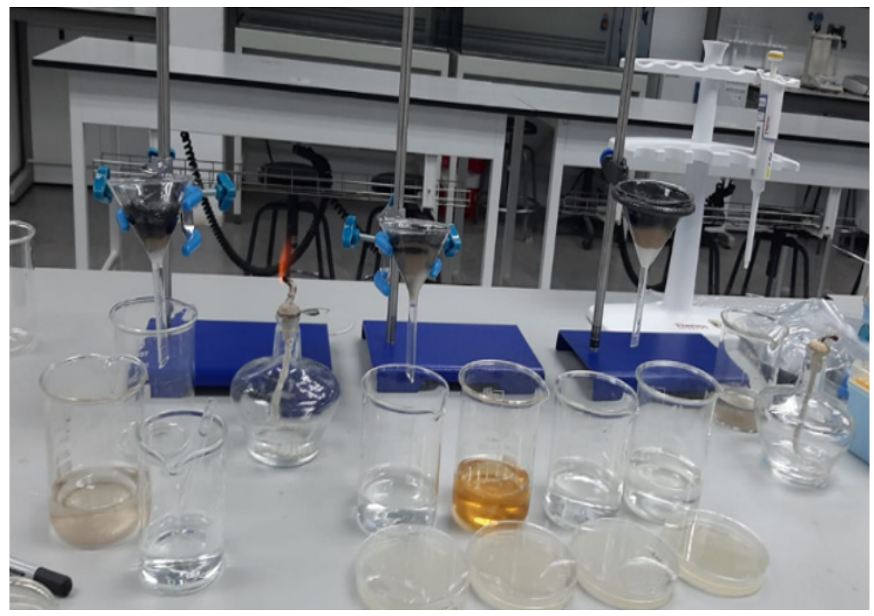
The water collected from an irrigation ditch in Imbabura, Urcuquí, had a pH of 8.2 and a brown color, indicating the presence of organic matter. In addition, it contained sediments and microorganisms, including Escherichia coli.

IV. Procedure.

Conventional filtration systems were assembled to compare the performance of bean-activated carbon, commercial-activated carbon, and its enhanced versions with silver nanoparticles.

The filtration systems were constructed with the same combination of materials: cotton, sand, and activated carbon (Figure 2). Only the type of activated carbon used varied. In the first system bean CA was used, in the second commercial CA (control), in the third commercial CA with NPs-Ag, and in the fourth bean CA with NPs-Ag. Figure 2.

Figure 2. *Ensamblaje de los sistemas de filtrado, 2024.*



Filtered water samples were characterized by pH measurement, color evaluation, and detection of visible solids. For microbiological analysis, they were seeded on agar plates and incubated for 24 hours at 37°C. Subsequently, a comparative visual analysis was performed to detect changes in bacterial growth.

III. RESULTS AND DISCUSSION.

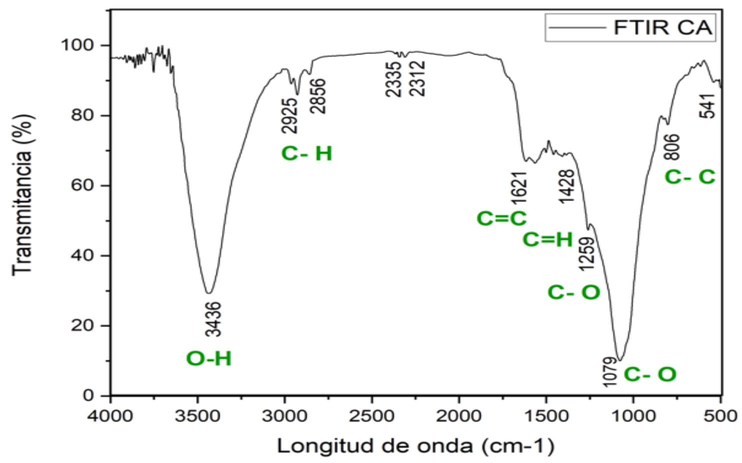
A. Characterization of bean activated carbon.

