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### RESEARCH ARTICLE

#### EVALUATION OF LIQUORICE *GLYCYRRHIZA GLABRA* L. ROOT POWDER AS A NEW ADSORBENT IN THE REMOVAL OF TEXTILE DYES IN AQUEOUS MEDIUM.

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#### Abstract

The liquorice *Glycyrrhizaglabra* L. root powder (GGRP) was evaluated to remove the two textile dyes in water. The physical-chemical analyzes using the scanning electron microscope and infrared spectroscopy showed that this material has irregular surface and chemical groups capable of retaining chemical pollutants in aqueous medium. In addition, our results demonstrated that it was possible to obtain a satisfactory adsorption percentage of the indigo blue (IB) and congo red dye (CR) when used 2.0 g of GGRP and stirring time at 5 minutes. The maximum adsorption capacity for IB (1.696 mg.g<sup>-1</sup>) and CR (0.768 mg.g<sup>-1</sup>) obtained through the Langmuir Mathematical Model suggest GGRP as an possible adsorbent for removal both textile dyes. This conclusion could be sustained through batch column experiments where GGRP powder was more efficient than activated carbon (13.5 ± 2.1 % for IB and 16.9 ± 3.2% for CR) in the removal of IB (98.2 ± 1.2 %) and CR (82.1 ± 3.5%). Finally, this work provided a new alternative for the use of GGRP an environmental decontaminant and not only for medicinal use.

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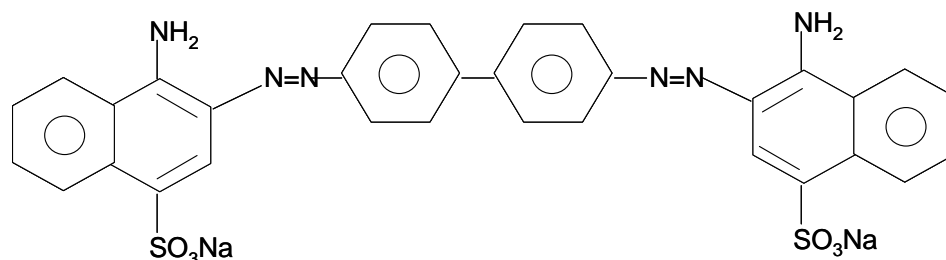
#### Introduction:-

A large number of dyes have been synthesized in the last hundred years (Guaratini&Zanoni, 2000). The high demand for these chemicals, mainly textile dyes, shows the high textile industry growth and consequently a considerable increase in the discharge of textile effluents contaminated with several types of dyes. This is worrying since most of these effluents, in many countries, are not treated or the treatments are not satisfactory (Mathur et al., 2005). The textile effluents have strong color due to the non-fixing of excessive dye added to fibers during tinting process (Kunz et al., 2002). Every year tons of textile dyes are discarded in the aquatic environment in countries such as Brazil and India (Guaratini&Zanoni, 2000; Mathur et al., 2005). Most discarded dyes have stable chemical structures and, therefore, are difficult to biodegrade (Forgacs et al., 2004). For example, congo red is an azo dye (R-N=N-R) (figure 1) considerably very stable in aqueous medium. This organic compound denominated the disodium salt of 3,3'-([1,1'-biphenyl]-4,4'-diyl)bis(4-aminonaphthalene-1-sulfonic acid) is very soluble in water and largely utilized for tinting of cellulose fiber such as cotton and viscose (Guaratini&Zanoni, 2000). Another dye widely used in the textile industry is indigo (figure 2), predominantly blue. This dye was extracted from plants, principally

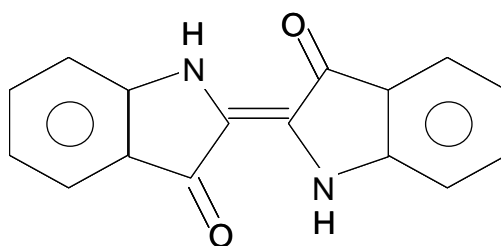
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*Indigoferatinctoria*. However, its industrial synthesis is the most used today. Indigo blue is commonly used in textile industry for the dyeing principally of common cotton and jeans (Baig, 2012).



**Figure 1:-** Chemical structure of congo red



**Figure 2:-** Chemical structure of indigo blue

One of the main problems of dyes comes from their accumulation and bioavailability. The excess of these organic compounds can be dangerous to the environment and human health (Guaratini&Zanoni, 2000). Textile dyes can affect the photosynthesis process, as well as cause respiratory problems, skin sensitivity, mutagenicity and others (Guaratini&Zanoni, 2000; Mathur et al., 2005; Raymundo et al., 2010). Therefore, studies for the development of technologies for the removal of textile dyes in water are very important. Some processes are being evaluated such as biodegradation (Adedayo et al., 2004), chemical degradation (Chen et al., 2018), photochemical degradation (Pagano et al., 2018) and adsorption processes using synthetic (Farrokhi-Rad et al., 2018) and natural adsorbents (Maghri et al., 2012).

