ADSORPTION WITH ACTIVATED CARBON PRODUCED FROM TUNG FRUIT (*Aleurites fordii*) HUSKS

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Abstract— In this study, we investigated adsorption using activated carbon produced from the husks of tung (*Aleurites fordii*) fruits. These husks are obtained in the tung oil production. Activated carbon was produced from ground husks that were either treated or not treated with $ZnCl_2$ and then carbonized at 650°C, increasing from 3°C min^{-1} to 700°C (10 min), followed by -5°C min^{-1} to 200°C in an inert atmosphere or in an aerobic atmosphere. The adsorption capacity of the activated tung carbon was evaluated in the laboratory using methylene blue (MB) dye as a standard adsorbate. The results demonstrated that the method for producing the most effective active carbon from tung husks involved $ZnCl_2$-treatment prior to carbonization. This activated carbon product adsorbed 83% of the MB dye in 60 min and removed more than 97% of the MB within 24 h. Activated carbon produced with a precursor material with lower granularity (>0.5 mm) displayed better adsorption efficiency. Scanning electron microscopy of the $ZnCl_2$-treated carbonized material revealed a heterogeneous, irregular surface with tubular structures that most likely increased the surface area. BET analysis demonstrated that the chemical treatment increased the surface area of the carbonized husks from 340.32 m^2 g^{-1} to 2201.57 m^2 g^{-1}.

Keyword- chemical treatment, methylene blue, active carbon, tung husk

I. INTRODUCTION

One of the crops currently under study for biodiesel production is the tung (*Aleurites fordii*) plant, which is of Asian origin and whose extracted oil is widely used in the chemical industry [1]. In the process of extracting oil from the tung plant, lignocellulosic byproducts such as seed cake and/or fruit husk are generated. Given the increased cultivation of the tung plant and the increased production of its oil, the uses of the tung by-products are being studied with the goal of applying them in technological processes.

The tung is one of the vegetables that can be used for biodiesel production. Tung is favored in southern Brazil for its longevity and oil content and also the climate for planting is advantageous. The productivity of tung oil ranges from 300 to 450 kg ha^{-1}, the oil content of the fruit can range from 14-20% and from 30-40% in the seeds. Consequently, the solids waste are produced from fruit (husk) and from oil extraction. Tung husks are 38,5 % of the fruit.

Using tung by-products to adsorb organic contaminants is one possible application, as other adsorbents have produced from plants, such as those from the stalks, seed cake and husk of *Ricinus communis* and those from rice straw [2,3].

Activated carbon is one of the adsorbent materials produced from plant waste. Activated carbon is a carbonaceous material with a porous structure, typically with a high surface area and high porosity that enables it to adsorb molecules present in liquid and gas phases. Currently, this material is used in treatments such as purification, deodorization, detoxification, filtration, discoloration, dechlorination, and the removal of organic and inorganic substances, among others. Those applications have made activated carbon a product of great interest to many economic sectors in various fields. The features of activated carbon are affected mainly by what precursor material is selected and the methods used in its preparation. Furthermore, the adsorption capacity of carbon is highly dependent on the surface area, pore distribution and the occurrence of functional groups on the surface of the adsorbent material [4].

Altenor, *et al.* [5] described activated carbon as a key material for molecular adsorption; however, activated carbon is expensive, even though it is frequently used. Some of its environmental applications include the

removal of heavy metals from aqueous solutions, in particular, copper and mercury [6,7] ions as well as the adsorption of dyes [8-10], gases such as methane [11] and hydrogen sulfide [12,13].

The precursor materials of activated carbon are enriched during the heat treatment, which is performed so as not to melt them but rather to promote the formation of micropores. When the porosity of the precursor material is low, it must be activated. Thus, agricultural wastes are commonly used as raw materials for the production of activated carbon because they are carbon- and lignocellulose-rich materials that require less expensive processing [14].

Koroishi, *et al.* [15] studied the adsorption of Red Remazol RG textile dye by activated carbon produced from coconut husks. In comparison with sawdust and seaweeds, it had the highest adsorption capacity among the three adsorbent materials.

Gonçalves, *et al.* [16] used yerba mate waste as raw materials for activated carbon production. Yerba mate was carbonized and tested as an adsorbent in the treatment of three different wastewaters: those containing reactive red dye, methylene blue dye, or the herbicide atrazine. The activated carbon obtained exhibited a high specific surface area with abundant micropores and demonstrated satisfactory removal of the pollutants.

