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Sugarcane bagasse fibre-polyhydroxybutyrate biocomposite – the study of adsorption capacity

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Abstract: This study aimed to develop a biocomposite based on cellulose acetate (CA), extracted from sugarcane bagasse, and polyhydroxybutyrate (PHB), to enhance the hydrophobic properties of the resulting biomaterial and improving their efficiency in adsorption processes. It was hypothesised that the incorporation of PHB into the CA matrix would significantly increase the material's hydrophobicity. The CA/PHB biocomposite was successfully prepared, and water adsorption tests confirmed that the addition of PHB imparted greater hydrophobic character compared to its precursors (bagasse and cellulose). Notably, the biocomposite exhibited the lowest water adsorption (20% in 120 h). Scanning electron microscopy (SEM) analysis confirmed that the PHB adhered correctly to the biocomposite surface, resulting in a material with high porosity and uniform biopolymer distribution. Additionally, thermogravimetric analysis (TGA) demonstrated that the cellulose obtained presents a good thermal stability compared to values reported by other investigations, with decomposition starting at 280°C. The Fourier transform spectroscopy (FTIR) further showed the characteristic functional groups of PHB in the biomaterial and it was also evidenced that the -OH groups contribute significantly to the efficiency of the process. Finally, the adsorption study showed efficiencies of 99%, 91%, and 87% for sugarcane bagasse, cellulose, and CA/PHB, respectively, at 10 ppm and doses of 30 mg. Overall, it was concluded that CA/PHB is the biocomposite with the highest hydrophobic character, maintaining a compelling performance in the adsorption process and highlighting its future use in adsorption columns.

Keywords: adsorption, biocomposites, cellulose acetate, polyhydroxybutyrate, sugarcane bagasse

INTRODUCTION

Over the years, water pollution has increased due to industry's rapid growth, affecting water quality, human health, and ecosystems. The main sources of contamination are mainly harmful substances such as organic matter, dyes, and pharmaceuticals (Rathi, Kumar and Vo, 2021; Pan *et al.*, 2025), among others. Some of these compounds are associated with increased health risks, making water purification a major global issue (Zhou *et al.*, 2021; Li *et al.*, 2025). Additionally, pollutants such as oils, phosphates, and heavy metals affect the growth of flora and fauna, accelerate eutrophication, and affect human health (Monney, Donkor and Buamah, 2020).

Various methods are available for the treatment of contaminated water, including adsorption (Wu *et al.*, 2025), photocatalysis (Zhang *et al.*, 2024), electrolysis (Zhang *et al.*, 2025), coagulation/flocculation (Ribau Teixeira *et al.*, 2024), reverse osmosis (Chen *et al.*, 2025), ion exchange (Shao *et al.*, 2024), and membrane separation (Fang *et al.*, 2025), among others. Among these, adsorption emerges as an economical and simple-to-operate technique for removing contaminants, generally operating under ambient conditions. In addition, adsorption does not require additional expensive materials such as catalysts. For adsorption to occur, an adsorbent agent is needed (Pang *et al.*, 2020; Caicho-Caranqui *et al.*, 2024). A major drawback of synthetic adsorbents lies in their non-biodegradablility and potential release of toxic impurities to the environment during reuse, in addition to their high cost. Unlike synthetic materials, natural fibres derived from agricultural waste have significant advantages due to their high cellulose content, biodegradablility, abundance, reusability, and environmentally friendly profile (Nata *et al.*, 2020; Shen, Liao and Li, 2021). Therefore, in certain cases, these materials show limited adsorption behaviour. To address this issue, different investigations have been carried out to develop biocomposites that combine high adsorption capacity values and good physicochemical properties (Kamran *et al.*, 2022).

Sugarcane bagasse is a lignocellulosic residue obtained after the extraction of sugarcane juice. Over the years, this material has been studied for different applications due to its abundance, affordability, and biodegradability. Several investigations have proposed its use as an adsorbent to evaluate adsorption capacity (Ponce *et al.*, 2021).

Despite their advantages, natural fibres have disadvantages such as low thermal stability, insufficient mechanical properties, and hydrophilic character (Li *et al.*, 2020) among others. Natural fibres are highly hydrophilic which causes the material to degrade over time and affects its physicochemical properties. The hydrophilicity of these materials limits the formation of biocomposites. It also directly affects the adsorption processes, as demonstrated by the research. For instance, Liu *et al.* (2021) investigated the adsorption of benzene using activated carbon in a packed column and demonstrated that increasing relative humidity reduced adsorption efficiency from 65 to 27%. The hydrophilic character of these fibres can generate hydraulic problems in adsorption columns due to the agglomeration of the particles, making the use of anti-caking agents necessary (Benito-González *et al.*, 2020).

In recent years, various studies have focused on the development of adsorbent biomaterials derived from agro-industrial residues such as sugarcane bagasse (Noreen *et al.*, 2020), maize husks (Barzegarzadeh, Hazrati and Amini-Fazl, 2025), potato peels (Abdulhameed *et al.*, 2025), palm leaves (Islam *et al.*, 2025), banana pseudo stems (Nguyen Thi *et al.*, 2024), rice husk fibres (Kong *et al.*, 2025), and cocoa shells (Prasad *et al.*, 2024), among others. These studies have yielded promising results that encourage continued research in this field. For example, Kerrou *et al.* (2021), studied the adsorption capacity of sugarcane bagasse for the dye methylene blue, obtaining an adsorption capacity of 49.3 $mg \cdot g^{-1}$. Kamran *et al.* (2022) carried out a study of the adsorption capacity of different biomaterials made from sugarcane bagasse, obtaining the best results for the biomaterial based on sugarcane bagasse/polypyrrole (205.1 $mg \cdot g^{-1}$). It should be noted that the experimental conditions of pH 2, adsorbent dose of 0.05 g, and initial concentration of 400 ppm for 60 min. Similarly, Luong *et al.* (2024) developed beads based on cellulose extracted from sugarcane bagasse, functionalised with sodium alginate to enhance the adsorption capacity of methylene blue. The results showed an adsorption efficiency of up to 85.33%, corresponding to a capacity of 4.27 $mg \cdot g^{-1}$ at an optimal pH of 8. Further, Ropak *et al.* (2025) formulated a composite based on sugarcane bagasse and Fe_3O_4 to improve the adsorption of rhodamine B dye in solution. The results revealed a maximum removal capacity of 93.6 ±1.16% under optimal conditions of pH 3, with an adsorbent dose of 1 g, and an initial contaminant concentration of 10 $mg \cdot dm^{-3}$, suggesting its potential as an efficient, environmentally friendly, and low-cost material. Abdelmonem *et al.* (2024) modified cellulose with polyacrylonitrile/amidoxime, producing a material effective in the adsorption of Cd^{2+}. The results demonstrated the material's efficiency reaching an adsorption capacity of 123.23 $mg \cdot g^{-1}$ at an optimal pH of 5 and an initial contaminant concentration of 50 $mg \cdot dm^{-3}$.