Adsorption is a process of accumulation and selective concentration of one or more adsorbates on solid surfaces of adsorbents. The adsorbate may be present in gases (Mohammadi-Moghadam et al., 2013) or liquids (Maghri et al., 2012). In relation to the treatment of effluents the adsorbates are pollutants such as heavy metals (Jordão et al., 2010), pharmaceuticals (Ribeiro et al., 2016) textile dyes (Pereira et al., 2009) and others (Amin et al., 2015). Adsorption using natural adsorbents has been one of the most studied due to considerable availability and low cost (Ramakrishna & Viraraghavan, 1997). Some examples can be cited as: sugar cane bagasse was evaluated to removal of congo red dye (Raymundo et al., 2010) and malachite green dye (Tahir et al., 2016), vermicompost was able to removal of crystal violet and methylene blue in water (Pereira et al., 2009), wood sawdust was studied to removal of textile dyes (SahMoune&Yeddou, 2016), and banana peel powder was studied to removal of anionic dyes from aqueous solutions (Munagapat et al., 2018).

The liquorice *Glycyrrhizaglabra* L. root is more related to its medicinal properties and culinary or flavour potential (Fenwick, 1990). The use of *Glycyrrhizaglabra* L. root powder (GGRP) as an adsorbent to removal pollutants is almost absent in the literature. In 2013 researchers reported the application of *Glycyrrhizaglabra* L. root as a novel adsorbent in the removal of toluene vapors (Mohammadi-Moghadam et al., 2013). However, studies on GGRP as an adsorbent for pollutants in aqueous media are scarce. Therefore, this work aimed to evaluate the ability of GGRP as adsorbent for the removal of textile dyes in water. The congo red (CR) and indigo blue (IB) dyes were used as pollutants in this study. For this evaluation we performed studies on the physico-chemical characteristics of GGRP, influence of the GGRP mass in adsorptive process, and influence of stir time between CR or IB and GGRP. Subsequently, the maximum adsorptive capacities (MAC) of GGRP for CR and IB were determined. Finally the efficiency of GGRP was compared with activated carbon for the removal of both dyes in batch column experiments.

## Materials and methods:-

### Materials:-

The liquorice *Glycyrrhizaglabra* L. root powder (GGRP) was obtained from Empório Figueiras (Santo André-SP, Brazil), The activated carbon (AC) was obtained from Casa das Químicas (Flores da Cunha-RS, Brazil). Congo red dye (CR) (figure. 1) was purchased from Vetec Company (Duque de Caxias -RJ, Brazil). Indigo Blue (IB) (figure. 2) was purchased from Tupy Industry (São Paulo-SP, Brazil). The following equipment was used in laboratory tests: laboratory oven (Quimis Q-317 B model), particle size sieves (Granutest), Analytical balance (Shimadzu AY 220 model), sputter coater (Shimadzu, IC-50 Ion Coater model), scanning electron microscope (Shimadzu, SSX 550 model), infrared spectrophotometer (Perkin-Elmer Spectru -100 model), pHmeter (PHTEK), magnetic stirrer (Warmest), UV/Vis spectrophotometer (Biospetro SP-220 model), microcentrifuge 13,000 rpm (Evlab), and vacuum filtration pump (Primatec). Several common laboratory glassware was used such as glass columns, beakers, erlemeyers, kitassato, burette, glass funnel and others.

### Methods:-

#### Preparation of the adsorbent:-

The liquorice *Glycyrrhizaglabra* L. root powder (figure 3) was washed with distilled water pH 6.63 and pH adjusted to 7.01 using sodium hydroxide 0.01M. Subsequently the adsorbent material was dried in a laboratory oven for 15 hrs at 40°C. Finally the liquorice GGRP was sieved to obtain particles of size smaller than 1.19 mm. The material was finally stored in hermetically sealed containers for use in the later stages.



Figure 3:- The liquorice *Glycyrrhizaglabra* L. root powder

#### Scanning electron microscopy:-

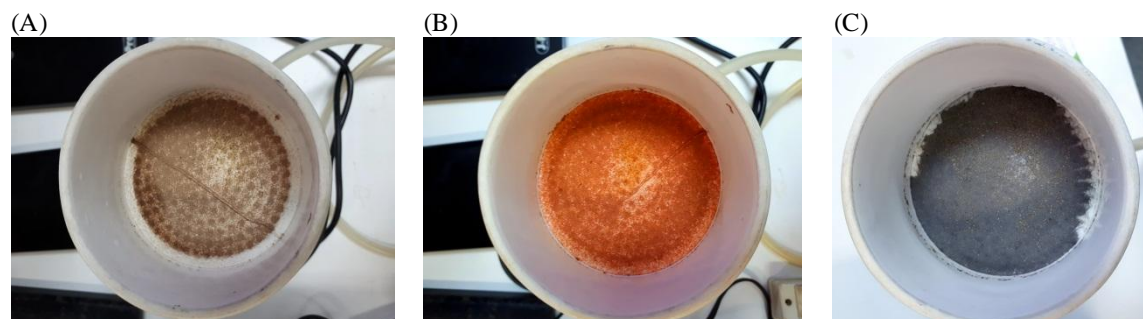
In order to know the characteristics of the GGRP surface, an analysis was performed using scanning electron microscopy (SEM). For this, a small sample of GGRP was immersed in a thin layer of gold using the spray coating. Then the surface of this sample was visualized in SEM (electron beam of 10 kV) as described in Ribeiro et al. (2018).

#### Infrared spectroscopy:-

To describe the chemical groups present in GGRP, an infrared spectroscopy (IR) analysis was performed as described in Ribeiro et al. (2016). This methodology was performed by Fourier transform IR spectroscopy using KBr discs to prepare the GGRP samples. The spectral range varied from 4000 to 500  $\text{cm}^{-1}$ . This stage aims to determine if the chemical groups of GGRP are compatible with the chemical structures of CR and IB for the occurrence of interactions favorable to the adsorptive process.