Brum, *et al.* [17] characterized the activated carbon produced from the waste of coffee processing. The raw material, carbonized and chemically activated with $ZnCl_2$, was subjected to adsorption tests with MB dye and demonstrated a satisfactory maximum adsorption capacity that was comparable to that of commercial activated carbon.

The potential of olive pomace for activated carbon production was evaluated in a study published by Demiral, *et al.* [18]. The precursor material was physically activated with water vapor and carbonized at a temperature of 900°C for a period of 45 minutes, resulting in a surface area of 1,106 $m^2\ g^{-1}$ with a total pore volume of 0.6067 $cm^3\ g^{-1}$.

In most of these studies, and at the industrial level, activated carbon is obtained through two steps: precursor material carbonization via burning and activation. The performance of the activated carbon is related to its chemical properties and the quality of it porous structure. Although the processing conditions can affect the structure and properties of the final product, these are primarily determined by the nature of the precursor material.

Activation can be performed through physical or chemical processes. The activation processes aim to obtain a microporous carbon by removing organic components and other residues that may clog the pores. These techniques result in electrically unsaturated sites with an enhanced adsorptive capacity [19].

In the chemical activation process, the precursor is impregnated with an activating agent, which may be phosphoric acid (H_3PO_4), calcium chloride ($CaCl_2$) [20] , zinc chloride ($ZnCl_2$), sulfuric acid (H_2SO_4), potassium hydroxide (KOH) [21] or sodium hydroxide (NaOH), among others [22]. These agents have in common a dehydrating capacity that affects decomposition by pyrolysis [3]. Dehydration reactions in the initial pyrolysis are intramolecular and give carbonyl groups and unsaturated carbon bonds forming intermolecular crosslinks between fibres. As result, the obtained material is more rigidity [23].

Alkaline hydroxides are important activating agent. Activated carbon produced from rice husk by treatment with NaOH as the activating agent, carbonization at 800°C and washing with hydrofluoric acid (HF) and water (to obtain a silica-free material) was evaluated and displayed a surface area of 1,380 $m^2\ g^{-1}$ with a micropore volume of 0.76 $cm^3\ g^{-1}$ [3]. The precursor material without treatment, washed with water and HF only, exhibited a surface area of 530 $m^2\ g^{-1}$ with a micropore volume of 0.29 $cm^3\ g^{-1}$. With KOH activation, pine wastes carbon presented a total micropore volume of 0.678 $cm^3\ g^{-1}$ and surface area of 1,908 $m^2\ g^{-1}$ [21].

Mestre, et al. [22] evaluated the adsorption potential of activated carbon prepared from sisal waste, which was chemically activated with K_2CO_3. The carbonization temperature was 700°C (1 h), yielding carbon with a surface area of 1,038 $m^2\ g^{-1}$ with a total pore volume of 0.49 $cm^3\ g^{-1}$, comparable to the values for commercial activated carbon (1,065 $m^2\ g^{-1}$ surface area /0.70 $cm^3\ g^{-1}$ pore volume).

Two different activation methods were tested in the study [5]: physical activation with water vapor, which resulted in a surface area of 1,185 $m^2\ g^{-1}$ and a total pore volume of 0.69 $cm^3\ g^{-1}$; and chemical activation with H_3PO_4, which resulted in a carbon product with a surface area of 1,272 $m^2\ g^{-1}$ with a pore volume of 1.19 $cm^3\ g^{-1}$. The micropore volume of the Vetiver roots carbon in both processes was similar: 0.36 $cm^3\ g^{-1}$ for the physically activated carbon and 0.39 $cm^3\ g^{-1}$ for the chemically activated carbon. The difference in the total pore volumes was due to their mesopore volumes, which were 0.33 and 0.80 $cm^3\ g^{-1}$, respectively.

Chemical activation with $ZnCl_2$ and carbonization at 800^0C also provided excellent results in the production of activated carbon from olive pits. The result was a microporous carbon (0.6 $cm^3\ g^{-1}$) with few larger pores [11].