Despite the large body of research on bioadsorbents derived from lignocellulosic biomass, limited attention has been given to the development of hydrophobic biocomposites combining cellulose acetate (CA) derived from sugarcane bagasse with polyhydroxybutyrate (PHB) for water treatment applications. Therefore, the objective of the present study was to prepare a biocomposite based on cellulose acetate (CA), extracted from sugarcane bagasse fibre, with PHB as the polymeric matrix, and to evaluate its efficiency and adsorption capacity for methylene blue (MB). This research seeks to address the existing gap by developing and evaluating a low-cost biocomposite, based on renewable sources, with potential as a sustainable and effective alternative for the treatment of dye-contaminated water.

MATERIALS AND METHODS

GENERAL INFORMATION

This section describes the materials and experimental procedures used to evaluate the performance of the biocomposite in the adsorption tests. The hypothesis of the study is that the incorporation of PHB into cellulose acetate derived from sugarcane bagasse enhances the hydrophobic character of the material, thereby improving its adsorption efficiency. To obtain the CA biocomposite reinforced with the PHB polymeric matrix (CA/PHA), laboratory equipment used included a heating plate, an analytical lance, an oven, a UV-VIS spectrophotometer, an electric sieve, a pH meter, ultrasonic equipment, and a stirrer. Sodium hydroxide (NaOH) was used as a reagent to extract the cellulose and adjust pH. It used sodium chlorite ($NaClO_2$) as a bleaching agent in cellulose extraction. Acetic acid (CH_3COOH) was used in cellulose bleaching and CA synthesis. Acetic anhydride $(CH_3CO)_2O$ was used for the synthesise of CA. Sulphuric acid (H_2SO_4) was used as a catalyst in the synthesis of CA. As a CA modifier, PHB $(C_4H_6O_2)n$ was used. In addition, solvents such as chloroform, acetone, dichloromethane, ethanol, and phenolphthalein were used.

BIOCOMPOSITE SYNTHESIS

For the synthesis of the biocomposite, sugarcane bagasse fibres were first subjected to size reduction until a particle size of 0.3 mm was achieved. Subsequently, cellulose was extracted using a double alkaline extraction method. In this process, 20 g of pre-washed sugarcane bagasse were immersed in a 4% sodium hydroxide (NaOH) solution for 45 min at 75°C. The resulting mixture was filtered to collect the fibres, which were then thoroughly washed with water until a neutral pH (7) was reached. This extraction step was repeated to complete the double extraction.

The bleaching of the cellulose was carried out using 500 cm^3 of distilled water, 50 g of sodium chlorite ($NaClO_2$), and 50 cm^3 of acetic acid. Initial and final weights were recorded to determine mass loss, attributed to the removal of hemicellulose and lignin.

To obtain cellulose acetate (CA), 12 g of bleached cellulose were mixed with 24 cm^3 of acetic acid and stirred for 60 min. Then, 0.08 cm^3 of sulphuric acid was added as a catalyst, along with an additional 40 cm^3 of acetic acid, and the mixture was stirred for a further 45 min. Afterwards, 28 cm^3 of acetic anhydride and 0.6 cm^3 of sulphuric acid were added to the reaction mixture, which was then stirred for 90 min and left to stand for 24 h.

Finally, a solution of 10 cm^3 distilled water and 20 cm^3 glacial acetic acid was prepared and added to the reaction mixture, which was stirred for 60 min. Then, 500 cm^3 of distilled water was added to stop the reaction, and the resulting CA was filtered, thoroughly washed with water until neutral pH (7) was achieved, and dried at 70°C for 2 h.

The CA obtained was used to prepare a biocomposite using solvent casting methodology. Consequently, acetone was used to dissolve the CA in a mass ratio of 1 to 10 for 2 h, while PHB was dissolved in dichloromethane, using an ultrasonic bath for 30 min at 60°C in the BIOBASE ultrasonic equipment. After this, 20 cm^3 of the cellulose acetate solution and 5 cm^3 of the PHB solution were deposited in Petri boxes and left to stand for 24 h at room temperature until total evaporation of the solvents was achieved (Abu Aldam *et al.*, 2020).

CHARACTERISATION OF THE BIOCOMPOSITE

Considering the hydrophilic nature of the base raw material (sugarcane bagasse), a water absorption test was carried out on the materials before and after the modifications to analyse the change in their hydrophilicity. For this test, the methodology described by Behera, Gautam, and Mohan (2022) was used, following the ASTM D570-98 standard. In this procedure, materials are immersed in water for 24 to 192 h (8 days), and the amount of water absorbed is calculated using Equation (1). The tests were carried out at the Universidad de Cartagena, Piedra de Bolivar campus, specifically in the Energy and Environment Laboratories of the chemical engineering programme.

$$\frac{W_F - W_i}{W_i}100 \tag{1}$$

where: W_F = mass of the sample after absorption, W_i = initial mass of the dry sample.

Characterisation tests were also carried out on the PHB-modified and unmodified biomaterials. The surface charges of the biocomposite were studied using zero charge point tests (pHpzc), and a thermogravimetric analysis (TGA) was performed in a thermogravimetric analyser, TA INSTRUMENT, series: 0600-11099, model: SDTQ600, to determine the thermal stability and composition of the cellulose. Fourier transform spectroscopy (FTIR) determined the functional groups in the biomaterial biomasses and composites. Samples were prepared before and after adsorption under the best conditions and carried out on an IRAffinity-1, FTIRSHI-MADZU, series A213749, on TES-CAN equipment, model MIRA. To observe the morphological properties of the biomass surface before and after modifications, scanning electron microscopy (SEM) was performed. No special sample preparation was required for these tests.

ADSORPTION TEST

To calculate the zero-pH point of the adsorbents, the pH of the distilled water was adjusted to a range from 3 to 11 using a base (NaOH at 0.1 M) and an acid (HCl at 0.1 M). Subsequently, 5 cm^3 of distilled water were added for each pH in 11 different centrifuge tubes, in which 50 mg of each adsorbent were added. The samples were then agitated for 24 h at a room temperature in the shaker. After 24 h (considered sufficient contact time between adsorbent and adsorbate), the final pH was measured to calculate the variation from the initial value. The difference was plotted against the initial pH, and the zero-loading point (PZC) was determined from the intersection of the curve with the abscissa axis (x) (Tejada-Tovar, Villabona-Ortíz and Ortega-Toro, 2023).

Prior to the adsorption experiments, calibration of the methylene blue tracer was performed to establish a standard curve. A series of dilutions were made with concentrations from 5 to 50 ppm, and the absorbance values were measured at 664 nm wavelength using a spectrometer (Oliveira de *et al.*, 2023). Subsequently, the plot of concentration versus absorbance was constructed to obtain the calibration curve.

A pH adjustment test was carried out before the adsorption tests to analyse the influence of pH on the adsorption capacity and efficiency. Three pH values were chosen above the PZC. Experiments were conducted using a fixed dose of 35 mg, with an initial concentration of methylene blue of 40 ppm, and a sample volume of 10 cm^3 (González-Delgado, Villabona-Ortíz and Tejada-Tovar, 2022).

