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RESEARCH ARTICLE

BREWERY WASTE FOR INDIGO BLUE ADSORPTION

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Abstract

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Brewery waste (BW) is among the natural adsorbents that have been evaluated for different chemical pollutants removal present in the aqueous medium. In this work, we investigated the BW capacity to remove the commercial indigo blue dye (IB) from laboratory aqueous solutions and river water samples. Results utilizing laboratory distilled water showed a satisfactory maximum capacity (166.94 mg/g) of BW for IB. The ΔG value (-16.53 Kj/mol) and other parameters reveal a favorable adsorptive process. Furthermore, tests in glass columns using river water samples enriched with IB (1,000 mg/L) revealed high BW efficiency for IB removal (96.0 %). These results are important to demonstrate that BW can be more a new alternative for IB removal from water. In addition, this study provides an alternative for the reuse of BW.

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Introduction:-

Different natural adsorbents (NA) have been studied for chemical pollutants removal present aqueous medium [1]. Among these pollutants, textile dyes are extensively studied [2]. Vermicompost [3], sugar cane bagasse [4], wood sawdust [5], coconut residual fiber [6], and polysaccharides of Carica papaya seeds [7] were evaluated for congo red removal. For IB dye removal, the following have already been studied: vermicompost [3], licorice root powder [8], chitosan [9], and Zizyphus joazeiro peel [10]. The chemical and physical NA characteristics are important factors for textile dye retention [3]. In many kinds of NA, the morphology and presence of some chemical groups are associated with the presence of macromolecules such as cellulose, hemicellulose, lignin, protein, and others [11]. Many NA were studied for chemical pollutant removal are biomass waste from different agroindustrial activity. Among these waste biomass are: sugar cane bagasse [4], orange peel [12], banana peel [13], green coconut mesocarp [13], and brewery waste [14]. Some authors studied the brewery solid waste for treatment of water contaminated with methylene blue dye [15], acid green dye [16], remazol dye [14], and others [17]. In this work, the efficiency of BW as a removal agent for indigo blue (IB) in aqueous medium was investigated. The physical and chemical BW characteristics were carried out through scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR) respectively. Furthermore, the influence of different parameters such as BW granulometry, BW mass, rotational speed and stirring time between BW and IB, and IB concentration (mg/L) in the adsorptive process, was investigated. After these parameters, optimization was possible to obtain the maximum adsorptive capacity (MAC) (mg/g) between BW and IB. Finally, a river's sample water was enriched with IB (1,000 mg/L) and filtered through three glass column containing: BW, sand, crushed stone, and gravel.

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Material and Methods:-
Material:-
The brewery solid waste was supplied by the beer production laboratory of the Federal Institute of Espírito Santo (Vila Velha-ES, Brazil). Indigo Blue (IB) was purchased from Tupy Industry (Sao Paulo-SP, Brazil) in the presence of sodium chloride, detergents, surfactants, and dispersants. The IB aqueous solutions were prepared in distilled water obtained from a water distiller Quimis (Q241-22 model, Diadema-SP, Brazil). For laboratory tests, other types of equipment were needed such as a blender (Mondial power 2i 500 W model, Brazil), a conductivity meter for water (Instrutemp, Sao Paulo-SP, Brazil), a pH-MV-TEMP-meter (Lutron, Taiwan), a magnetic stirrer (Warmnest, 78HW-1 model, Brazil), a laboratory oven (Quimis Q-317 B model, Brazil), a particle size sieves (Granutest, Brazil), analytical balance (Shimadzu AY 220 model, Japan), sputter coater (Shimadzu, IC-50 Ion Coater model, Japan), scanning electron microscope (Shimadzu, SSX 550 model, Japan), infrared spectrophotometer (Cary 630 model, Agilent Technologies, Santa Clara, California, USA), and UV/Vis spectrophotometer (Even, IL-562 model, Brazil). The data was analyzed through a graphical/statistical program Origin version 6.1 (OriginLab Corporation, Northampton, MA 01060, United States).

Methods:-
BW prepare:-
BW was washed using distilled water and dried for 24 h in a laboratory oven at 70°C. After the drying, the adsorbent was triturated and sieved to obtain different particle sizes (Figure 1).