H_3PO_4 and $ZnCl_2$ were tested as chemical activation agents of the pits of the sea-buckthorn fruit, which, following burning for 3 h at 550°C, resulted in products with 1,071 and 829 $m^2\ g^{-1}$ of specific surface area,

respectively. Furthermore, both of these materials demonstrated efficiency in the adsorption of lead – Pb (II) [24].

$ZnCl_2$ was also reported as a promising compound for producing activated carbon from lignin (700 $m^2\ g^{-1}$ of surface area) [14] and from palm tree seeds [25] that exhibited better adsorption efficiency than that of activated carbon treated with H_3PO_4. In addition, Khalili, *et al.* [26] evaluated paper waste as a precursor for activated carbon production and conducted the experiments with different $ZnCl_2$: precursor material ratios. They observed that both the specific surface area and the pore volume were increased by increasing the $ZnCl_2$: precursor material ratio. The most effective carbon was obtained using a ratio of 2.5:1 ($ZnCl_2$: precursor material), which had a surface area of 1,249 $m^2\ g^{-1}$ and a total pore volume of 1.128 $cm^3\ g^{-1}$, 0.486 $cm^3\ g^{-1}$ of which were micropores.

Thus, given the need for new applications for the plant waste from the oil extraction step of biodiesel production, the present study aimed to evaluate the use of tung (*Aleurites fordii*) husk for activated carbon production and its potential use in the removal of dyes, including methylene blue, from aqueous solutions. For this purpose, the material characteristics and its optimal processing and operation conditions, the effect of the time of contact between the adsorbent and the adsorbate, the particle size of the adsorbent material used, and the initial concentration of the adsorbate on the efficiency of dye removal from the solution were examined.

II. EXPERIMENTAL SECTION

II.1 Sampling

The precursor material used for absorbent production was tung husks. These husks were provided by Cooperfumos do Brazil SA (Santa Cruz do Sul/RS). The ground tung husks were passed through sieves for fractionation into three sizes of particles: < 0.5 mm, 0.5-1.0 mm, and 1.0-2.0 mm. After sieving, the samples were washed with deionized water and dried at 110°C for 1 h.

II.2 Activated carbon production

The activated carbon was produced in a furnace with N_2 entrance (Metal Trend MT49) at 650 °C, increasing 3 °C min^{-1} up to 700 °C (10 min), followed by -5 °C min^{-1} to 200 °C and , with or without N_2 atmosphere (2.6 L min^{-1}). The samples were previously prepared with a 24-h chemical treatment with $ZnCl_2$ in 1:1 proportion in relation to raw material. After the material burning with $ZnCl_2$, the solids were washed with hydrochloric acid solution (10%) and it was added water until pH 7 was reached. The adsorbents will be identified in the graphics as comminuted tung husk (T), activated carbon from tung husk (CT), activated carbon from tung husk that was treated with $ZnCl_2$ (TCT), and activated carbon from tung husk that was treated with $ZnCl_2$ and carbonized in an inert atmosphere (TCT-N_2).

II.3 Adsorption assay

The methylene blue adsorption assays (MB - $C_{16}H_{18}ClN_3S.3H_2O$, λ_{max} = 660 nm) were performed with all of the adsorbents produced.

The adsorption assays were conducted in triplicate in erlenmeyers flasks with lids,with 30 mL of MB solution in an orbital shaker (MA 420 – Marconi) rotating at 50 rpm, at 30°C and pH 7. After adsorption assay the samples were filtered in syringe filter (40 µm, Whatmann).

In each adsorption assay, the MB concentration was determined by visible spectroscopy in equipment from Shanghai Mapada Instruments Co. model V-1200.

The adsorption time was evaluated using 0.2 g of the adsorbents prepared from the three different sized particles in a 10 mg L^{-1}MB solution avoiding the MB dimer formation in aqueous solution [27]. Different quantities of the adsorbents quantities and MB concentrations were also tested.

The statistical analyses were performed with ANOVA using Minitab® statistical software 3.00.