To evaluate the efficiency and adsorption capacity of the prepared biomaterial, seven concentrations of methylene blue solutions were prepared, from 5 to 100 ppm. The doses of adsorbent chosen for the study were 10, 20, and 30 mg. The pH of all the solutions was adjusted for each adsorbent, considering the best result obtained in the pH adjustment tests. The volume of solution was 10 cm^3, and the samples were continuously agitated at 200 rpm and temperature of 35°C for 24 hours. The following day, the final concentration was determined by measuring the respective absorbance of the samples with the help of the spectrophotometer and the equation obtained from the calibration curve.

With the help of Equation (2), the adsorption capacity of the adsorbent could be calculated (Pang *et al.*, 2020) as follows:

$$q = \frac{(C_0 - C_e)V}{m} \tag{2}$$

where: q = amount ($mg \cdot g^{-1}$) of adsorption at equilibrium, C_0 = initial concentration ($mg \cdot dm^{-3}$) in the dye liquid phase, C_e = final concentration ($mg \cdot dm^{-3}$) at equilibrium, V = volume (dm^3) of adsorbate solution, and m = mass (g) of adsorbent used.

Now, to calculate the adsorption efficiency of methylene blue, Equation (3) is used as follows:

$$E = \frac{C_0 - C_e}{C_0}100 \tag{3}$$

where: E = the adsorption efficiency (%).

RESULTS AND DISCUSSION

BIOCOMPOSITE PREPARATION

From the cellulose extraction and purification processes, final masses of 11, 16, and 22 g were obtained, corresponding to an average yield of 54.4%. This reduction in mass is due to the removal of hemicellulose, residual lignin, and other impurities. These results agree with those reported by Melesse, Hone, and Mekonnen (2022), who achieved a cellulose yield of 58.71% after extraction and bleaching. Candido and Gonçalves (2019) evaluated the purity of cellulose obtained by alkali, acid, and bleaching treatments, receiving a purity result of approximately 89%, which confirmed the effective removal of most hemicellulose and lignin from the fibres.

The loss of lignin in the fibres causes the colour transition from brown to white, as shown in Photo 1. A similar behaviour was reported by Sriwong and Sukyai (2022), who bleached cellulose extracted from sugarcane bagasse using sodium chlorite as the bleaching agent.

THERMOGRAVIMETRIC ANALYSIS (TGA)

The thermal stability of the cellulose obtained through the double alkaline extraction method with sodium chlorite bleaching was evaluated by thermographic analysis (TGA) (Fig. 1). The results are presented as a thermogravimetric curve (black line) and its first derivative (DTG, red line). The analysis was conducted over a temperature range of 30 to 950°C, with a heating rate of 10°C·min^{-1}.

The thermogravimetric analysis of the cellulose extracted from sugarcane bagasse revealed several distinct stages of thermal degradation. The first stage, occurring between 29 and 250°C, showed a mass loss of approximately 7%, primarily due to the loss of moisture. It is expected, as sugarcane bagasse is a lignocellulosic residue with hydrophilic properties and a tendency to absorb environmental moisture (Khan *et al*., 2021). The second stage, observed between 280 and 340°C, accounted for a mass loss of about 53%. This phase corresponds to the degradation of silane, a main component of residual hemicellulose, along with the cleavage of glycosidic bonds in cellulose and the initial decomposition of lignin (Sankhla, Liao and Li, 2021). Beyond 350°C, further mass loss was recorded, corresponding to the complete decomposition of cellulose and residual lignin from the previous stage (Vanitjinda, Nimchua and Sukyai, 2019).

The analysis revealed that the initial decomposition temperature of the cellulose was approximately 280°C. Similar behaviour was reported by Phinichka and Kaenthong (2018), who prepared cellulose from sugarcane bagasse using the bleaching method with alkaline extraction, obtaining an initial decomposition temperature of 280°C. Laluce *et al.* (2019) performed seven different treatments on sugarcane bagasse to obtain cellulose. They used sodium hydroxide, sodium hypochlorite, and magnesium sulphate, among other chemical compounds, to carry out

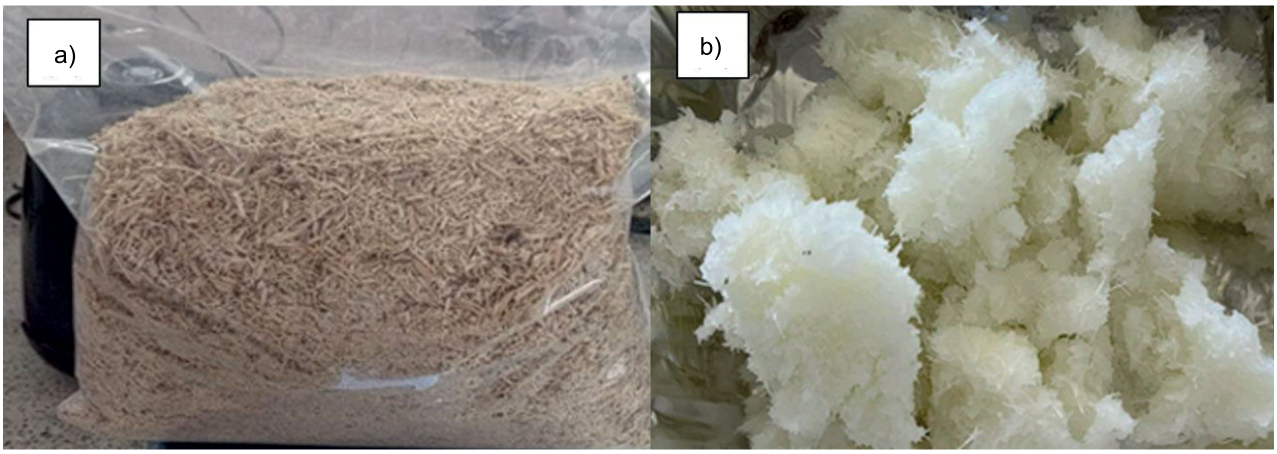


Photo 1. Obtaining cellulose: a) sugarcane bagasse, b) bleached cellulose (phot.: *N. Osorio-Beltrán*)

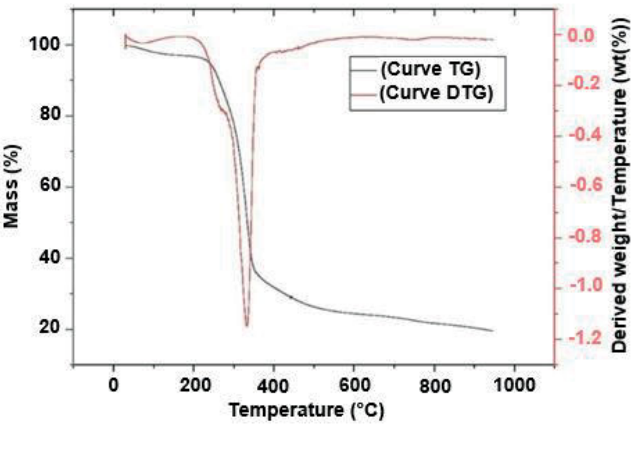


Fig. 1. Thermogravimetric cellulose curve; source: own study

these treatments. Their TGA result showed initial decomposition temperatures ranging from 199 to 263°C.