#### Stirring time:-

The 50 ml of aqueous solutions (pH 7.01) containing 87 mg/L of IB or 25 mg/L of CR were magnetically stirred (1000 rpm) at different times (minutes) at  $25 \pm 1^\circ\text{C}$ . After this procedure, the solutions were filtered at vacuum for retention of the adsorbent using quantitative paper filter (Unifil). The supernatants were centrifuged at 13,000 rpm and analyzed in a spectrophotometer at 500 nm to CR and 573 nm to IB to evaluate the amount (%) of dye retained in the adsorbent (figure 4). This step had as objective to evaluate the influence of the contact time between dyes and adsorbent in the adsorption process [6]. The temperature, stirring speed, and pH were optimized in our laboratory.



**Figure 4:-**The aqueous solutions filtered at vacuum for retention of the adsorbent GGRP in absence of dyes (A) and in the presence of CR (B) and IB (C).

#### Adsorbent mass:-

Using the conditions previously described and optimized agitation time in the previous step (5 minutes) the aqueous solutions (50 ml) (pH 7.01) containing IB (87 mg/L) or CR (25 mg/L) were magnetically stirred at different GGRP mass (g). After this procedure, the solutions were filtered at vacuum for retention of the adsorbent using quantitative paper filter (Unifil). The supernatants were centrifuged at 13,000 rpm and analyzed in a spectrophotometer at 500 nm to CR and 573 nm to IB to evaluate the amount (%) of dye retained in the different adsorbent mass. The finality of this step was to evaluate the influence of the adsorbent mass in the adsorption equilibrium process.

#### Maximum adsorption capacity:-

The equations 1 and 2 were fundamental to determine the values of maximum adsorptive capacities (MAC) of GGRP to CR and IB. These values represent the maximum amounts of these dyes which can be adsorbed by 1 g of GGRP. A high MAC value reveals that the adsorptive process is efficient:

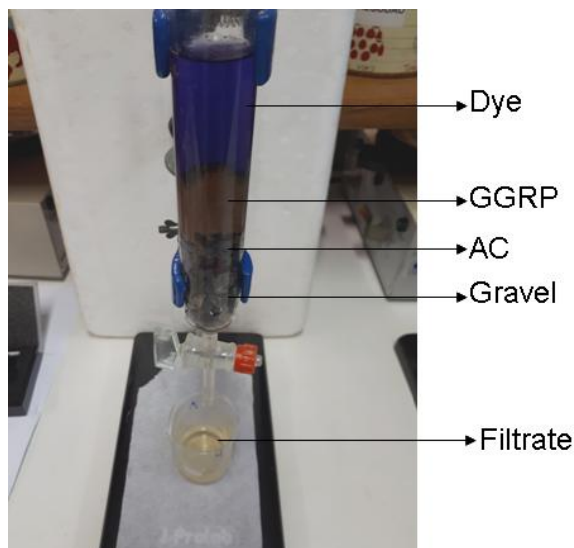
$$q = a \times b \times Eqc \times (1 + a \times Eqc)^{-1} \quad (1)$$

$$Eqc/q = (a \times b)^{-1} + (b)^{-1} \times Eqc \quad (2)$$

where  $q$  is the quantity of adsorbed dye in GGRP (mg/g),  $a$  is a constant related to adsorption energy (L/mg),  $b$  is the maximum dye adsorption capacity of the GGRP (mg/g), and  $Eqc$  is the equilibrium dye concentration (mg/L). Equation 2 is obtained by linearizing equation 1, which permits exact calculations of  $b$  values. From these equations, it was possible to calculate the maximum adsorption capacities ( $b^{-1} = MAC$ ) of GGRP to CR and IB. To obtain these MAC values, experiments were performed in which aqueous solutions (50 ml) of IB or CR at different and increasing concentrations (mg/L) were stirred in the conditions previously established (Stirring time: 5.0 minutes; stirring speed: 1,000 rpm; adsorbent mass: 2.0 g; pH 7.01; temperature:  $25 \pm 1^\circ\text{C}$ ). After this procedure, the solutions were filtered at vacuum for retention of the adsorbent using quantitative paper filter (Unifil). The supernatants were centrifuged at 13,000 rpm and analyzed in a spectrophotometer at 500 nm to CR and 573 nm to IB to evaluate the amount (%) of dye retained in the different dyes concentrations.

#### Dyes removal in glass columns:-

To test the adsorbent under almost real conditions, water samples from Espírito Santo Brazilian State Sanitation Company were enriched with 25 mg/L CR or 87 mg/L IB. The pH of these samples was around 7.0 with standardized values for colour (4 mg PT-Co/L; Hazem Unity), total dissolved solids (160 mg/L) and turbidity (2.17 NTU; Nephelometric Turbidity Unity). Subsequently, three glass columns (38 x 3 cm) were filled with 5.0 g of activated carbon (AC) in  $3.0 \pm 0.4$  cm of height and 12 g of gravel in  $5.0 \pm 0.8$  cm of height. Another three glass columns were equally filled, but with the addition of 5.0 g of GGRP in  $4.5 \pm 0.6$  cm height. Then 50 ml of dye (CR or IB) aqueous solutions were percolated through the columns (figure 5). The flow rate used was 5.0 mL / min. for all columns (optimized in our laboratory). The filtered solutions were centrifuged at 13,000 rpm and analyzed in a spectrophotometer at 500 nm to CR and 573 nm to IB to evaluate the amount (%) of dye retained in the column (Ribeiro<sup>b</sup> et al., 2016).