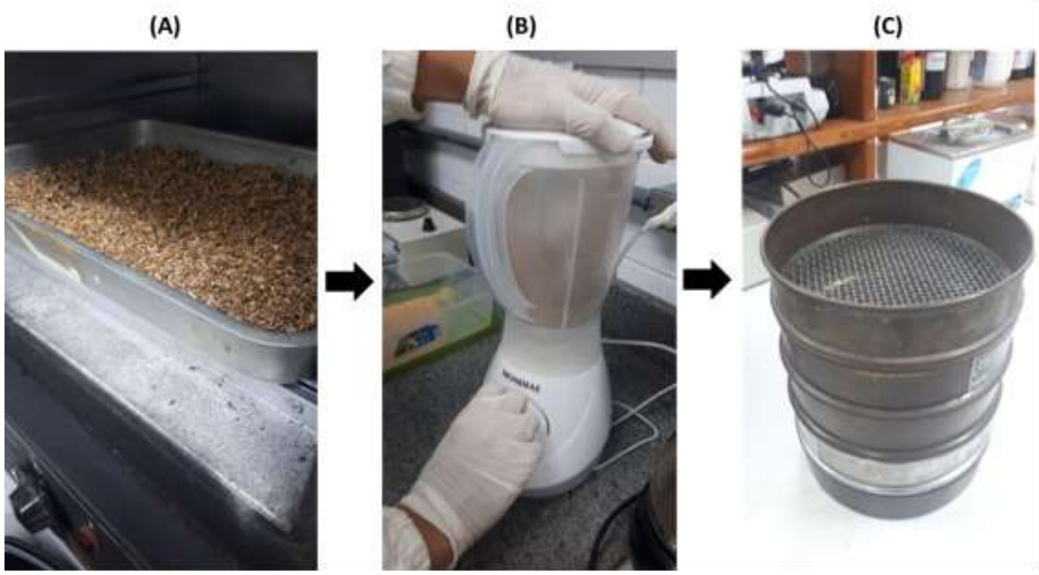

Figure 1:- Drying (A), crushing (B), and sieving (C) of BW.

BW granulometry influence:-
Aqueous solutions (25 mL) containing IB 1,000 mg/L (pH 6.5; T: 298.15 K) and 2.0 g of BW (< 0.435 mm or 0.435 mm - 1.19 mm) were magnetically stirred for 8.0 minutes at 600 rpm. Then decantation was used for 10 minutes to separate solid BW. The remaining BW was removed after centrifugation at 13,000 rpm for 3.0 minutes (T: 298.15 K) (Figure 2). After centrifugation was possible to see a precipitate in the microtube. In this separation process, the paper filter is not recommended due to the presence of cellulose in its composition. This polysaccharide is one of the main macromolecules present in several biomass [11]. Subsequently, 3.0 mL of supernatant was transferred from the microtube to a glass cuvette (Figure 3) and the IB absorbance, in 570 nm (λ_{max} of IB) was obtained using a UV/Vis spectrophotometer. The blank mean absorbance value was obtained through the same process, but the IB solution was substituted by distilled water (25 mL) containing 2.0 g of BW. Through IB absorbance value in the supernatant, it was possible to calculate the IB percentage (%) retained in BW of different granulometry.

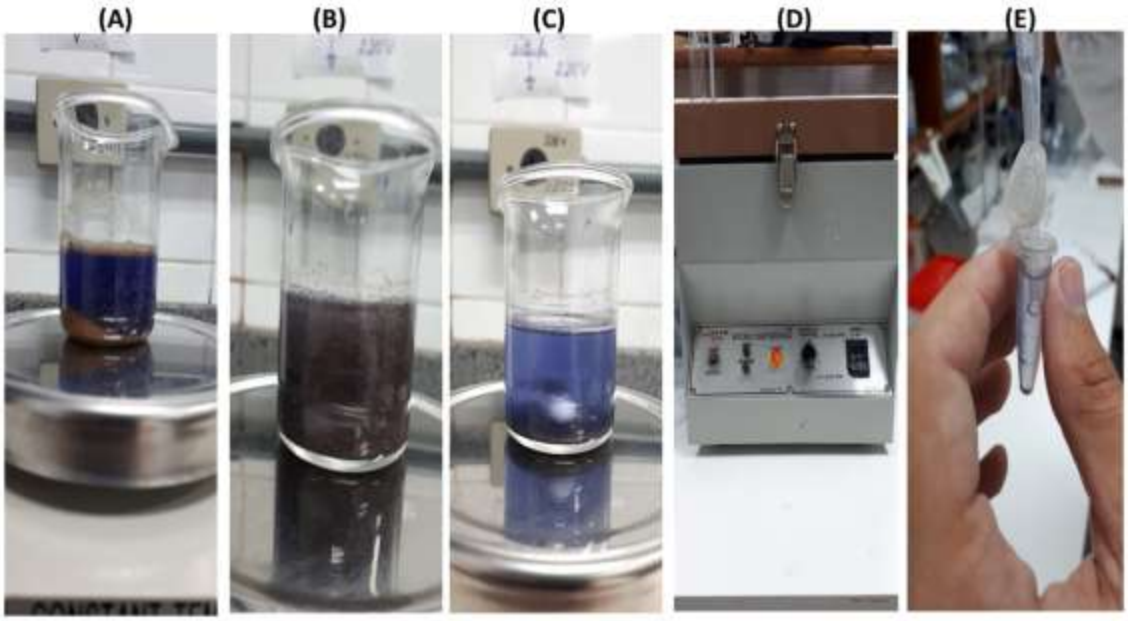

Figure 2:- IB solution with BW (A), magnetic stirring (B), decantation (C), centrifugation at 13,000 rpm (D), and supernatant for IB absorbance determination (E).

Figure 3:- Glass cuvette containing IB supernatant obtained through centrifugation at 13,000 rpm.

BW morphology analysis through SEM:-

Initially, a small amount of BW (<0.435 mm) was covered with gold particles using a metalizer. Subsequently, to visualize the morphology of BW particles, a scanning electron microscope (SEM) (electron beam of 20 kV) was used. The procedure was performed as described in the literature [3] [5] [10].