Finally, it is observed that the cellulose obtained has a good thermal behaviour due to the removal of most non-cellulosic components from the sugarcane bagasse, achieved through alkaline extraction and bleaching with sodium chlorite (Vanitjinda, Nimchua and Sukyai, 2019).

For the preparation of cellulose acetate, 12 g of cellulose were subjected to the procedure described in the methodology. The initial and final masses were recorded to determine the yield of the process. The procedure used serial acetylation with acetic acid, acetic anhydride, and sulphuric acid to achieve a yield of 87%. Based on bibliographic references, the casting method was applied, in which cellulose acetate was dissolved together with PHB to obtain the final biocomposite. This was inspired by the study of Abu Aldam *et al*. (2020), who obtained biocomposites based on PHB-cellulose acetate and polylactic acid (PLA)-cellulose acetate (PHB-PLA), concluding that the presence of cellulose acetate in the biocomposite significantly affects the mechanical properties and surface morphology compared to other materials.

According to the methodology of Abu Aldam *et al*. (2020), the calculated amount of biopolymer (PHB) was directly dissolved in 50 cm^3 of dichloromethane. During preparation, it was observed that as the amount of CA increases, the mass and thickness of the composite biomaterial also increases. For example, the sample with 10 g of CA has a mass of 3 g and a thickness of 860 μm. In comparison, the sample with 8 g of CA has a mass of 2.7 g and a thickness of 620 μm, resulting in the different properties and characteristics of the composite biomaterial.

CHARACTERISATION OF THE BIOCOMPOSITE

To determine changes in the hydrophilic character of the materials, a water absorption study was carried out (Fig. 2). The results show that both sugarcane bagasse and the cellulose obtained present a highly hydrophilic character, with rapid moisture absorption during the first 48 h of the test. The adsorption then stabilised after 72 h, resulting in a final mass increase of approximately 100%.

Including PHB in the CA material reduced water absorption to 21%, attributed to the decrease in hydroxyl groups (-OH) resulting from the presence biopolymer, which exhibits very low hydrophilicity. When combined with cellulose acetate, this effect further enhanced this behaviour (Kang and Yun, 2022). The lower moisture absorption tendency of CA/PHB is positive for overcoming possible caking problems during adsorption processes in packed columns. Reaffirming the above, Jayakumar *et al*. (2020) developed a nanocomposite based on silver and PHB for food packaging applications, and reported that the inclusion of the biopolymer imparted a highly hydrophobic surface to the final material.

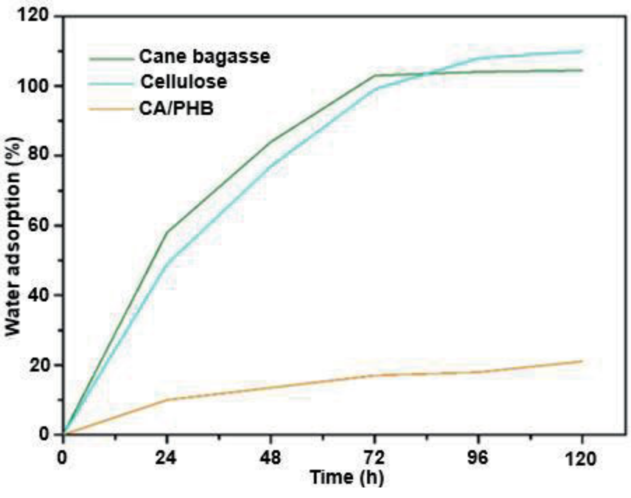


Fig. 2. Water adsorption test of adsorbents; source: own study

SCANNING ELECTRON SPECTROSCOPY – SEM ANALYSIS

The SEM analysis results are shown in Photo 2. The surface structure of sugarcane bagasse is presented in Figure 4a, revealing the existence of "cavities" and a high roughness – features that contribute to a larger surface area (Mehrzad *et al*., 2022). A fibrous structure of the sugarcane bagasse can be observed, with each fibre containing smaller microfibers within it (Leon *et al*., 2020).

In the purified cellulose (Photo 2b), a series of "gaps" or "voids" can be observed along the surface of the fibre. These features result from treatment with sodium hydroxide (NaOH 4%), removing impurities that can be found on the surface of the fibre, such as oils or fats, thereby increasing the amount of hydroxyl groups present in the cellulose (Ponce et al., 2021). The SEM analysis for the CA/PHB at 500× and 1000× magnification are presented in Photo 3. The results show that the biocomposite has a smooth, compact, uniform surface, with numerous pores. The presence of these pores is a positive factor as they enhance adsorption.

FOURIER TRANSFORM SPECTROSCOPY (FTIR) ANALYSIS BEFORE ADSORPTION

For sugar cane bagasse (Fig. 3a) and cellulose obtained (Fig. 3b), a series of peaks from 3,314 to 3,485 cm^{-1} were present, corresponding to -OH functional groups. The peaks are characteristic of lignocellulosic derivatives and are attributed to the presence of cellulose. In Figure 3b, this peak is accentuated to a greater extent due to the purity of the cellulose in comparison to sugar cane bagasse alone. Additionally, the peaks found between 2,888 and 2,987 cm^{-1} are directly attributed to the presence of C-H bonds, characteristic for this type of material. A series of peaks and troughs are observed from 1,369 to 1,716 cm^{-1} in the untreated sugarcane bagasse, indicating the presence of the CH_2 group. However, this behaviour is not observed in the cellulose obtained. This can be explained by the removal of the acetyl groups from the hemicellulose, in addition to the removal of

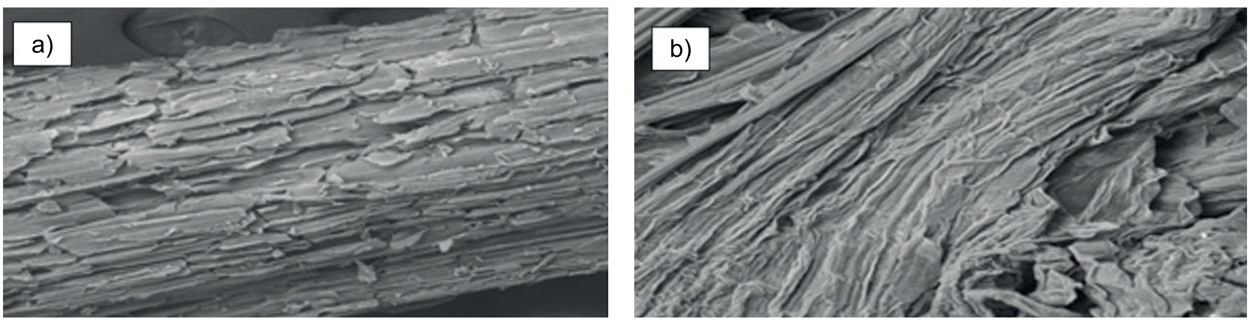


Photo 2. Scanning electron microscopy analysis of: a) sugarcane bagasse, b) cellulose (phot.: *C.N. Tejada-Tovar*)