The FTIR analysis of activated carbon derived from bean pods reveals the presence of several characteristic functional groups (Figure 3) Vibrations of -OH groups are identified at 3436 cm^{-1}, aliphatic (C-H) at 2925 and 2856 cm^{-1}, possible carbonyl (C=O) or alkene (C=C) at 1621 cm^{-1}, and aromatic rings at 1567 and 1498 cm^{-1}, as well as C-O groups at 1259 and 1079 cm^{-1}.

In comparison, previous studies on activated carbon obtained from reed grass leaves show similarities in the functional groups identified. Vibrations of -OH groups at 3600-3200 cm^{-1}, aliphatic CH stretches at 2800-3000 cm^{-1}, possible C=C vibrations at 1630 cm^{-1}, and C-O stretches at 1000-1300 cm^{-1} are observed (Xu et al., 2014). Both studies employed the same activating agent, suggesting consistency in the

These findings support the efficacy of the activation technique used in this research and the feasibility of using bean pods as the main material for activated carbon production. Figure 3.

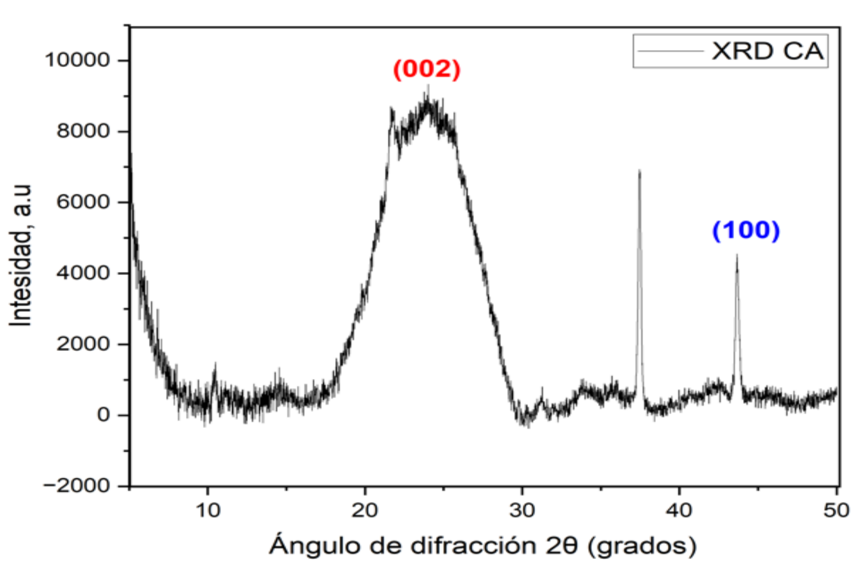
Figure 3.*FTIR de carbón activado de Phaseolus Vulgaris L., 2024.*



The XRD analysis reveals distinct peaks at 2θ = 21.65°, 23.77°, 37.47° and 43.66°. The peaks at 2θ = 21.65° and 23.77° are associated with amorphous carbon, while the peaks at 2θ = 37.47° and 43.66° indicate the presence of crystalline structures. Comparing these results with previous studies showing peaks in the (002), (100) and (101) planes of graphitic carbon confirms the formation of a well-defined crystalline structure (Kalagatur et al., 2017). Moreover, the sharp peak at 2θ = 26° corresponding to the turbostratic crystal structure suggests a better structural organization.

Similarly, according to Bedia et al.(2020), the XRD analysis reveals the presence of distinct peaks around 25° and 43°, consistent with the (002) and (100) crystalline planes of the carbon (Figure 4). These results suggest a well-defined crystalline structure in the bean-activated carbon, indicating higher structural stability and a larger active surface area for adsorption. The XRD results of this study show a mixture of amorphous and crystalline phases.

Figure 4. *XRD de carbón activado de Phaseolus Vulgaris L., 2024.*



B. Gravimetric analysis of sample composition.

The efficiency of obtaining activated carbon from bean bagasse was evaluated by gravimetric analysis based on APHA standard section 2540 G (Bridgewater et al., 2017). The results were:

Table 1.
Resultados de análisis gravimétrico, 2024.

