



PROCESS FOR CHROMIUM (VI) REMOVAL FROM MECHANIZED TANNERY WASTEWATER USING ACTIVATED CARBON OBTAINED FROM TANNERY SOLID WASTES

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ABSTRACT

This study offers an integrated process for treatment and recycling of tannery waste, it has a dual purpose. First the activated carbon has been prepared from leather shaving and buffing dust by physical activation. Both the raw material (leather waste) and the prepared activated carbon were analyzed by DTG, Thermo Gravimetric Analysis TGA, and scanning electron microscope. The adsorption tests of methylene blue and iodine onto the raw material and the prepared adsorbent were carried out and it was found that the adsorption capacity of the activated carbon was enhanced by the physical activation. The Activated Carbon was then characterized by equation of Brunauer-Emmett-Teller surface area and Fourier transforms infrared spectroscopy (FTIR). The Brunauer-Emmett-Teller surface area was found to be 491.05 and 242.60 m²/g for activated carbons prepared from Leather Savings and Buffing Dust respectively and the functional groups on the adsorbent surface were mainly CN, NH, OH, CO and CS. Secondly, the performance of the prepared activated carbon was assessed by adsorption of chromium (VI) from a synthetic solution, and then the chromium (III) present in the tanning effluent. The results revealed a decrease of chromium by 76% and 73% for the activated carbons prepared from Leather Savings and Buffing Dust, respectively.

Keywords: Leather Shaving, Buffing dust, Tannery effluent, Activated Carbon, Adsorption and Chrome ion.

INTRODUCTION

Tanneries industries generate a lot of waste that has negative impact on the environment and human health. The threat is the dumping of its solid and liquid waste that contains dangerous leftovers such as proteinaceous colloids, fats, tannins, flesh and hair, as well as toxic elements such as sulfides and chromium. All these "tailings" are a source of pollution and wastage that the leather industry cannot afford specially during this crisis period that lasted for several years, and is going from bad to worse. Note that 7 million tons of solid wastes per year are released by the global tanning industry (Booth, 2016). According to some estimation, approximately 1.8 million tons of chromium is generated by tanning industry annually worldwide (Sreeram et al, 2014). For the city of Kano (Nigeria) pollution by the tannery industry due to the release of heavy metals in the environment, mainly chromium is estimated at 70 tons of chromium generated per year, which can easily result in disrupting the biological degradation of effluent (Sbihi et al, 2012). When chromium Cr(III) is released in the nature, it is oxidized to hexavalent chromium Cr(VI) form which is known to be both acutely and chronically toxic to human, even when present in low concentrations. This form

of chromium Cr(VI) has been considered as one of the sixteen most toxic pollutants. It is therefore important to remove both Cr(III) and Cr(VI) from the contaminated water before it is discharged.

In recent years adsorption process has been widely used for the treatment of the tanneries wastes. For instance chromium-collagen wastes were used to remove tannins from wastewater (Sreeram et al, 2014) the diatom *Planothidium lanceolatum* (Brebisson) Lange-Bertalot (*P. lanceolatum*) was used for biosorption of hexavalent chromium Cr(VI) from aqueous solutions (Sbihi et al, 2012), powder of potato peelings (Mutongo et al, 2014) and an activated carbon prepared from coconut tree sawdust (Farenzena et al, 2015) were also used as adsorbents for removal of chromium Cr(VI) from aqueous solution, while leather wastes was employed as adsorbent to remove dye from solution (da Gama et al, 2011). In this work, the solid waste from the tanneries is transformed into activated carbon in order to enhance its adsorbent capacity for the Cr(VI) ions removal. Activated carbon is a carbonaceous material with highly developed internal surface area and porosity; it is widely used in industries for air and water treatment, in medicine as anti-toxin and in research as a

catalyst and support to catalysts (Fiessinger et al, 1985). The use of activated carbon to remove chromium from tanneries wastewater is an effective method for removing chromium ions from aqueous solution before disposal (Abdoul et al, 2017).