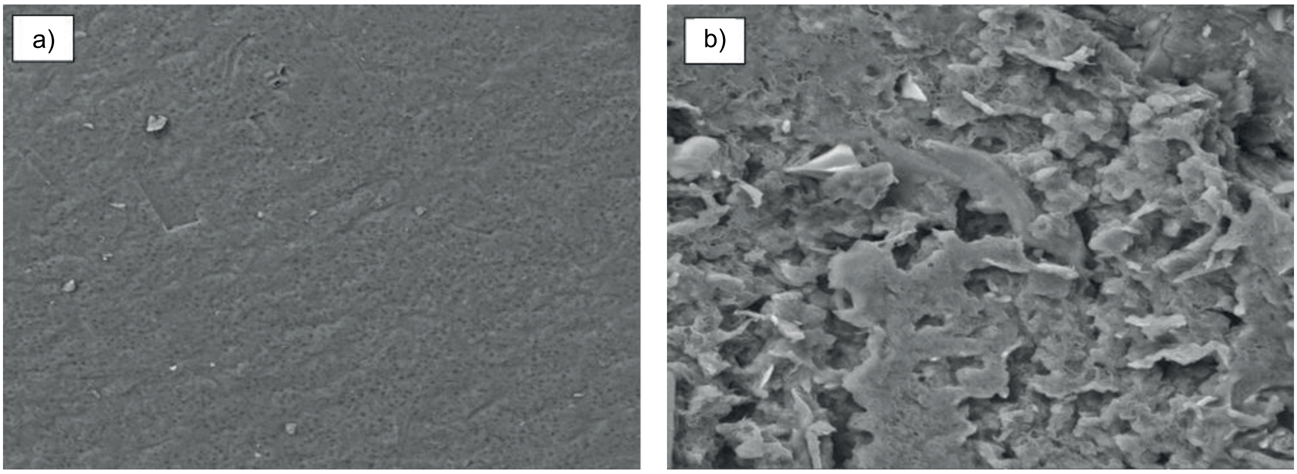


Photo 3. Scanning electron spectroscopy analysis of cellulose acetate / polyhydroxybutyrate: a) view 500×, b) view 1000× (phot.: *C.N. Tejada-Tovar*)

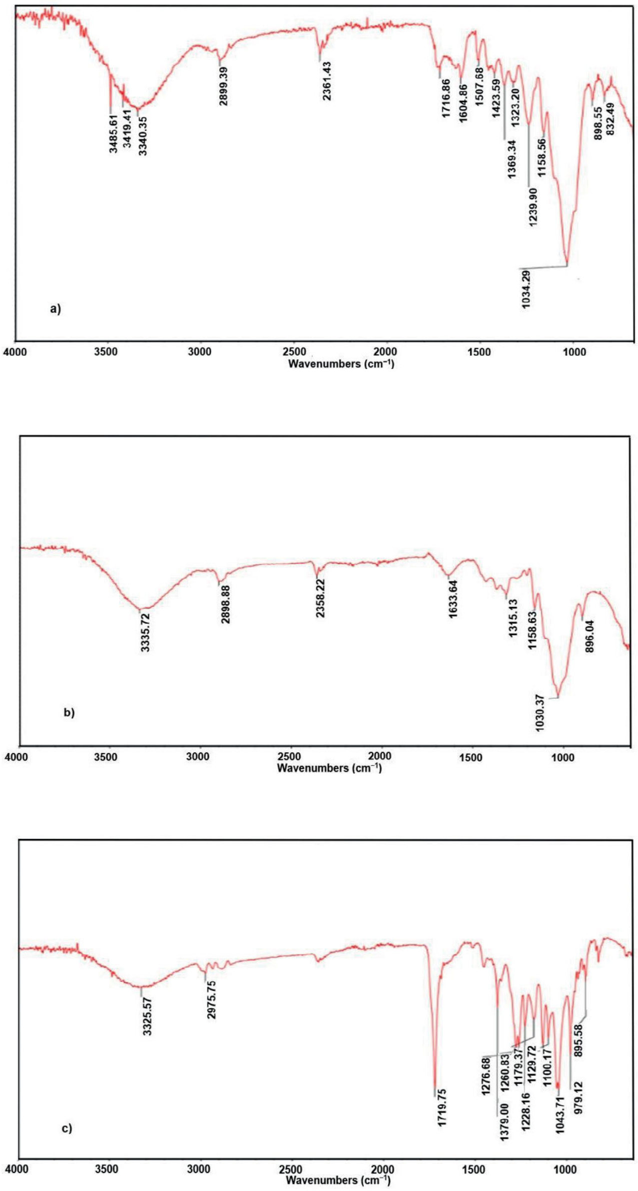


Fig. 3. Fourier transform spectroscopy analysis before the adsorption process: a) sugarcane bagasse, b) cellulose obtained, c) cellulose acetate / polyhydroxybutyrate; source: own study

a large part of the residual lignin. For the wavelengths of 1,034 and 894 cm^{-1}, new peaks can be observed in Figures 3a and 3b due to cellulose's C-O-C pyranose ring and beta-type glycosidic bond.

Finally, when preparing the CA/PHB biocomposite (Fig. 3c), a behaviour similar to that of its precursor is evidenced; however, the peak at 1,720 cm^{-1}, associated with the carbonyl ester group, C=O, showed markedly higher intensity in this case. This indicated that no chemical reaction occurred between PHB and CA, but rather that intermolecular interactions were established between these two materials. Considering the above, it can be seen that the CA/PHB was prepared correctly, demonstrating the presence of both materials in the biomaterial (Gouda *et al.*, 2022; Saiful *et al.*, 2022).

ADSORPTION TESTS

Influence of pH on adsorption tests

As shown in Figure 4, the pHpzc corresponding to sugarcane bagasse, cellulose, and CA/PHB is 4.55, 5.63, and 5.50, respectively.

For composite biomaterials based on sugarcane bagasse with PHB (Fig. 4), no prior studies have been reported. However, other

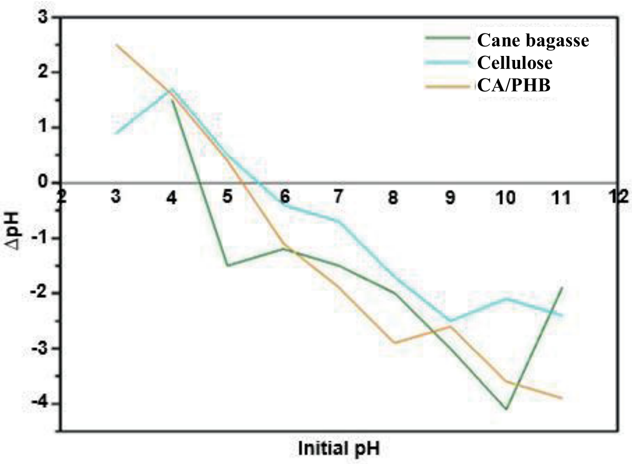


Fig. 4. Zero charge point analysis of base materials; CA = cellulose acetate, PHB = polyhydroxybutyrate; source: own study

studies on composite biomaterials, such as the one by Gallardo-Cervantes *et al.* (2021), evaluated the behaviour of agave when modified with PHB, obtaining results with differences of 0.3 and determining that the main modifications are morphological and mechanical. Their results show no significant variations concerning the unmodified biomaterials. Therefore, it can be inferred that unmodified biomaterials and biomaterials composed with PHB have similar or identical pHpzc values.