BW chemical analysis through FTIR:-

Fourier- transform infrared spectroscopy (FTIR) can be used for the detection of several functional chemical groups (FCG) present in different materials. The FCG presence is very important for chemical interaction between NA and pollutants in aqueous médium. In this work was realize the FTIR analysis for BW (< 0.435 mm). The procedure was realized as described in the literature [3] [5] [10].

BW mass, stirring speed, and stirring contact time between BW and IB:-

For these laboratory tests, aqueous solutions (25 mL, pH 6.5, T = 298 K) containing IB 1,000 mg/L were magnetically stirred at different time (0.5, 2.0, 4.0, 6.0, 8.0, 10.0, and 12.0 min), different speed (50, 250, 600, 1,000, and 2,000 rpm), and in the presence of different BW mass (0.25, 0.50, 0.75, 1.00, 1.25, 1.50, 1.75, and 2.00 g) of small granulometry (< 0.425 mm). After stirring, the remaining procedures were carried out as described in the step for BW granulometry influence.

Determination of MAC and other parameters:-

After optimization of BW granulometry, BW mass, stirring speed, and contact time it was possible to determine the maximum adsorption capacity of BW to the IB dye. The MAC value represents the pollutant amount (mg) that can be retained per 1.0 g of adsorbent [27]. To determine MAC and other parameters, aqueous solutions (25 mL, pH 6.5, T = 298 K) containing increasing concentrations of IB (100 – 2,000 mg/L) were prepared. The solutions were stirred at 500 rpm for 6.0 minutes. After decanting (10 min.) 3.0 mL of each solution was centrifuged at 13,000 rpm for 3.0 minutes. Subsequently, the IB absorbances (570 nm) in the supernatants were obtained and the different concentrations of IB were obtained through a standard curve. The data obtained made it possible to obtain a non-linearized isotherm using equation 1.

$$q = K_L \,.\, MAC \,.\, C_{eq} \,.\, (1 + K_L \,.\, C_{eq})^{-1} \quad (1)$$

q: amount of IB (mg) retained per gram of BW (mg/g)
K_L: Langmuir constant (L/mg)
MAC: maximum adsorption capacity betwenn BW and IB (mg/g)
C_{eq}: concentration of equilibrium of IB present in the supernatant (mg/L)

After linearizing the non-linearized isotherm, it was possible to obtain the linearized isotherm (equation 2):

$$C_{eq}/q = (K_L \,.\, MAC)^{-1} + (MAC)^{-1}.C_{eq} \quad (2)$$

Finally, were determined the values of MAC, K_L, variation of Gibbs Energy value (ΔG), and dimensionless constant (R_L) through equations 3, 4, 5, and 6 respectively.

$$MAC = 1/AC \quad (3)$$

AC: Angular coefficient

$$K_L = 1/(MAC \,.\, LC) \quad (4)$$

LC: Linear coefficient

$$\Delta G = -RT\ln K_L \quad (5)$$

ΔG: variation of Gibbs energy of adsorptive process between BW and IB
R: gas's constant
T: temperature in Kelvin (K)
$K_{L;}$ Langmuir constant (L/mg)

$$R_L = 1/(1 + K_L \,.\, C_0) \quad (6)$$

R_L: dimensionless constant
$C_{0:}$ the highest dye concentration for IB dye (mg/L)

IB removal from natural water sample in glass columns:-
Three water samples of Itaunas River (IR), Itaunas-ES, Brazil (Figure 4) were collected and transported to a biochemical laboratory of Espirito Santo Federal University, Vitória-ES, Brazil. In this laboratory, the pH and electrical conductivity in 50.0 mL of these IR samples (50.0 mL) were measured. These procedures were realized before contamination with IB dye (1,000 mg/L), after contamination, and after filtration. For the filtration process, the contaminated IR samples (50.0 mL) were filtered in a glass column (30.0 x 3.0 cm) containing crushed stone (16.7 g), gravel (15.3 g), sand (10.0 g), and BW (1.0 g). After filtration, 3.0 mL of filtered samples were transferred to a glass cuvette and the IB absorbance, in 570 nm (λ_{max} of IB) was obtained. Through IB absorbance value was possible to calculate the IB percentage (%) retained in the glass column. It is necessary to highlight that during the filtration process, increasing volumes of IB (50, 100, 150, 200, 250, 300, 350, 400, 450, and 500 mL) were added to evaluate the saturation of BW.

Localization -18.418743864575553, -39.70675444635576

Figure 4:- Local for water samples collect (Maxar Tecnologies: Google Maps, 2024).

Results and Discussion:-

BW's preparation:-
The sieving of BW produced two different particle sizes: < 0.435 mm and 0.435 - 1.19 mm (Figure 5).