MATERIALS AND METHOD

Experimental Procedure of Manufacturing Activated Carbon

10 g sample of raw material (leather wastes) is placed in a sample holder that is put in the oven. It is brought under a nitrogen flow of 100 mL/min at a heating rate of 15°C/min to a temperature of 500°C which is maintained for 1 hour as residence time. After carbonization, the activation process is carried out under a nitrogen flow of 100 mL/min, with a heating rate of 10°C/min to reach the desired activation temperature, and then the sample is placed under steam flow rate of 0.1 mL/min, which was maintained for the desired residence time. Activated carbon is recovered, washed with distilled water for 12 hours to remove the ash, then dried, weighed, ground and kept for adsorption tests. The total yield of sample is calculated by the following equation

$$\text{yield} = \left(\frac{m}{M} \right) \times 100 \dots \dots \dots (1)$$

Where **M** (g) is the mass of the leather wastes, **m** (g) is the mass of obtained Activated Carbon (Abdoul et al, 2017).

Characterization Method

DTG and Thermo Gravimetric Analysis were performed on the raw material with a temperature between 25°C and 850°C. High resolution images of the raw material were obtained by the scanning electron microscope.

The textural characteristics of the selected samples of activated carbon prepared were obtained using adsorption/desorption of nitrogen at 77 K. The surface area and pores sizes were calculated by the equation of Brunauer-Emmett-Teller. The surface morphology of the selected active carbons was obtained using the scanning electron microscope. The functional groups of the selected active carbons were analyzed by Fourier transforms infrared spectroscopy (FTIR) (Abdoul et al, 2017).

Adsorption Experiment

Iodine and Methylene Blue

The ability of the activated carbon prepared to remove iodine and methylene blue from a synthetic aqueous solution was determined. Standard iodine solution was added over activated carbon (10 mg) and after an equilibration time of 4 hours, the residual iodine concentration was determined by titration with standard sodium thiosulfate using starch as an indicator. The same quantity of carbon samples were mixed with methylene blue in 100 mL stoppered Erlenmeyer flasks. Batch adsorption experiments were carried out in the shaker (400 rpm) at room temperature for 4 hours. The solutions were filtered and the concentration of methylene blue was determined by UV spectrophotometer (660 nm). The activated carbon with

improved adsorption capacity of methylene blue and iodine was then chosen in order to finally test for adsorption with respect to chromium (VI) and chromium (III) (Abdoul et al, 2017).

Adsorption of Chromium (VI) and Tannery Effluent (Cr (III))

Batch adsorption studies for the removal of chromium (VI) by activated carbon prepared from Leather Shaving and Buffing Dust were conducted to determine pH effect and were performed by mixing 0.01 g of activated carbon with 100 mL of synthetic solution of chromium (VI) in 250 mL bottles that were left under stirring for 4 hours at 30°C.

For chromium (VI), at the end of stirring the filtrates were analyzed by reaction with 1, 5-diphenylcarbazide monitored by measuring absorbance at 540 nm using a UV-visible spectrophotometer.

For the chromium (III) tanning raw effluent samples, a treatment was specifically performed to separate out particles present in liquid phase by filtering on filter paper. After the separation process the aqueous solutions were analyzed to determine the concentration of chromium and were then subjected to adsorption in the same conditions as those applied for the synthetic solution of chromium. All the adsorption experiments were conducted at 18°C and the samples shaken at the rate of 400 rpm. For all the tests the adsorption capacity is calculated by the following equation:

$$qe = \frac{(C1 - C2)v}{w} \dots \dots \dots (2)$$

Where **C₁** (mg/L) is the initial concentration of adsorbate Methylene blue, Iodine and Chromium Cr(VI)), **C₂** (mg/L) is equilibrium residual concentration of Methylene blue, Iodine and Chromium in solution, **v** is volume of solution (L), and **w** (g) is mass of the selected activated carbon (Abdoul et al, 2017).