In this study, the pH values of 6, 7, 8, 9, and 10 were selected for evaluation, all of which are higher than pHpzc. Since methylene blue is a cationic dye, operating at pH values higher than pHpzc ensures that the surface of biomaterial is negatively charged, thereby promoting electrostatic attraction with positively charged ions.

In addition, a calibration curve (Fig. 5) was evaluated as a graph that relates two variables from a linear regression model used to validate a measuring instrument. The calibration was performed using the methylene blue with concentrations of 5, 10, 15, 20, 25, 30, 35 and 40 ppm to obtain the absorbance values in a spectrophotometer at a wavelength of 664 ppm.

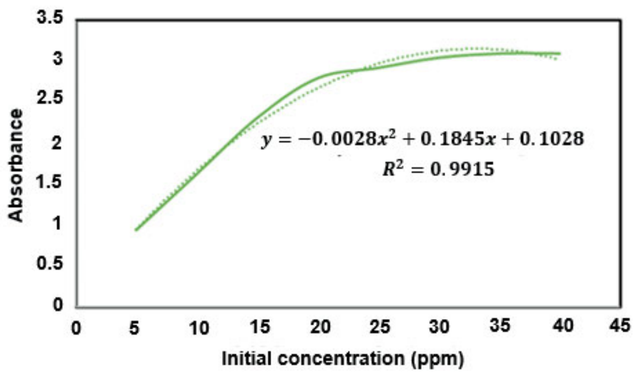


Fig. 5. Calibration curve for methylene blue; source: own study

Therefore, the initial concentration was plotted vs. absorbance and then different models were evaluated. The model that best fit the theory was the quadratic model, since it presented the value closest to 1, with an R^2 value of 0.9915. From the quadratic model, x was cleared to obtain an equation of concentration (Co) as a function of absorbance (A).

$$Co = -0.1845 \pm \frac{\sqrt{0.03515 - 0.0112(A)}}{-0.0056} \tag{4}$$

Sugarcane bagasse has the highest efficiency (Fig. 6a) compared to cellulose (Fig. 6b) and CA/PHB (Fig. 6c). These results are attributed to the main functional groups found on the surface of the biomaterial, which provide abundant active sites for adsorption. In contrast, cellulose and CA/PHB undergo treatments that eliminate lignin and hemicellulose – components that also contribute to important active sites (Mishra and Basu, 2020).

For sugarcane bagasse (Fig. 6a), the highest adsorption efficiency was observed at pH = 9, reaching 97% removal and an adsorption capacity of 11.12 mg·g^{-1}. Similar findings were reported by Al-Mokhalelati, Al-Bakri and Al Shibeh (2021), who evaluated methylene blue adsorption on sugarcane bagasse within pH = 5 to pH = 9, and demonstrated that the adsorption capacity increased with pH, achieving removals of up to 97%.

However, cellulose (Fig. 6b) showed high removal percentages of up to 93%, with an adsorption capacity of up to 10.62 mg·g^{-1}. Although the highest efficiency for cellulose was

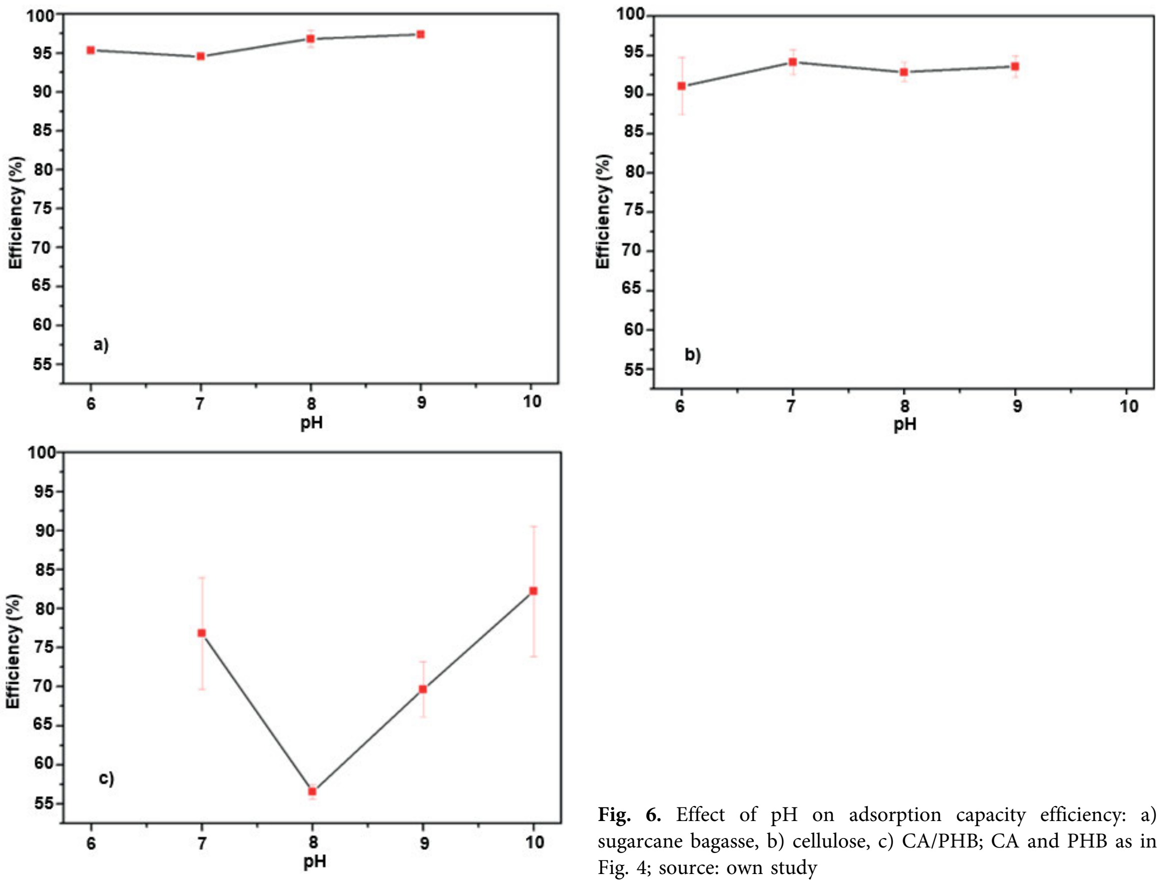


Fig. 6. Effect of pH on adsorption capacity efficiency: a) sugarcane bagasse, b) cellulose, c) CA/PHB; CA and PHB as in Fig. 4; source: own study

obtained at pH = 7, it can be inferred from Figure 6b that from pH = 6 to pH = 9 the efficiency does not vary more than 3%. This trend is consistent with the findings of El Naeem *et al.* (2022), who studied cellulose derived from sugar cane bagasse over a pH range of 2 to 11 and reported the highest adsorption efficiencies between pH = 5 and pH = 9.

For the CA/PHB (Fig. 6c), the highest removal efficiency of approximately 85% was obtained at pH 10, while the lowest of 56% at pH = 8. Compared with the other biomaterials, CA/PHB showed a slight decrease in performance. This is attributed to the high purity of the acetate which, being largely free of lignin and hemicellulose, provides fewer active sites for adsorption. In contrast, unmodified sugarcane bagasse fibres retain these components, offering additional sites for the capture of adsorbate molecules.