II.4 Superficial characterization

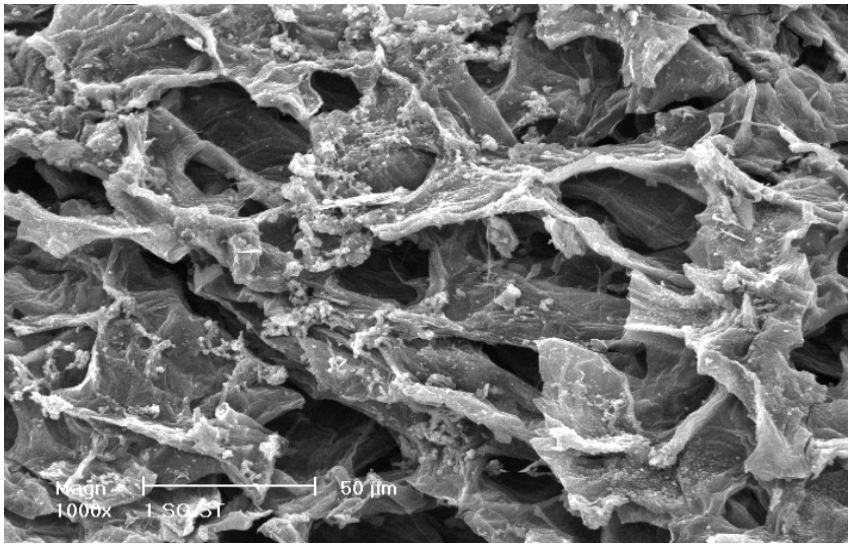
The activated carbon products were characterized by scanning electron microscopy using Vega Plus TS 5136MM, TeScan and JEOL JSM 6060 microscopes. The specific surface area was obtained by Brunauer-Emmett-Teller (BET) method, using an Autosorb Quantachrome Instrument (model Nova 1000). The BET analysis consisted of passing a gas through a cell containing a known mass of a solid sample that had been pretreated by heating it at 70°C for 3 h in a vacuum. Five N_2 adsorption points were recorded at the temperature of -196°C (liquid nitrogen). The equipment used was in the LACER Laboratory in the Federal University of Rio Grande do Sul.

III. RESULTS AND DISCUSSION

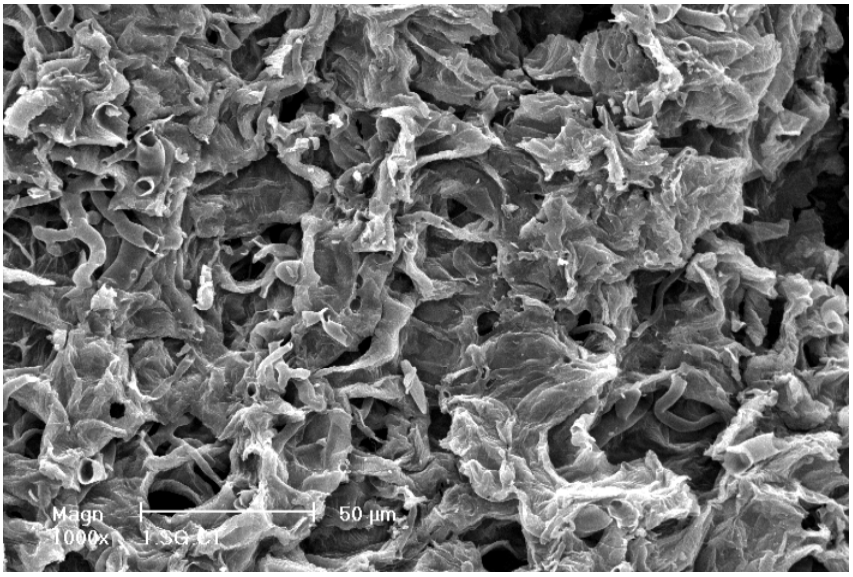
III.1Characterization of the activated carbon produced

In comparing the scanning electron microscopy (SEM) images of the *in nature* material and tung husk carbon prepared with and without $ZnCl_2$ treatment, it was observed that all of the samples had an irregular surface with heterogeneous, disordered grooves throughout (Figures 4a and 4b).