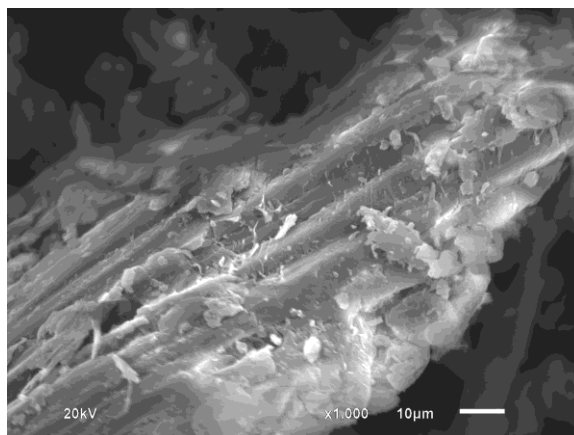


**Figure 5:-** Glass column to filtration of aqueous solutions containing CR or IB in the presence of GGRP, AC and gravel. The figure of the column in the absence of GGRP does not appear.

## Results and Discussion:-

### Scanning electron microscopy:-

The SEM analysis revealed that the GGRP surface is not homogeneous (figure 6). Surface with irregularities are crucial for efficient adsorption and have been observed in natural adsorbents studied for the removal not only of dyes (Pereira et al., 2009; Ribeiro et al., 2018) but also of other pollutants (Ribeiro et al., 2014; Ribeiro et al., 2016; Ribeiro<sup>b</sup> et al., 2016).



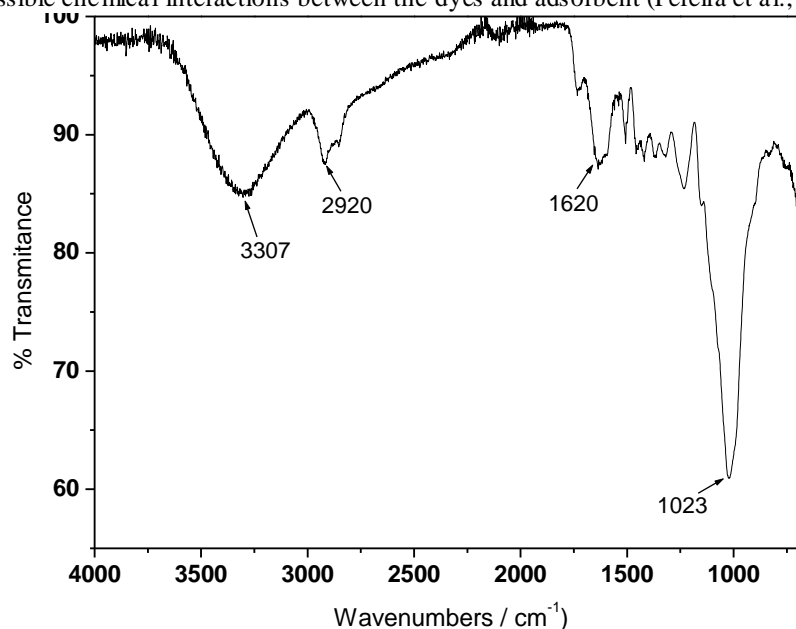
**Figure 5:-** Scanning electron microscopy of *Glycyrrhizaglabra* L. root powder with magnifications of 1000x

Pereira et al. (2009) verified that not homogeneous surface of vermicompost was important for cationic dyes removal in aqueous medium. Ribeiro et al. (2018) and Ribeiro et al. (2016) showed that heterogeneous surface of wood sawdust powder and green mesocarp coconut could be related to the efficient adsorption of CR and drugs respectively. Additionally Ribeiro et al. (2014) proposed that the irregular surface of sugarcane bagasse could be one of the factors responsible for the efficiency in the removal of tetracycline in water. Finally Pereira et al. (2012) showed that SEM and other techniques revealed desirable adsorption of heavy metals by sediments, clay and total organic matter (% , m/m) in JacuibeRiver's estuary (northeastern Brazil).

### Infrared spectroscopy:-

The chemical composition of liquorice root is complex (Fenwick, 1990). Approximately 5 to 24% of its composition is due to a biologically active triterpene glycoside (glycyrrhizin), 30% starch, and 3-16% sugar (Duke, 1985) such as glucose, maltose, sucrose, and fructose. It was also noted the presence of dextrans, pentosans, lignin, and others

(Fenwick, 1990). One can not fail to mention the presence of others several chemical compounds such as flavonoids, isoflavonoids, chalcones, coumarins (Lutomski, 1983), aminoacids, and others low molecular weight compounds (Fenwick, 1990). The presence of this diversity of chemical compounds gives the aqueous extracts of the liquorice many therapeutic and others properties (Fenwick, 1990). The IR analysis of GGRP (figure 6) shows similar characteristics to the spectra of other adsorbents of vegetable origin (Ribeiro et al., 2014; Ribeiro et al., 2018). The presence of spectroscopic bands related to some chemical groups possibly is due the occurrence of biomolecules such as cellulose, hemicellulose, lignin and others (Ribeiro<sup>b</sup> et al., 2016; Ribeiro et al., 2018). The IR of GGRP (figure 6) revealed the presence of O-H group ( $3307\text{ cm}^{-1}$ ) probably from the alcohol and other compounds with axial deformation due to intermolecular hydrogen bonds. The C-H from methyl group ( $2920\text{ cm}^{-1}$ ) and probably C=O (carbonyl) in  $1620\text{ cm}^{-1}$ , also were revealed. Additionally the presence of C-O group was also detected at approximately  $1023\text{ cm}^{-1}$ , probably due to axial deformation present in C-O-C system (Wong, 2015). Most of these chemical groups must belong to insoluble biomolecules, since the process of washing the powder with water should remove most of the soluble compounds. The presence of these and other chemical groups is to fundamental relevance to the possible chemical interactions between the dyes and adsorbent (Pereira et al., 2009).