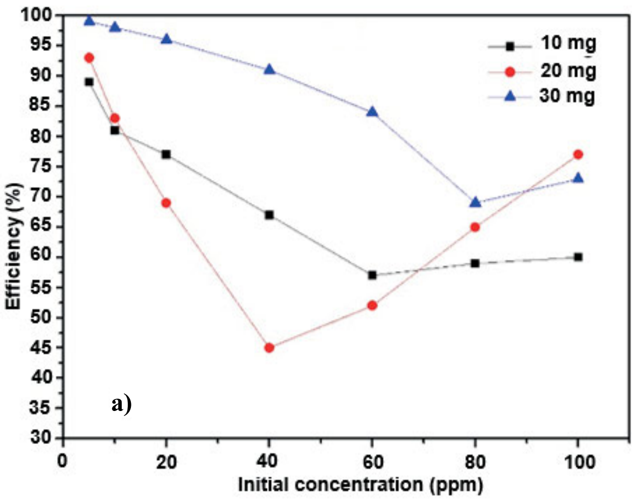
Effect of adsorbent dosage on adsorption efficiency

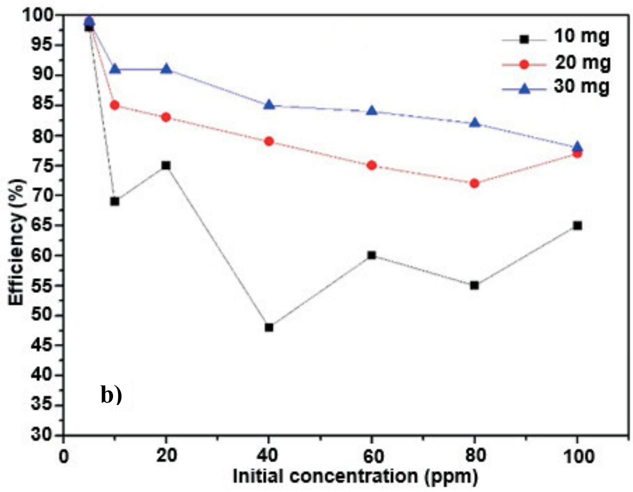
In this study, adsorption capacity was evaluated using different adsorbent doses of the biomaterials (10, 20, and 30 mg) and initial methylene blue concentrations of 5, 10, 20, 40, 60, 80, and 100 ppm. As shown in Figure 7, bagasse has higher efficiency at greater adsorbent doses and lower initial concentration of methylene blue (Fig. 7a). For instance, with a 10 mg dose, efficiency decreased to 57% at 60 ppm, whereas with a 30 mg dose, efficiency increased to 68% even at 80 ppm. These results indicate that adsorbent dose was the most important factor influencing the performance, as higher adsorbent doses provided a greater number of active sites available for adsorption at the same contaminant concentration.

The three doses of cellulose are compared, as shown in Figure 7b. At a dose of 30 mg, the material achieved a minimum efficiency of 78% at 100 ppm. In contrast, with only 10 mg of adsorbent, efficiency dropped to 48%.

Finally, for the CA/PHB biocomposite (Fig. 7c), the adsorbent dose plays a very important role in the adsorption process. At doses of 10 and 20 mg, the material showed insufficient adsorption at concentrations higher than 40 ppm. In contrast, with a dose of 30 mg, the biocomposite reached an adsorption efficiency of approximately 60% at a concentration of 60 ppm. This slight decrease in performance could be explained by the inclusion of PHB into the system. However, the material still showed good adsorptive behaviour, reaching efficiencies close to 90% at 30 mg dose and 5 ppm concentration.

The main difference observed when varying the adsorbent dose appears when we compare the same initial concentrations regardless of whether it is sugarcane bagasse, cellulose, cellulose acetate, and/or any biomaterial composed with PHB. At concentrations lower than 20 ppm, increasing the adsorbent dose to 20 mg or 30 mg consistently improved efficiency of the process compared with 10 mg. This behaviour can be explained by increased amount of material available per unit of solution, which increases the number of active sites for adsorbate





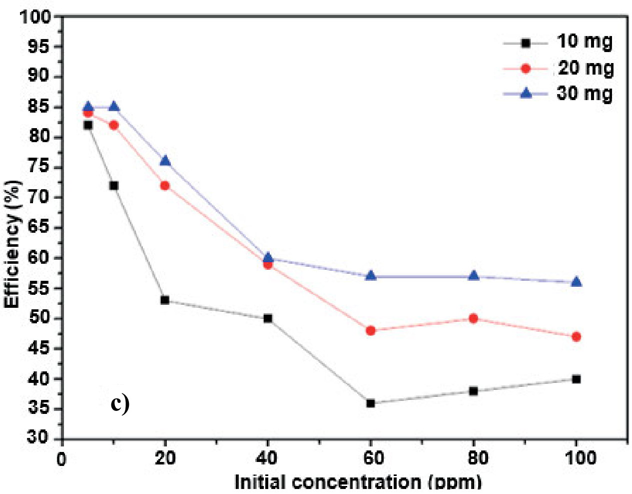


Fig. 7. Effect of adsorbent dosage on adsorption tests for 10, 20, and 30 mg of: a) sugarcane bagasse, b) cellulose, c) CA/PHB; CA and PHB as in Fig. 4; source: own study

molecules (Villabona-Ortíz, Ortega-Toro and Pedroza-Hernández, 2024). Among the biocomposites, CA/PHB exhibited the lowest efficiencies (about 60% at 40 ppm and 30 mg dose). However, at lower concentrations (5–20 ppm), the biocomposite showed efficiencies of approximately 80%. This decrease in efficiency and adsorption capacity can be attributed to the substitution of -OH groups, which favour adsorption, by acetyl groups as well as to modification with PHB. nonetheless, this effect is partly compensated by the removal of more than 60% in the hydrophilic character compared with the unmodified fibre.

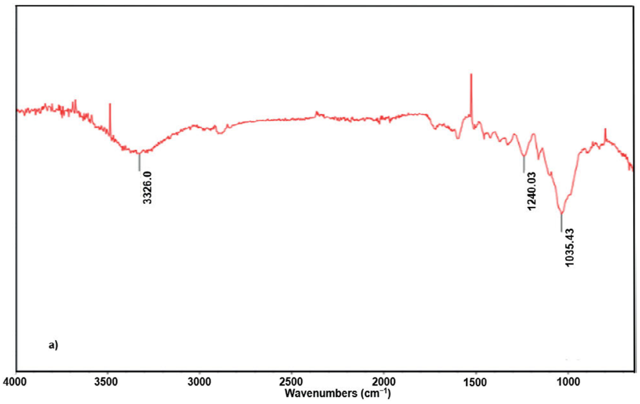
Fourier transform spectroscopy analysis after adsorption

The FTIR spectra of all materials after adsorption are presented in Figure 8. Changes were observed in the intensity and frequency of the adsorption peaks. For sugarcane bagasse, the peak in the wavelength shifted from 3,340 to 3,326 cm^{-1}, while for cellulose, it shifted from 3,335 to 3,319 cm^{-1} (Figs. 8a and 8b, respectively).