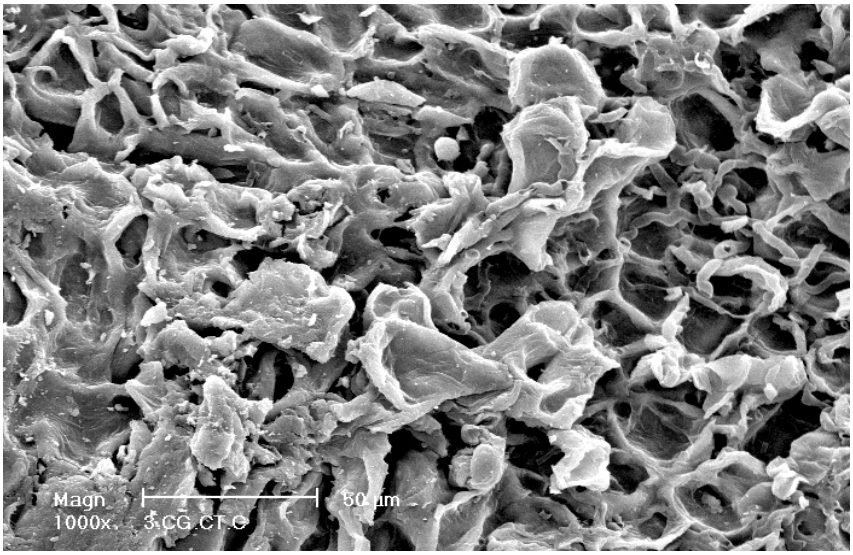
It is noteworthy that the husk carbon that was chemically treated with $ZnCl_2$ contained tubular and apparently concave structures that were not found in the *in nature* material. These structures would provide a greater sedimentation area to the processed husk material, explaining its high rate of dye adsorption. The carbon also appears to have smaller pores and in larger quantities than the *in nature* material which implies a greater surface area available for the adsorption process. The SEM images revealed little physical difference between the $ZnCl_2$-treated carbon that was carbonized with or without an inert atmosphere present during the process. Thus, the results described for the similar MB dye adsorption of the activated carbon produced under these two conditions could be confirmed.



(A)



(B)



(C)

Figure 1. A)Micrograph of in nature tung husk; B) Micrograph of tung husk carbon chemically treated with $ZnCl_2$ and C) Micrograph of tung husk carbon chemically treated with $ZnCl_2 – N_2$.

The surface areas of *in nature* tung husk and $ZnCl_2$-treated tung husk carbon were characterized. The sample of *in nature* tung husk displayed a surface area of 340.32 $m^2 g^{-1}$. In contrast, the $ZnCl_2$-treated (optimized condition) tung husk carbon exhibited a surface area of 2,201.57 $m^2 g^{-1}$.

A significant increase in the surface area of the material following chemical treatment and carbonization was noted. The carbon surface area that was demonstrated by BET analysis was high compared with those of activated carbons described in the literature, which typically exhibited values between 1,000 and 2,000 $m^2 g^{-1}$. Hameed, *et al.* [28] evaluated the activated carbon prepared through pyrolysis of bamboo and chemical treatment with KOH, and observed a surface area of 1896 $m^2 g^{-1}$ under the optimal assessment conditions. PELEKANI and Pelekani, *et al.* [29] produced activated carbon from a phenolic resin with a surface area of 1,918 $m^2 g^{-1}$ in its best condition. In addition, Hayashi, et al. [14] showed that lignin-activated carbon produced by chemical treatment with K_2CO_3 and carbonization at 800 °C possessed a surface of 2,000 $m^2 g^{-1}$.

III.2 Adsorption of Methylene blue

The tung husk mass recovery was approximately 40 % in the carbonization process. That mass loss resulted from the volatilization or carbonization of organic compounds and the production of gases, which are released due to the high temperature and unclog the pores, producing an adsorbent material. The results regarding the analysis of the MB dye adsorption by the produced adsorbent materials are organized with respect to the effect of precursor materials' particle size and the mass of the adsorbent material, the adsorbate mass and the surface characterization of the absorbent products.

III.3 Particle size of the precursor material

Figure 2 provides the comparative results of adsorption by activated carbon produced from precursor material of different particle sizes and given different treatments. Activated carbon produced from precursor materials of different particle sizes had different adsorption capacities. The adsorption of MB dye by carbon from tung husk that was previously treated with $ZnCl_2$ was more efficient than that of untreated carbon and *in nature* tung husk. The presence of N_2 throughout the carbonization process did not lead to a significant increase ($p < 0.5$) in the adsorption capacity of the samples. The statistical analysis demonstrated that the carbon product of the smallest particle size used (<0.5 mm) was significantly different in its MB-adsorptive efficiency relative to those produced from materials of greater particle sizes. Similar findings have been reported in a study characterizing activated carbon prepared from yerba mate material of three different particle sizes. The carbon produced from the finest particles exhibited a higher maximal adsorption capacity for MB dye and the herbicide atrazine, compared to those produced with particles of the other two sizes used. The study noted that the larger specific surface area of the carbon produced with smaller sized particles increased its adsorption capacity [30].