Sólidos totales	40 **[mg/kg]**
Sólidos volátiles	9.0025 [mg/kg]
Sólidos fijos	75.32 [g/kg]
Porcentaje de humedad	18.17%

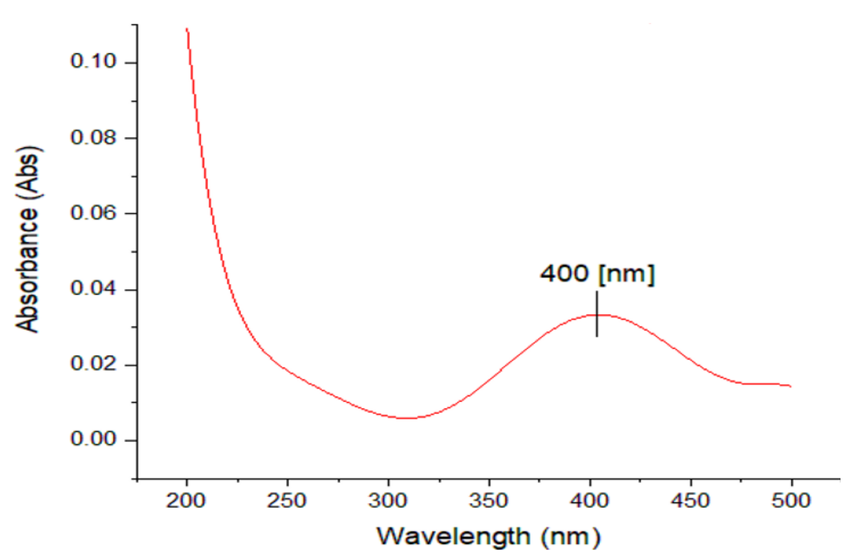
Activated carbon production efficiency reached 63.83%, with a total loss of 42.69 g from the initial sample of 118.1 g after the drying and pyrolysis process.

The high proportion of fixed solids indicates resistance to calcination, which benefits the quality of the activated carbon obtained. On the other hand, the low volatile solids content suggests a significant loss of organic matter during carbonization, which contributes to the formation of a porous structure of the activated carbon.

C. Characterization of activated carbon with silver NPs.

The Ag-NPs exhibit a characteristic absorption band between 400 nm and 500 nm, which causes a color change in solution from yellow to (Sodha et al., 2015; Mulfinger et al., 2007). The impregnation of these nanoparticles on activated carbon was confirmed by UV-vis spectrum analysis (Figure 5).

Figure 5. *Espectro de UV-vis de Carbón activado y NPs de Ag, 2024.*

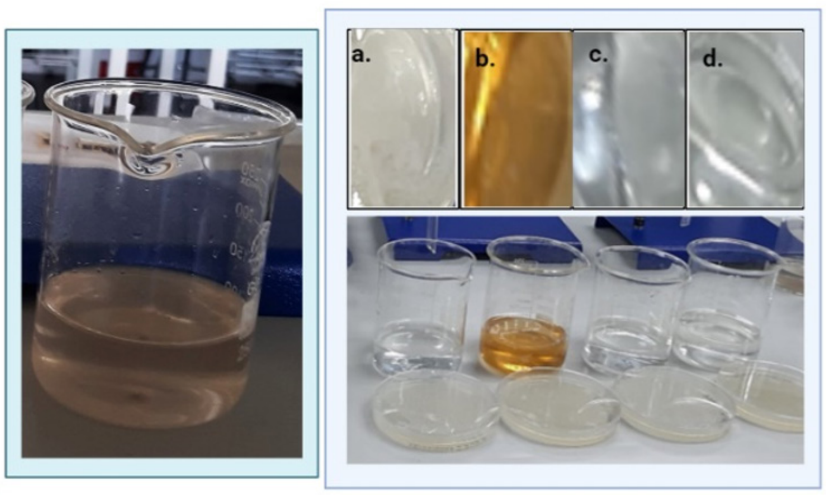


D. Microbiological analysis of filtered water..

1. Characterization.

Figure 6 depicts the color variation observed in the water samples after filtration using different types of charcoal: a) commercial activated carbon, b) charcoal, c) charcoal derived from beans (Phaseolus vulgaris), and d) charcoal with silver nanoparticles.

Figure 6.
Colorimetría del agua filtrada, 2024.



Despite the yellow-colored characteristic of the Ag nanoparticles, the filtered water shows a transparent and crystalline appeareance **(Figure 5.d)**, in contrast to the typical yellow color of Ag-NPs.

The characterization of the filtered water samples is described in detail in Table 2.