**Figure 6:** -IR spectro of of *Glycyrrhizaglabra* root powder

#### **Stirring time:-**

The time of contact between adsorbent and adsorbate is important to determine in which time equilibrium will be established in the adsorption percentage. His step determines one of the parameters required in the test that establishes conditions for calculating the MAC value. Raymundo et al. (2010) demonstrated the percentage of CR adsorption by sugarcane bagasse increases according to the stirring time. However, after a certain time, the adsorption decreases suggesting an adsorption process accompanied by desorption. A similar result was observed by Ribeiro et al. (2016) when evaluating acetylsalicylic acid retention by banana peel. However Ribeiro et al. (2018) demonstrated that the adsorption of CR by wood sawdust powder from *Corymbiacitriodor* increases up to 5 minutes and from this point remains constant. A similar result was found in this study. For both CR (figure 7) and IB (figure 8) the maximum adsorption time was in 5 minutes remaining constant from this point.

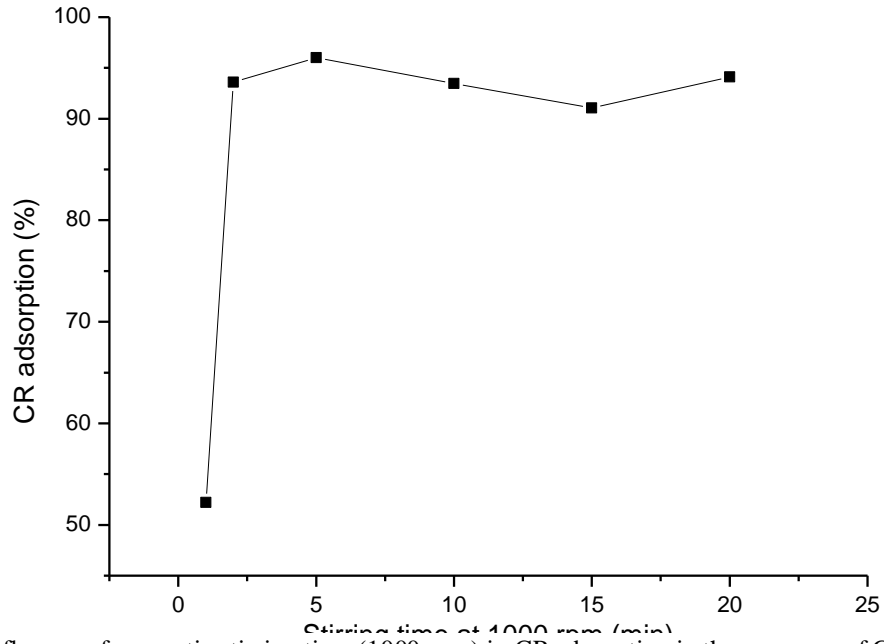


Figure 7:- Influence of magnetic stirring time (1000 rpm) in CR adsorption in the presence of GGRP