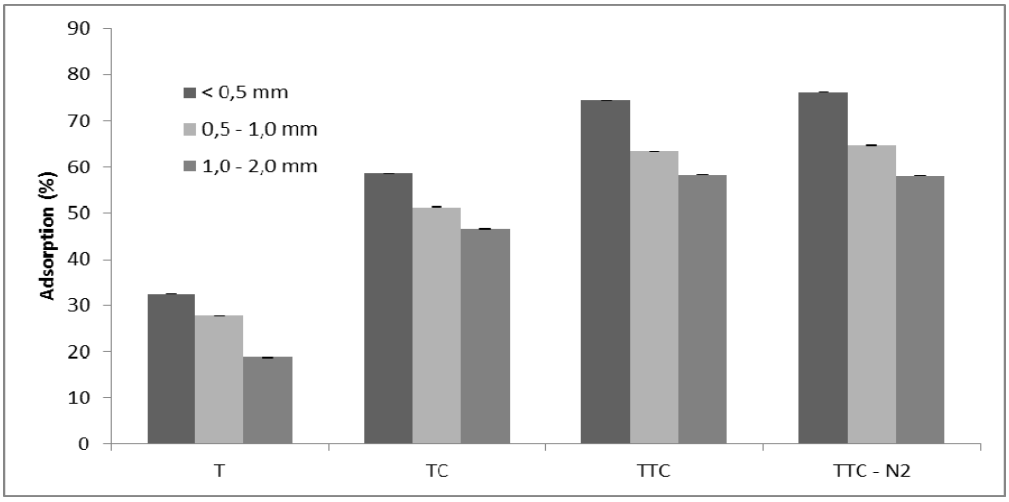


Figure 2. Comparison between adsorption of methylene blue by carbon produced from particles of three different sizes.

Besinella JR, *et al.* [31] also evaluated the effect of the size of the particles of coconut husk used to produce activated carbon on the adsorption of Remazol Golden Yellow RNL reactive dye and found a higher adsorptive capacity for smaller-sized samples, explaining this fact by the increase in area of particles that are in contact with the fluid phase.

III.4 Effect of adsorbent dosage

The effect of the activated carbon mass on the adsorption process is shown in Figure 3. The tests performed and statistically interpreted demonstrated a significant difference in the amounts of dye adsorbed per gram of the adsorbent material. Thus, the same amount of dye per gram was adsorbed when double the original mass was used. An equilibrium between the liquid and solid phases of the compound was reached with a given carbon mass; that is, the removal of a compound did not increase when the carbon dosage was increased.

Dye removal from textile wastewaters through an adsorption process, a given initial concentration of the solute, increasing the amount of adsorbent material, resulted in a greater surface area available for adsorption and, consequently, a greater adsorption rate. However, the amount of adsorbate removed per amount of adsorbent decreased, that is, the adsorption capacity of the activated carbon decreased. Weng, *et al.* [32] explained this fact as due to the decreased efficacy of dye diffusion resulting from the increased competition for a sedimentation site on the carbon surface. Similar findings were noted in a study of dye removal by adsorption to carbon produced from cyclodextrin [33].