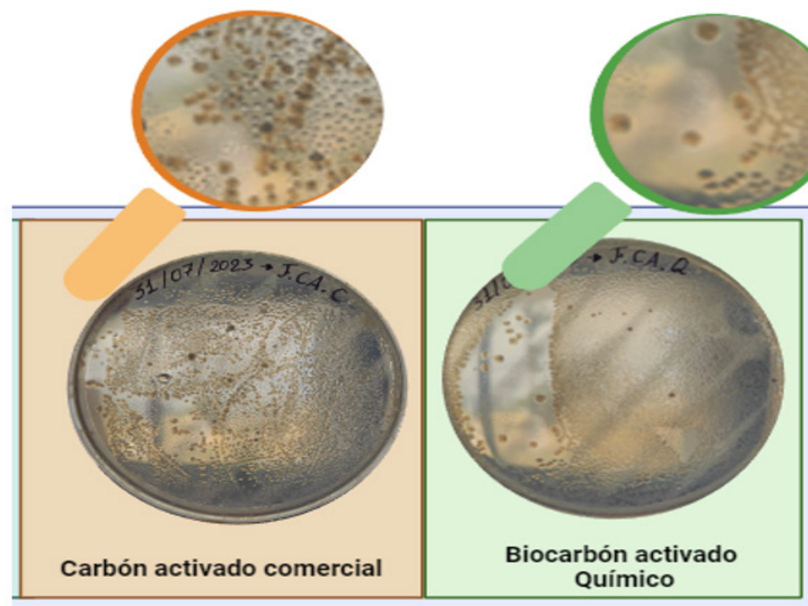
Table 2.
Caracterización de las muestras filtradas de agua, 2024.

Tipo de filtro	pH	Color	Sólidos presentes
Características de muestra de agua.	8.2	Marrón	Matéria orgânica Sedimentos Microrganismos *(Escherichia coli)*
Control (Carbón activado comercial)	7.73	Transparente	No presenta Microorganismos
Carbón activado de Frejol	7.68	Transparente	No presenta Microorganismos
Control CA comercial con NPs - Ag	7.41	Transparente Cristalino	No presenta Microrganismos
CA de Frijol*con NPs - Ag*	7.31	Transparente	No presenta Microorganismos

2. Invitro plate analysis

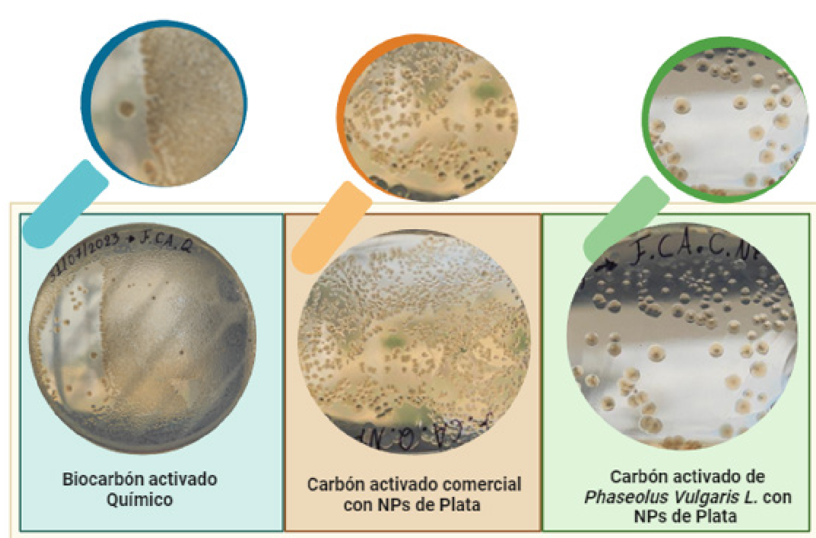
The microbiological analysis of filtered water samples showed that bean-derived activated carbon reduced the bacterial load more effectively than commercial activated carbon (Figure 7).

Figure 7.
Placas in vitro de muestras de agua filtrada, 2024.