Results and Discussion

Raw Material (Leather Wastes) DTG and TGA Analyses

TGA analysis indicated thermal events associated with the mass change between 25-850°C, figure 2a. For Leather Shaving it shows a first variation between 40.5-175°C which corresponds to a decrease in mass of 7.6%, due to evaporation of water. The weight loss was more significant from temperature 175.5-631°C; this is due to removal of volatile organic substances and moisture, with a weight loss of 52.1%. Beyond 631°C the mass varies slightly depending on the temperature. For DTG analysis, figure 2b, which is a thermal analysis technique to measure the endothermic and exothermic transitions as a function of the temperature, the analysis shows a peak between 38 to 318.7°C which corresponds to a first departure of water according to an endothermic reaction ($\Delta H = 693.5$ J/g), then the start of release of volatile organic materials according to an endothermic reaction ($\Delta H = 617$ J/g). For

Buffing Dust, the TGA curve (figure 1A) shows that the bristles have two events characteristic of mass loss, due to the thermal behavior of the material. The first event is observed between 42.5-183°C, which is due to the release of water and a mass loss due to the degradation of hair microfibrils with a loss of 9.3%. The second loss observed between 185-571.98°C is due to the degradation of hair, which leads to a carbon chain of keratin with a loss of 55.5%. Complete degradation of the

keratin and hair structure happens at 700°C (Farenzena et al, 2005) and (da Gama, et al, 2011). For DTG analysis, figure 1B, the first degradation is related to the loss of water as well as organic matter, it takes place according to an endothermic reaction ($\Delta H = 816.214 \text{ J/g}$) followed by another endothermic reaction ($\Delta H = 330.411 \text{ J/g}$) due to the degradation of hair microfibrils.

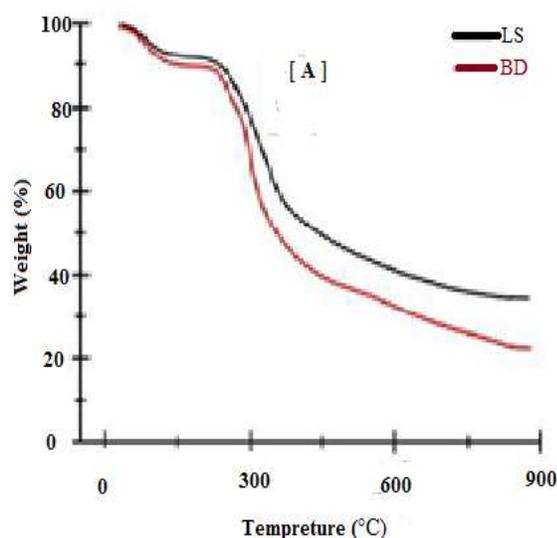


Figure: 1A

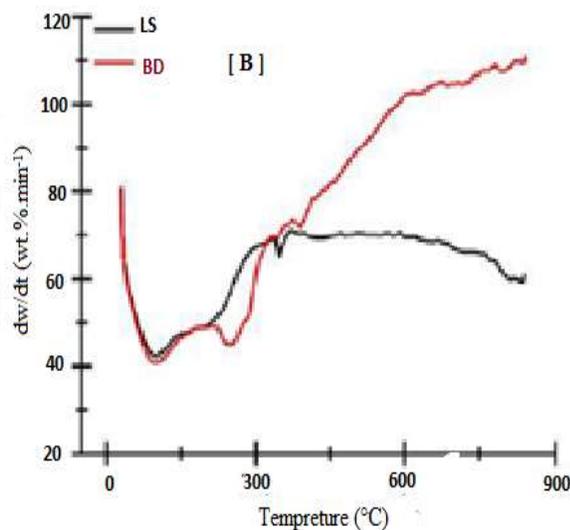


Figure: 1B

Figure 1: Thermo Gravimetric Analysis TGA/DTG curves of an untreated leather shaving and Buffing Dust, [A] is TGA and [B] is DTA of both raw materials before transformation into activated carbon.