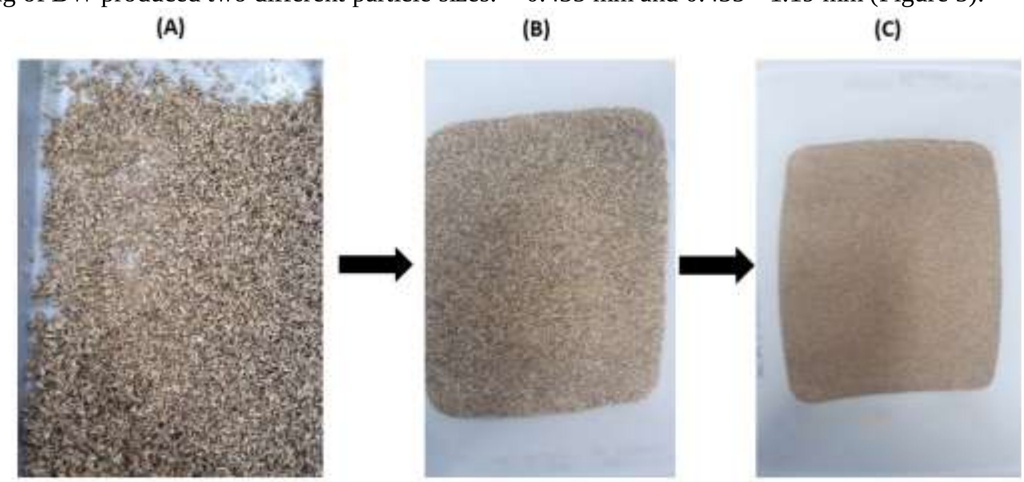

Figure 5:- BW before crushing (A). BW after crushing and obtaining particle sizes between 0.435 and 1.19 mm (B) and smaller than 0.435 mm (C).

BW granulometry influence:-
Ribeiro et al., 2019 [10] verified high IB removal (%) in aqueous solution using Ziziphus joazeiro powder of small granulometry more than major granulometry. Generally, smaller particle sizes have a larger contact surface, which favors chemical and physical interactions between the adsorbent and the pollutant. Furthermore, the flow of liquid through larger spaces between the adsorbent particles favors higher flow rates. This process provides lower percentages of adsorption and consequently, greater amounts of pollutant in the eluent. [18]. The results obtained at this stage of our work demonstrate that BW with a smaller grain size (< 0.435 mm) is more efficient than BW with a larger grain size (0.435 - 1.19 mm) (Figure 6).

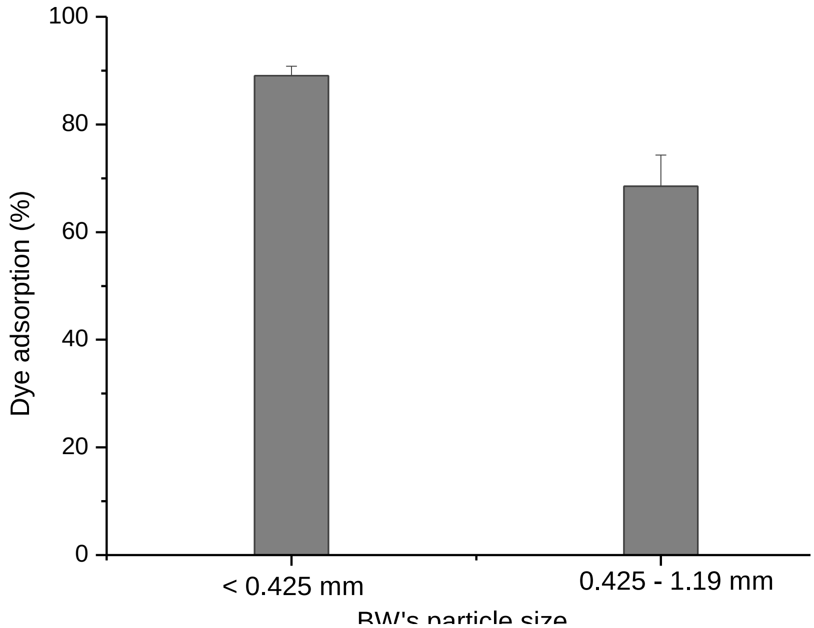

Figure 6:- Adsorption (%) of IB using two different size of BW particles.

BW morphology analysis through SEM:-
The image obtained through scanning electron microscopy (Figure 7) revealed a non-smooth surface for BW, but with the presence of regular concavities in some points. The image is similar to a set of tubes cut horizontally which is common in vegetal materials containing lignin and cellulose [19] [20]. These characteristics common to biomasses, originating from agro-industrial activities, favor the adsorptive process between natural adsorbent and chemical pollutants in aqueous medium [5] [9] [10].