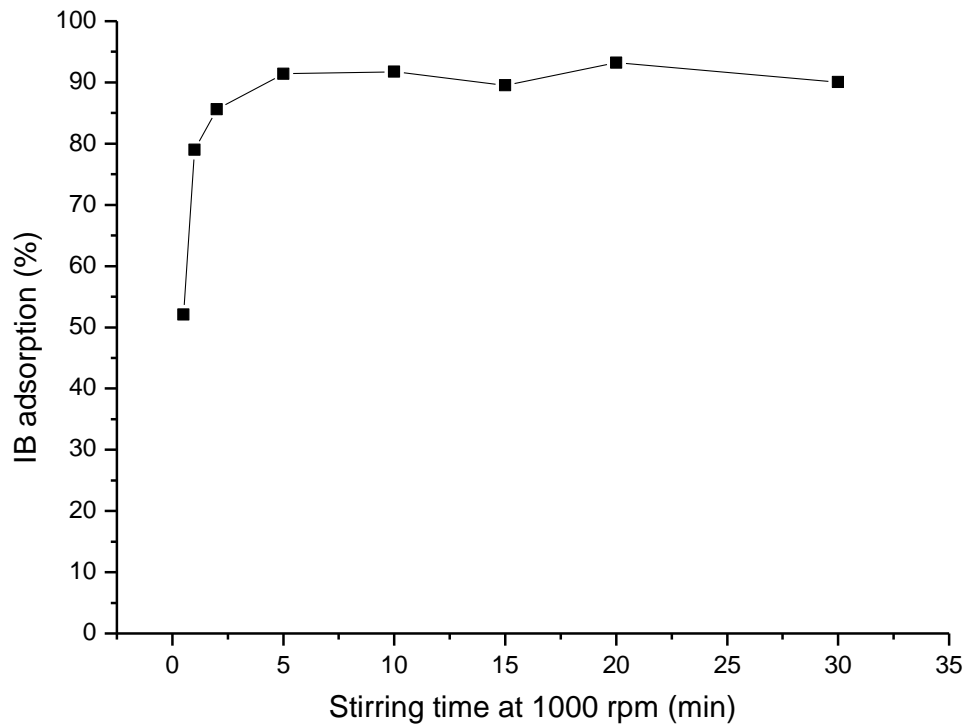
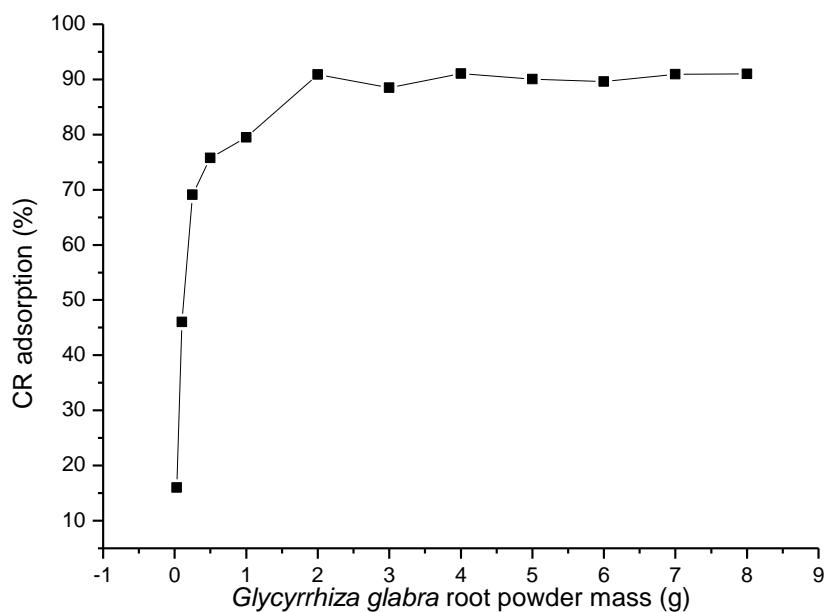


Figure 8:- Influence of magnetic stirring time (1000 rpm) in IB adsorption in the presence of GGRP

**Adsorbent mass:-**

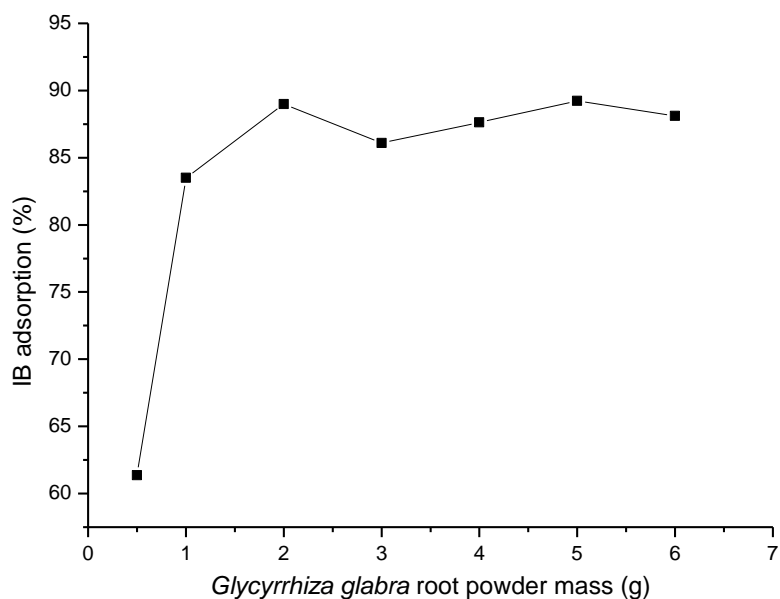
Ribeiro et al. (2018) evaluated the influence of the mass of the wood sawdust powder adsorbent from *Corymbiacitriodora* (WSPECC) to remove the CR in aqueous medium. The maximum percentage of CR adsorption

was obtained in the presence of 1.5 g of WSPECC. From this mass the percentage of adsorption remained constant and reached equilibrium. Raymundo et al. (2010) demonstrated that the increase in the mass of sugarcane bagasse powder increased the percentage of CR adsorption. The results indicated that the maximum adsorption (88%) occurred with the use of 10 g of adsorbent and then remained constant reaching equilibrium. Pereira et al. (2009) demonstrated that crystal violet dye was efficiently retained on vermicompost ( $99.9 \pm 0.07\%$ ) when 4.8 g of the referred adsorbent was employed. Moreover, a vermicompost mass of 0.6 g was necessary to retain  $99.0 \pm 0.1\%$  of methylene blue dye. According to the authors this expressive difference in the adsorbent mass can be attributed to steric hindrance, since molecules of methylene blue are smaller than those of crystal violet. It is suggested that the increase in the amount of adsorption with the increase of the mass of the adsorbent occurs due to the increase in the availability of adsorptive sites, until the process reaches the adsorption equilibrium. Similar results were observed in the present study. For both CR (figure 9) and IB (figure 10) the increase in GGRP mass resulted in an increase in the adsorption percentage of the dyes. Interestingly for both the maximum adsorption was established with the use of 2.0 g of GGRP. This may be related to similar aspects in the interactions of both dyes with the adsorbent.