Adsorption Raw Materials

The adsorption capacities of the raw materials towards iodine and Methyl Blue are shown in table 2. The results show that adsorption occurred at the surface of the both raw materials possibly on the functional group available at the surface of Leather Shaving and Buffing Dust, for example -CO-, -CO-NH-, and -NH₂. This can explain the low values of Methyl Blue and iodine found for Leather Shaving and Buffing Dust. The transformation of these raw materials into Activated Carbon is expected to enhance the adsorption capacity by creating porous sites and more functional groups at the surface.

Preparation and Characterization of Activated Carbon

Yield Analysis of Activated Carbon

Tables 1 and 2 present the conditions (final temperature, residence time, and yield) of preparation Activated Carbons. Analysis of the Activated Carbon from Leather Shaving (table 1) shows that the increase in temperature from 750°C to 850°C

with a fixed residence time is accompanied by a decrease in the final yield. Leaving the temperature fixed, the yield decreases with increasing residence time (activation) from 2 - 4 hours. It can be concluded that the increase in the temperature and the residence time leads to a decrease in the final yield, due to the intensification of gasification reactions and therefore the loss in weight of the obtained carbon. It is known that the lower the yield, the more micro porous is the adsorbent and the higher the specific surface area. The analysis of Buffing Dust yields (table 2) shows that the increase of residence time (activation) from 2-4 hours led to a decrease in the final yield; leaving the residence time (activation) fixed, increasing of temperature from 750-800°C does not lead to a large change in performance. This leads to the conclusion that with these conditions of Activated Carbon preparation from Buffing Dust, the performance is mainly controlled by the residence time (activation) caused by the reactions of carbon with the oxidizing medium.

Table 1: Preparation parameters and adsorption capacity of Methylene Blue and Iodine for activated carbon from Leather Shaving

Samples	Final temperature (°C)	Residence time (min)	Final yield	QADSMB (mg/g)	QADS I ₂ (mg/g)
LS	-	-	-	110.4	586.9
AC1	750	60	15.22	387.7	576.53
AC2	750	120	11.35	583.4	610.28
AC3	850	60	10.14	450.2	706.89
AC4	850	120	4.7	505.35	190.36

Adsorption Tests for Methyl Blue and Iodine**(i) Tests for Methyl Blue**

The results obtained for Activated Carbon from Leather Shaving are summarized in table 1. It can be observed that the adsorbed amount increases generally with increase of residence time of activation, and also increases with increasing temperature. According to (Fiessinger et al., 1985), (Wiesner et al, 1987) and (Wiesner et al, 2012), adsorption of organic compound mainly concern mesopores and macro pores, then the enhancement of residence time and temperature increases the pore diameter. It can be noticed that the amount of Methyl Blue adsorbed increases significantly when the raw materials are transformed into Activated Carbon.

Table: 2 Parameters and preparations of adsorption capacities for Methylene Blue and Iodine of activated carbons from Buffing Dust.

Samples	Final temperature (°C)	Residence time (min)	Final yield	QADSMB (mg/g)	QADS I ₂ (mg/g)
BD	-	-	-	218.1	80.7
AC1	750	60	17.16	278.1	613.27
AC2	750	90	12.3	385	694.8
AC3	800	60	17.85	345	645.31
AC4	800	90	11.84	392.95	190.4

(ii) Tests for Iodine

The influence of activation temperature and the activation residence time are also examined for adsorption capacity of iodine. Results are shown in tables 1 and 2. It is clear that uptake of iodine in aqueous solution were improved during the transformation process of raw materials into Activated Carbons. However, in these tables, we observe for all the samples that the amount of iodine adsorbed increases significantly with increase of temperature and activation residence time during preparation, except for Activated Carbons obtained for temperature up to 800°C and the residence time equal to 4 hours, AC4 and AC8 for which the iodine numbers are about 190mg/g. For the sample prepared by the residence time less than or equal to 90 minutes the adsorption capacity is higher, meaning that Activated Carbons

prepared contained an important amount of micropores. This can be attributed both to the structure of raw material and to the use of steam within the activation process; steam was observed to generate a narrow but more extensive microporosity.