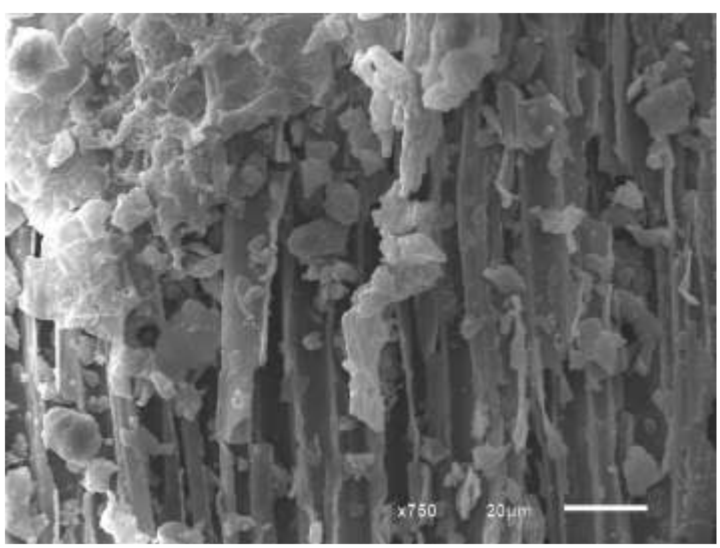

Figure 7:- SEM image for BW x750 and 20 µm.

BW chemical analysis through FTIR:-

Different chemical compounds may be found in BW such as: lipids, protein, starch, hemicellulose, cellulose, and lignin. These biomolecules have some in their structures that can favor the interaction with chemical pollutants [21]. The BW's FTIR spectra (Figure 8) are similar to the other NA's FTIR such as wood sawdust powder from Corymbia citriodora [5], banana peel [22], black jurema powder [23], green mesocarp coconut [24], blue-green marine algae [25], and the solid brewery waste itself [26]. With the use of the FTIR technique was possible to determine the presence of different FCG, very important for physicochemical interactions with IB dye. The Table 1 shows some important chemical groups identified in BW.

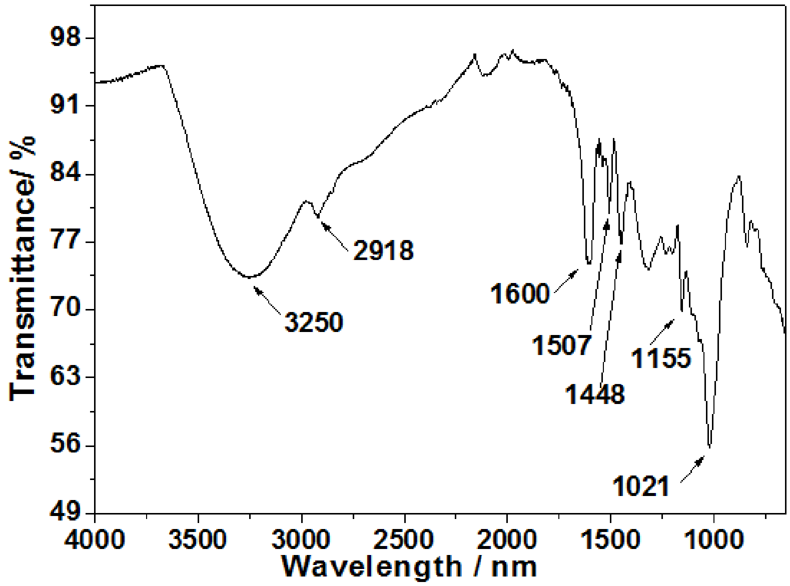

Figure 8:- FTIR spectra of BW.

Table 1:- Some important BW's FCG detected by FTIR.

FCG	Band (cm^{-1})
C-O	1,021
C=O	1,600
C-H sp^3	2,918
O-H	3,250

BW mass, stirring speed, and stirring contact time between BW and IB:-

The BW mass equilibrium was obtained with 1.0 g (Figure 9). The equilibrium of adsorption percentage occurs due to the saturation of sites adsorption present in the adsorbent. The better stirring speed (500 rpm) (Figure 10) and better contact time (Figure 11) between BW and IB (6.0 min.) was also determined. Other studies also found that the increase in the adsorbent mass led to an increase in the pollutant adsorption (%) until reaching an equilibrium in which the mass increase didn't result in more increase of pollutant removal from aqueous medium. The equilibrium of adsorption percentage occurs due to the saturation of adsorptive sites present in the adsorbent [8]. The same results were observed for the other two parameters evaluated. However, in some analyses of stirring and contact time, the occurrence of adsorption and desorption was observed [3], which wasn't observed in this work [1] [3] [5] [8] [22] [23].