**Figure 9:** - Influence of adsorbent mass in CR adsorption





**Figure 10:-** Influence of adsorbent mass in IB adsorption

**Maximum adsorption capacity:-**

Through the data obtained in this step the adsorption isotherms for CR (figure 11) and IB (figure 12) were elaborated. After linearization of these isotherms (figures 13 and 14) it was possible to calculate the MAC values for CR ( $0.768 \text{ mg.g}^{-1}$ ) and IB ( $1.696 \text{ mg.g}^{-1}$ ). As previously described, these values represent the maximum pollutant that can be retained in 1 g adsorbent (GGRP). The preparation of the isotherms and the calculation of the MAC values were performed following literature recommendations (Pereira et al., 2003; Maghri et al., 2012). The MAC values obtained in this work can be considered satisfactory and suggest GGRP as another alternative in the treatment of effluents contaminated with CR and IB. Obviously, GGRP can not be suggested as a filter for water treatment plants, since said adsorbent still needs to be evaluated for the removal of several other types of pollutants. Other works using different natural adsorbents for the removal of organic pollutants in aqueous medium also revealed satisfactory MAC values as described in Table 1

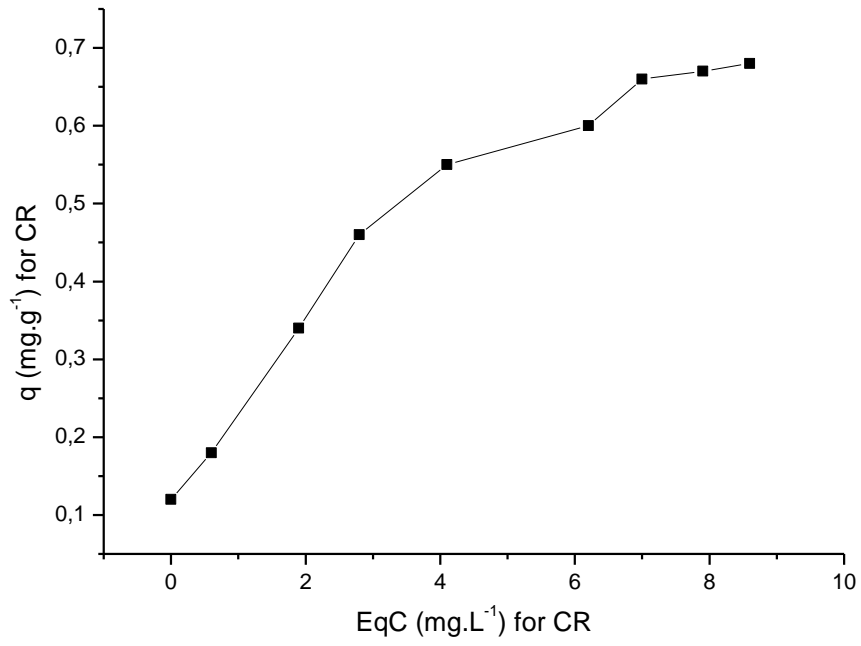


Figure 11:- Adsorption isotherm for CR

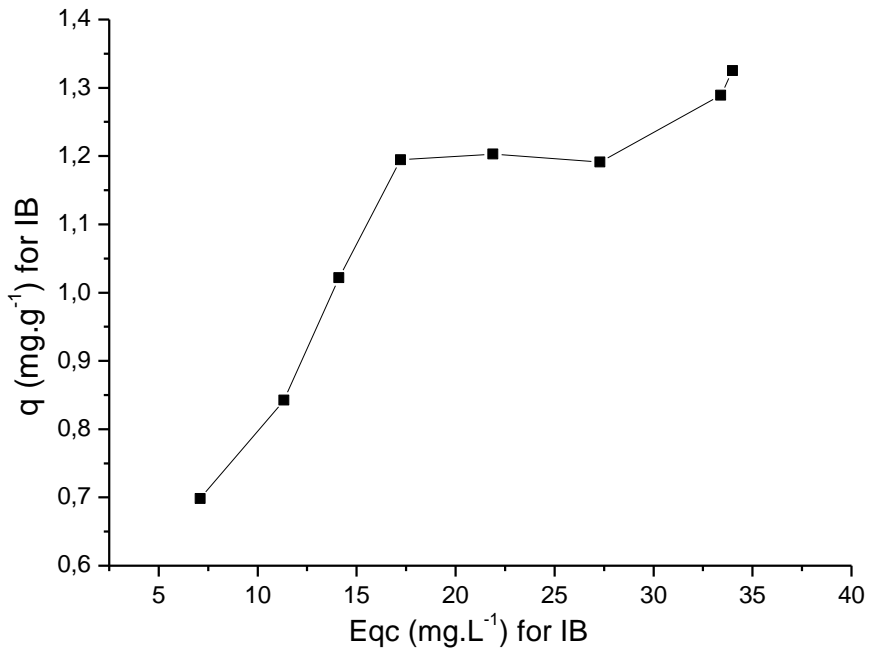


Figure 12:- Adsorption isotherm for IB

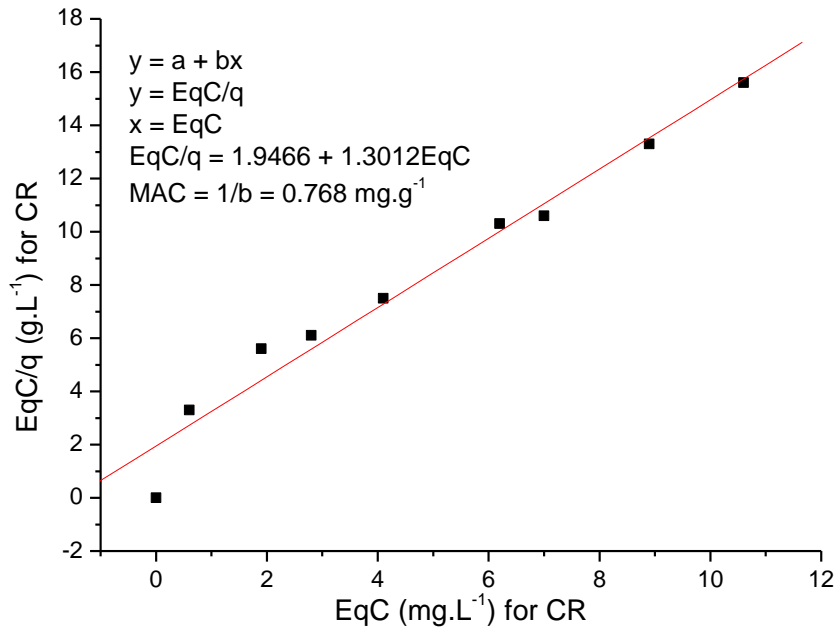


Figure 13:- Adsorption isotherms for CR after linearization.

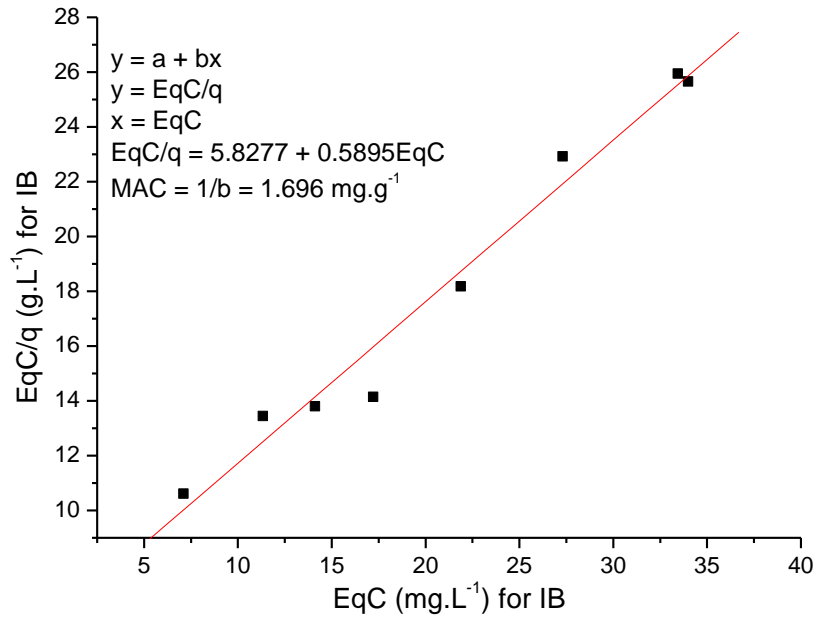


Figure 14:- Adsorption isotherms for IB after linearization.

Table 1:- Different MAC values for different natural adsorbents and organic pollutants

Adsorbent	Pollutant	MAC values (mg/g)	References
Red mud	Congo red	4.050	Namasivayam&Arasi, 1997
Sugarcane bagasse	Congo red	4.430	Ray mundo et al., 2010