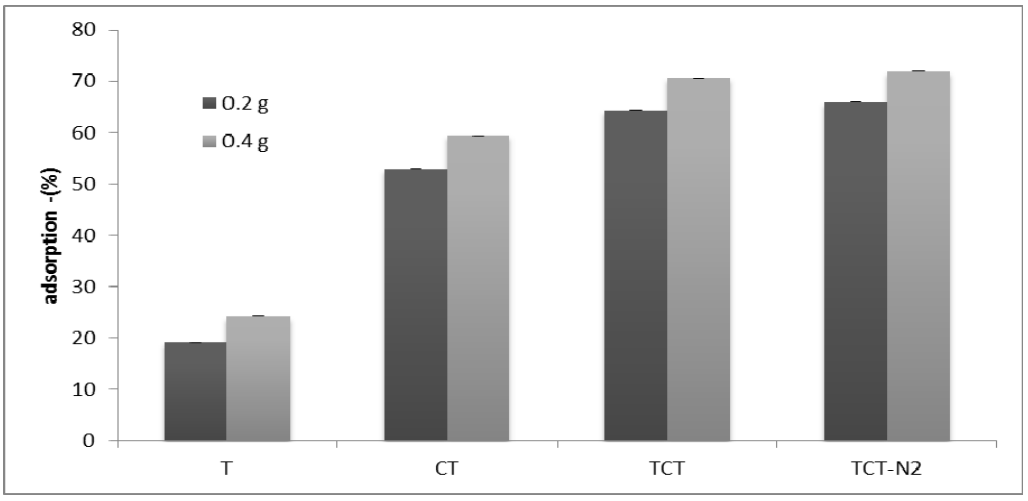


Figure 3. Amount of MB dye adsorbed by the activated carbon produced from tung husks.

Tung husk carbon previously treated with $ZnCl_2$ was more efficient than the other samples. The inert atmosphere of the carbonization process did not lead to a significant difference in MB dye removal, similar to the results of the previous assay. Furthermore, when analyzing the results for the ground-only tung husks (T) compared to the results for this husk's activated carbons (CT, TCT and TCT-N2), the production of activated carbon clearly improved the dye adsorption performance of the tung husk material, demonstrating the need for carbonization of this precursor material.

III.5 Adsorbate concentration

The present study was performed to assess the effect of adsorbate concentration (MB dye) on the adsorbent material produced. The most efficient material according to results discussed in the previous items was selected. Thus, the tung husk carbon of smallest particle size previously treated with $ZnCl_2$ was used in this assay. The results obtained in MB dye adsorption with a carbon mass of 0.03 g can found in Table 1.

Under these conditions, an ANOVA of two factors was used for statistical analysis. This analysis demonstrated in significant differences between the absorbed concentrations in the initial concentrations and detected no significant differences between the two treatments.

There was a high level of removal of MB dye from the solution at 24 h. Similar results regarding the quantity of adsorbed material were found when changing the amount of carbon used, however in this work does not occur the carbon saturation because the maximum relation between MB dye and carbon was 10 mg g^{-1} while there are reports with majors relations 300 mg g^{-1} or more [34].

Table 1. Results of MB dye adsorption at different initial concentrations, from 0.03 g carbon TCT and 24 h adsorption time.

Dye concentration (mg L^{-1})	**qe (mg g^{-1})**	**Removal efficacy (%)**
2	1.787	89.35
5	4.780	95.61
10	9.777	97.77
15	14.653	97.68
20	19.564	97.82

Another aspect worth noting is that even increasing the concentration of adsorbate, the carbon adsorbed nearly all dye from the solution, as previously described by Weng, et al. [32] in a study on the adsorption potential of activated carbon produced from pineapple leaf. Deng, *et al.* [35] explained that diffusion of dye molecules up to the adsorbent material's surface is affected by solution concentration. Increasing the solution concentration accelerates that diffusion as a result of the increase in driving force of the concentration gradient. In similar conditions, with MB 10 mg L^{-1} for 60 min the adsorption was 84 % and with carbon TCT-N2 it was not better than this. Therefore, it was considered that the carbon produced in inert atmosphere is more expensive and it was not worthwhile to improve its use.

IV. CONCLUSIONS

The present study showed that the best conditions for producing adsorbent material from tung husks was that of carbonization and pretreatment with $ZnCl_2$. The MB dye removal capacity of this product ranged from 75% to 83% during an adsorption time of between 10 and 60 min, and the removal capacity was greater than 97% after 24 h. The particle size of the precursor material was also analyzed and the carbon obtained from the precursor material with the smallest particle size studied (<0.5 mm) demonstrated the most efficient dye removal. Increasing the mass of the adsorbent did not significantly change the adsorption of MB dye from the solution. The surface area analysis showed an increase from 340.32 to 2,201.57 m^2 g^{-1} following chemical treatment and carbonization of the tung husks. Thus, these results demonstrate that the tung husks discarded in the initial stage of fruit processing for tung oil production can be used to manufacture activated carbon. The application of this product would contribute to the sustainability of the biodiesel production chain.

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