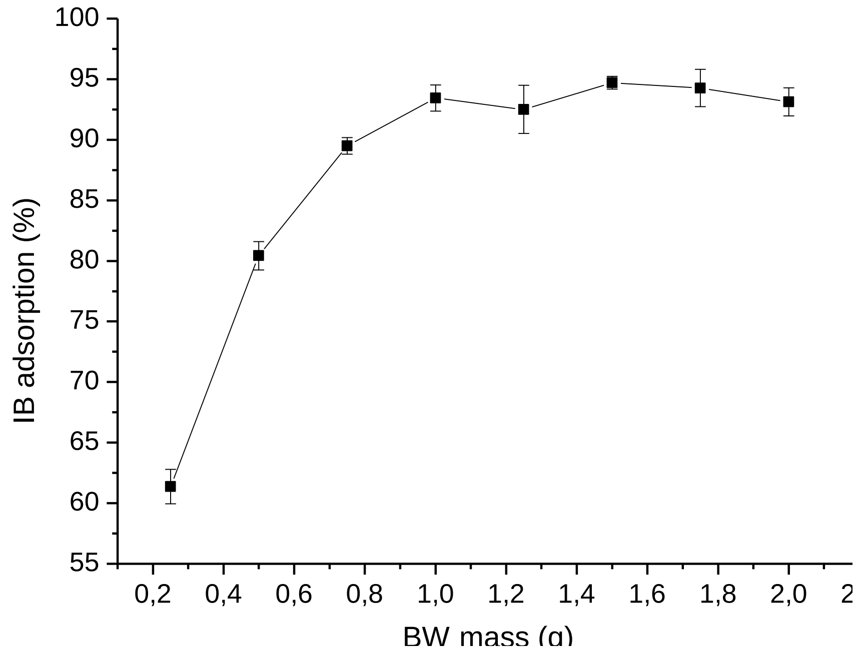

Figure 9:- Influence of BW mass (g) in IB adsorption (%).

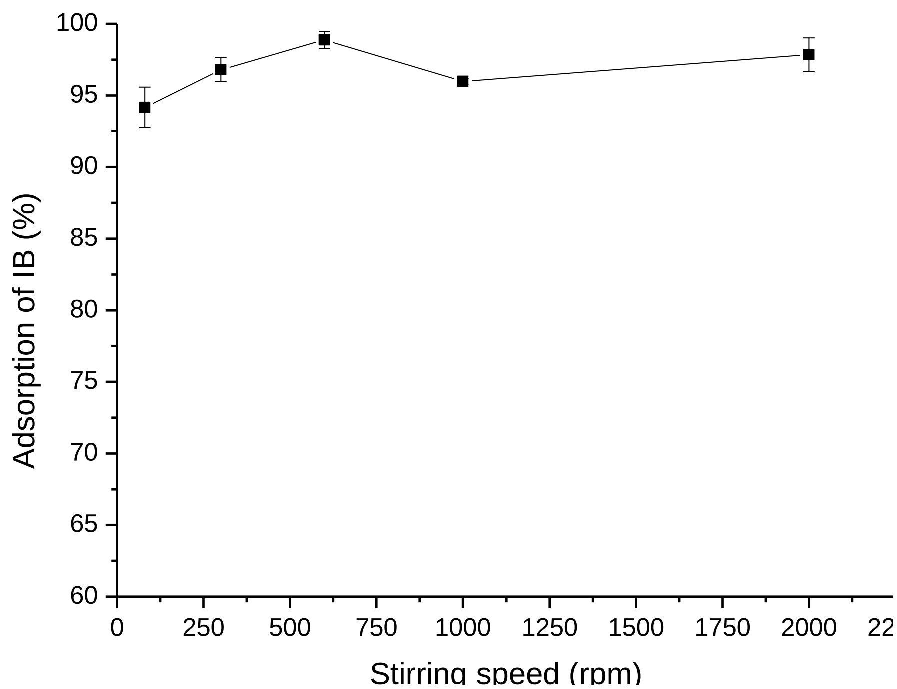

Figure 10:- Influence of stirring speed (rpm) in IB adsorption (%).

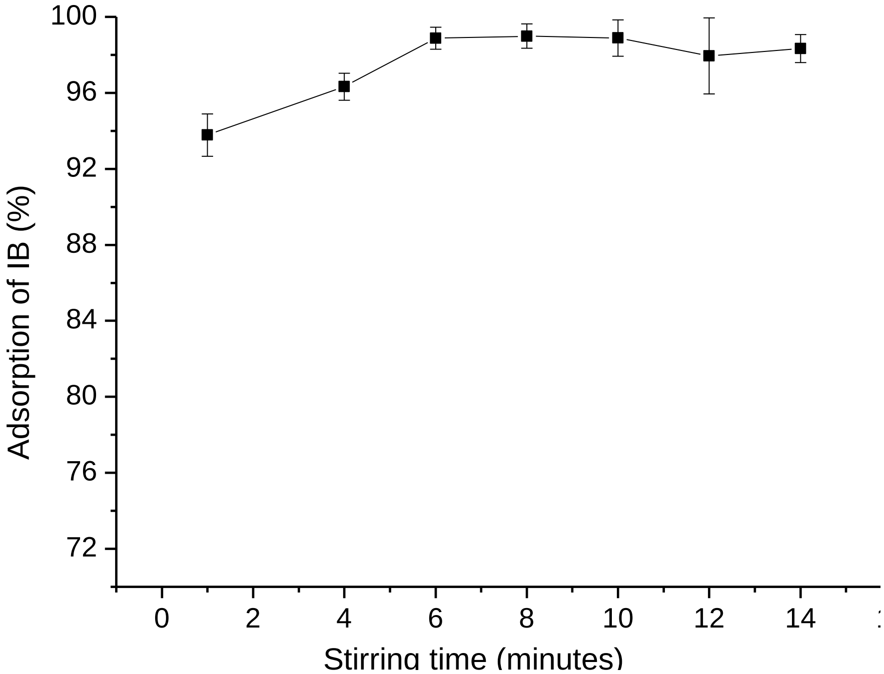

Figure 11:- Influence of contact time (min.) in IB adsorption (%).