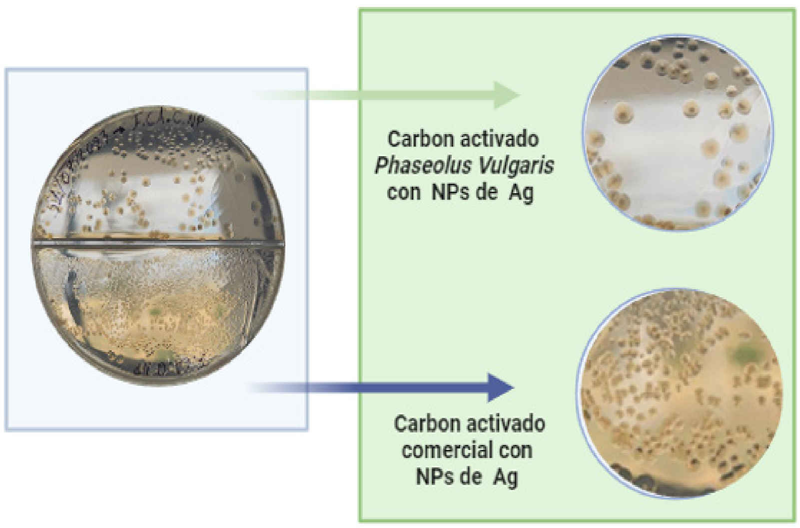
On the other hand, when comparing the reduction of bacterial load between commercial activated carbon (control), bean charcoal and its enhanced version with silver nanoparticles, it was observed that the best results were obtained when silver nanoparticles were implemented (Figure 8).

Figure 8.
Placas in vitro de muestras de agua filtrada, 2024.



Definitely, bean AC with NPs-Ag proved to be more effective in reducing bacterial colonies compared to commercial AC with silver nanoparticles. This suggests that when NPs are incorporated into bean activated carbon, they improve its ability to kill microorganisms, possibly due to the higher porosity and specific surface area of bean activated carbon (Figure 9).

Figure 9.
Detalle del logo tipo de JITEL, 2013.



IV. CONCLUSIONS.

The FTIR analysis of activated carbon derived from bean pods reveals the presence of characteristic functional groups, such as hydroxyl, aliphatic, carbonyl, and aromatic groups, similar to those of conventional activated carbon. These results highlight the feasibility of producing activated carbon from bean pods, underscoring the potential of using agricultural residues as a sustainable and economical alternative.

The XRD analysis reveals a well-defined crystalline structure with ordered domains in the bean-activated carbon, indicating its high quality and potential for filtration and purification applications. Good crystallinity and large crystallite sizes suggest high structural stability and higher active surface area for adsorption.

The gravimetric analysis showed that bean bagasse is highly effective in the production of activated carbon, with a conversion rate of 63.83%. The presence of a significant amount of fixed solids indicates adequate resistance to calcination, while the low amount of volatile solids suggests minimal loss of organic matter during pyrolysis.

Activated carbon derived from Phaseolus Vulgaris, commonly known as "bean", exhibits remarkable capabilities for the removal of impurities in water. However, its effectiveness is further enhanced when combined with silver nanoparticles, resulting in a significant reduction of microorganisms during the filtration process. This finding underscores the potential of bean-activated carbon as a promising tool for improving water quality.

This study has demonstrated the feasibility of obtaining activated carbon from bean pods using a carbonization and activation process. It was found that a carbonization process at 105°C for 4 hours and activation at 700°C for 30 minutes was effective in obtaining a quality product. In addition, it was observed that prolonging the activation time increased the amount of ash in the samples, suggesting not to modify the activation time.

For future research, it is suggested to explore more environmentally friendly activation methods and conduct detailed analysis using techniques such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) for better characterization of the obtained activated carbon. These efforts can provide a more complete understanding of the properties of the activated carbon and help to further improve the production process.

The study demonstrates that the production of activated carbon from bean (Phaseolus vulgaris L.) waste, enhanced with silver nanoparticles, is a viable and effective technical solution for water purification, sustainably addressing agricultural waste management and significantly improving water quality. This technology is aligned with the Sustainable Development Goals of the 2030 Agenda, promoting a circular economy, minimizing environmental impact, and contributing to community well-being.

V. ACKNOWLEDGMENTS.

We are grateful for the support of the laboratory technician in the Yachay Tech Laboratories of the School of Biological Sciences and Engineering, School of Chemistry and Engineering, School of Physics.

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