Sugarcane bagasse	Tetracycline	0.310	Ribeiro et al., 2014
Wood sawdust powder from Eucalyptus	Congo red	0.523	Ribeiro et al., 2018
Green coconut mesocarp	Tetracycline	1.610	Ribeiro <sup>b</sup> et al., 2016
Green coconut mesocarp	Paracetamol	0.110	Ribeiro <sup>b</sup> et al., 2016
Banana peel	Acetylsalicylic acid	2.290	Ribeiro et al., 2016
Vermicompost	Crystal violet	0.780	Pereira et al., 2009
MytilusEdulis Shells	methylene blue	3.430	Maghri et al., 2012

### Dyes removal in glass columns:-

Experiments using glass columns containing activated carbon and gravel in the presence and absence of GGRP demonstrated that the percentages of adsorption for both IB and CR are higher in the presence of GGRP than in the presence of activated carbon alone. Table 2 shows the values of the adsorption percentages for both dyes in the presence and absence of GGRP. In another work (Raymundo et al., 2010) using sugarcane bagasse, the authors demonstrated that this natural adsorbent was very efficient in the removal of CR in effluent ( $94 \pm 5\%$ ) enriched with this pollutant.

**Table 2:** Adsorption of CR and IB in the absence and presence of GGRP

System	IB adsorption (%)	CR adsorption (%)
AC + Gravel	$13.5 \pm 2.1$	$16.9 \pm 3.2$
AC + Gravel + GGRP	$98.2 \pm 1.2$	$82.1 \pm 3.5$

### Conclusion:-

The values of maximum adsorptive capacities and mainly the percentage of retention of the dyes in the columns can be considered satisfactory. The results of the experiments show that GGRP may possibly be used as a further alternative in the treatment of textile effluents containing CR and IB. However, more detailed studies will be needed to make GGRP a viable alternative filter. These studies will include, for example, the evaluation of GGRP as agent to remove other pollutants in aqueous medium and chemical composition of the filtrates after percolation in columns.

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