Determination of MAC, K_L, ΔG, and R_L values:-

Figure 12 shows the non-linear isotherm (q/Ceq) for the adsorptive process between BW and IB. The R^2 (0.97899) value suggests a suitable mathematical model for this study. However, we can't state that the adsorptive process occurs in perfect monolayers, as suggested by Langmuir's mathematical model. Studies with other natural adsorbents have obtained good results, using the same mathematical model [10] [27] [28]. After linearizing (Figure 13) the non-linearized isotherm, it was possible to obtain the linearized isotherm represented by equation 2. Using this equation, it was possible to derive equations 3, 4, 5, and 6 through which the values of MAC, K_L, ΔG, and R_L were obtained respectively (Table 2):

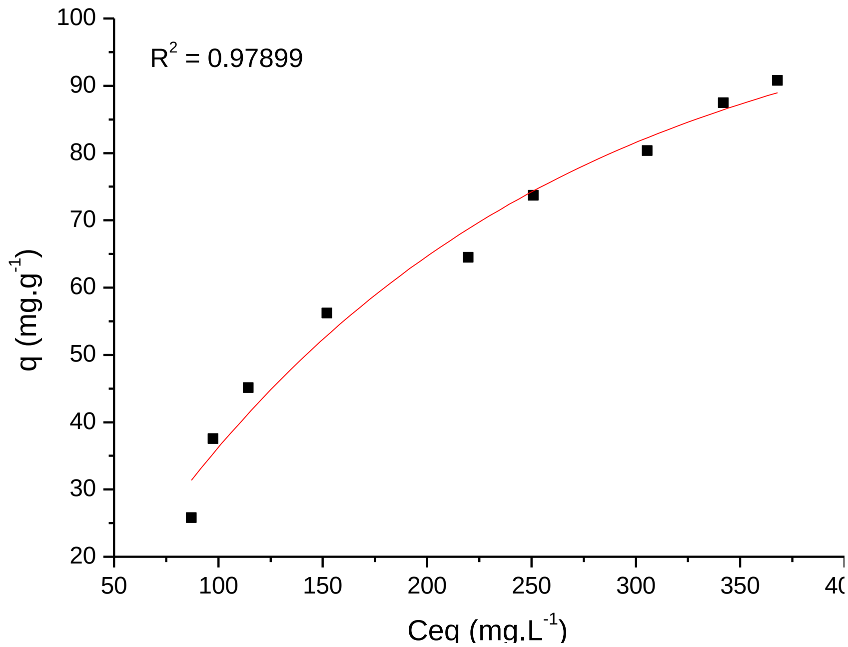

Figure 12:- Non-linearized isotherm for the adsorptive process between BW and IB.

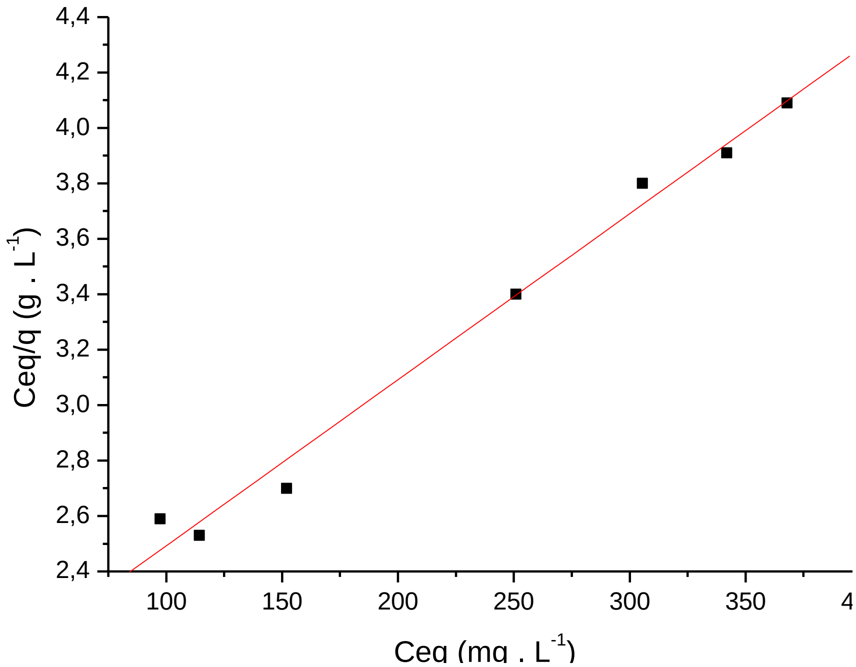

Figure 13:- Linearized isotherm for the adsorptive process between BW and IB.

Table 2:- Parameters obtained from linearized isotherm.

Parameter	Value
R	0.99391
K_L (L/mg^{-1})	0.003
ΔG (Kj/mol^{-1})	- 16.53
R_L	0.76
MAC (mg/g)	166.94

The R value (0.99391) demonstrates a satisfactory linear isotherm. The K_L value indicates good adsorption energy between BW and IB [30]. The negative value of ΔG (-16.53 KJ/mol) indicates a spontaneous interaction between BW and IB [3]. However, the interactions are more physical than chemical. The ΔG values for the predominance of chemical interaction are around -200.0 Kj.mol^{-1} [31]. The value obtained for R_L (0,76) demonstrates a favorable adsorptive process because $R_L < 1.0$ [3]. Finally, the MAC was calculated and showed a significative value (166.94 mg/g) when compared to the other values shown for synthetic and natural adsorbents used for IB removal (Table 3).