A slight decrease in the wavelength of this peak was observed, associated with the presence of the hydroxyl functional group –OH, which is characteristic of lignocellulosic residues. It suggests that hydroxyl groups played a significant role in the adsorption of methylene blue. The interaction between the dye molecules and the adsorbent was possibly mediated by hydrogen bonds (Wang *et al.*, 2020; Salah Omer *et al.*, 2022; Gong *et al.*, 2022).

In the CA/PHB biocomposite, the peak at 3,485 cm^{-1} observed before adsorption decreased in intensity and became difficult to identify after adsorption (Fig. 8c). This indicates that the remaining -OH functional groups after cellulose acetylation continued to participate in the adsorption process. The results obtained from the adsorption experiments also showed that CA/PHB exhibited lower efficiencies compared to fibrous biomaterials (bagasse/cellulose) (Dzoujo *et al.*, 2022; Rana *et al.*, 2022).

To provide a comprehensive assessment of the efficacy and versatility of the CA/PHB biomaterial, its performance was compared with that of other materials commonly used for treatment of contaminated water, as presented in Table 1. The data clearly show that, in several cases, the CA/PHB biomaterial outperformed its counterparts, often achieving higher removal capabilities even at lowered adsorbent doses.



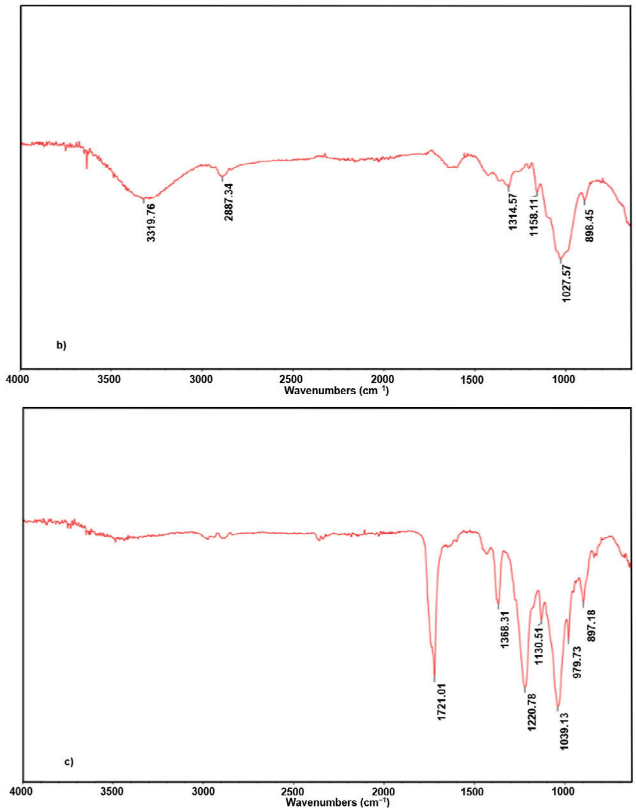


Fig. 8. Fourier transform spectroscopy analysis after the adsorption process: a) sugar cane bagasse, b) cellulose obtained, c) CA/PHB; source: own study

Table 1. Comparison with other studies

Precursor	Modifying agent/ Reinforcing matrix	Contaminate removed	Removal capacity (mg·g^{-1})	pH	Adsorbent dose (g)	Pollutant concentration (mg·dm^{-3})	Reference
Sugarcane bagasse	$CaCO_3$ and NH_4HCO_3	crystal violet	293.02 in 100 min	9	0.1	200–1000	Wang *et al.* (2020)
Raw sawdust	activation with NaOH	indigo carmine	9.39 in 3 h	2.5	5	10–50	Bhowmik, Chakraborty and Das (2021)
Millettia thonningii seed pod	activation with H_3PO_4	methylene blue	2.55 in 3 h	7	0.002–0.2	25–200	Jasper, Ajibola and Onwuka (2020)
Aspidosperma poly neuron sawdust	H_3PO_4 urea 6M	methylene blue	12.45 in 24 h	7	3.5	60	Ortega-Toro *et al.* (2023)
Sugarcane bagasse	–	methylene blue	49.26 in 2 h	10	0.05–0.5	25	Kerrou *et al.* (2021)
Sugarcane bagasse	polypyrrole- $FeCl_3.6H_2O$	acid red 1	205 in 2 h	2	0.05	400	Kamran *et al.* (2022)
Sugarcane bagasse	sodium alginate	methylene blue	4.27 in 24 h	8	0.2	10	Luong *et al.* (2024)

cont. Tab. 1

Precursor	Modifying agent/ Reinforcing matrix	Contaminate removed	Removal capacity ($mg \cdot g^{-1}$)	pH	Adsorbent dose (g)	Pollutant concentration ($mg \cdot dm^{-3}$)	Reference
Sugarcane bagasse	Fe_3O_3	rhodamine B	106.38 in 1 h	3	1	10	Ropak *et al.* (2025)
Cellulose	polyacrylonitrile/ami-doxime	Cd(II)	123.23 in 2.5 h	5	1	50	Abdelmonem *et al.* (2024)
Sugarcane bagasse fibre	PHB	methylene blue	8.2 in 24 h	10	0.03	40	present study

Explanations: T = temperature, t = time.
Source: own study.

CONCLUSIONS

The biocomposite was prepared using solvent coating and casting, which ensured correct adhesion of the biopolymer to the fibres and acetate. This was demonstrated through mass and thickness analyses, which confirmed PHB adhesion across all adsorbents. The results further indicated that the greater the amount of bagasse to be coated within the same volume, the greater the uniformity of the % PHB added to the system.

The sugarcane bagasse exhibited a pHpzc of 4.5, while cellulose and the CA/PHB showed values of 5.63 and 5.5, respectively. These results indicate that all three materials possess a negative surface charge at pH values above 6, enabling them to adsorb MB.

The characterisation analyses confirmed that the preparation of CA/PHB significantly decreased the hydrophilic character of sugarcane bagasse, with water absorption decreasing to as low as 20%. The SEM analysis showed that PHB was uniformly distributed on the surface of the material, generating a larger number of pores. Additionally, the FTIR analysis after the adsorption process showed a decrease in the intensity of characteristic peaks associated with MB functional groups. These changes indicate the adsorption of the dye onto the active sites of the adsorbent material.

The adsorption study showed that, while sugarcane bagasse achieved the highest removal efficiency, the CA/PHB biocomposite also reached yields of over 80% at pH 10. Importantly, the biocomposite exhibited excellent hydrophobic character, which promotes its application in adsorption columns, as it helps to prevent the agglomeration issues typically associated with unmodified lignocellulosic residues.

This finding lays the groundwork for future research focused on the development of sustainable biocomposites derived from sugarcane bagasse and modified with natural polymers such as PHB. It also suggests the need to explore techniques for assessing the material's behaviour against various types of contaminants, as well as evaluate its performance in real water samples, and assess its feasibility in pilot-scale applications.

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CONFLICT OF INTERESTS

All authors declare that they have no conflicts of interest.

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