Table 3:- MAC for IB removal by different adsorbents.

Adsorbent	Dye	MAC (mg.g^{-1})	Reference
sPEEK-MMT	IB	833.30	[32]
BW	IB	166.94	This work
Natural clay	IB	57.00	[34]
Activated carbon	IB	53.00	[34]
Ziziphus Joazeiro Peel	IB	50.00	[10]
Vermicompost	IB	40.39	[3]
Chemically Modified Coconut Shell	IB	13.68	[33]
Liquorice Glycyrrhiza glabra L. Root Powder	IB	1.70	[8]

IB removal from a natural water sample in glass columns:-

The conductivity and pH were measured in natural samples from the Itaúnas River before contamination, after contamination with 1,000 mg/L of contaminated IB, and after the initial filtration process using 50 mL of IB. The data are presented in the Table 4 and demonstrate that the pH showed a small reduction after contamination and a small increase after filtration. The conductivity showed a considerable increase after contamination with IB, probably due to the dye additives, such as sodium chloride. After filtering (50 mL) of water contaminated with IB, the conductivity decreased, suggesting that the filtration process using BW is also capable of reducing the amount of additives present in the commercial dye.

Table 4:- Condutimetry and pH of water river samples before contamination, after contamination with IB, and after filtration.

Samples of river	Before IB contamination	After IB contamination	After filtration
Condutimetry (mS. cm^{-1})	0.24 ± 0.01	4.39 ± 0.04	3.20 ± 0.11
pH	6.85 ± 0.18	6.31 ± 0.04	6.53 ± 0.12

In Figure 13 it can be seen that the removal of the dye, present in the river water, was quite significant from 50 to 300 mL of sample volume. However, after 350.0 mL there was a considerable decrease in the IB dye removal capacity. It can be suggested that the increase in the volume of contaminated river water causes the saturation of the adsorptive sites of the BW adsorbent by IB and its additives.

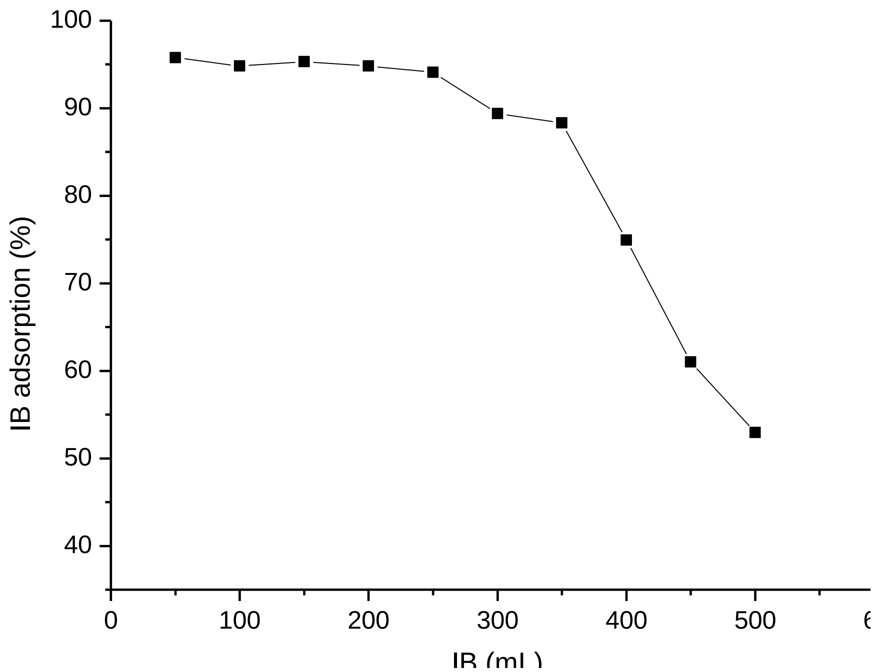

Figure 13:- Influence of the volume of river water contaminated with IB (1,000 mg/L) on the filtration process using glass columns containing crushed stone, gravel, sand and brewery waste (1.0 g).

Conclusions:-

The results obtained through SEM and FTIR suggest that the morphology and chemical groups of BW are among the factors responsible for the considerable capacity of this adsorbent in removing IB from distilled and river water. However, the optimization of BW mass, speed stirring, contact time, and IB concentration was very important to obtain the high MAC (166.94 mg/g) of BW for IB. The experiments using glass columns showed high percentage of IB removal from Itaúnas river water. Finally, the data obtained in this work suggest BW as more an alternative for IB removal in aqueous media and another alternative for BW reuse.

Conflicts of Interest:

We declare no interest's conflicts- L for this work

Acknowledges:-

We acknowledge the Cellular Ultrastructure Laboratory Carlos Alberto Redins (LUCCAR) by SEM analysis and the Instituto Federal of Aracruz by FTIR analysis.

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