The adsorption of Methyl Blue and Iodine shows that the transformation of raw materials into Activated Carbons using steam generates micropores and mesopores, existing mesopores serving as gateway to micropores. For samples AC4 and AC8, increasing temperature and residence time widens micropores to give mesopores or macropores. Sample AC3 and AC6 show highest values of iodine; this allows us to calculate their surface area and pore volume using Brunauer-Emmett-Teller analysis,

Table: 3 Brunauer-Emmett-Teller analysis performed on activated carbons obtained from leather shaving after physical activation at 850°C during 4 hours AC3 and activated carbon obtained from Buffing dust after physical activation at 750°C during 60 minutes AC6.

Samples	Surface areas (m ² /g)	Total pore volume (cm ³ /g)
AC3	491.05	0.421
AC6	242.60	0.138

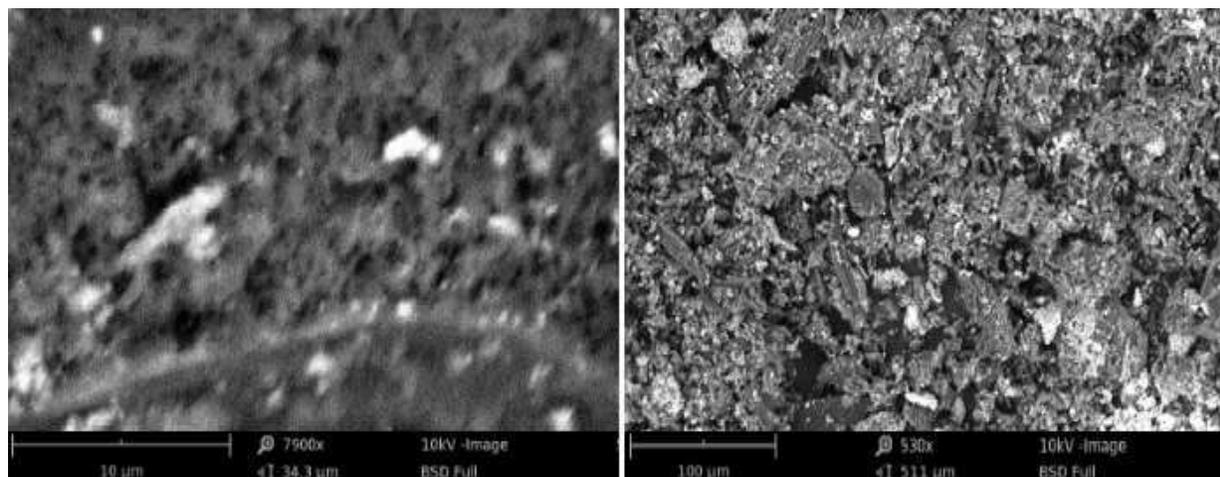
Analysis of AC3 and AC6

Scanning Electron Microscope (SEM), Brunauer-Emmett-Teller and IR Analyses

Typical SEM micrographs of the surfaces of AC3 and AC6 are illustrated in figure 2: The surface morphology of the carbon atoms shows that the carbon atoms of the surfaces are heterogeneous with an irregular pore structure. Pores of different sizes and forms have been observed in these images.

The IR spectra of activated carbons obtained from leather shaving and Buffing dust are superimposed and represented in figure 4: The IR spectra for Activated Carbon from leather

shaving as well as from Buffing dust reveal the presence of characteristic band at 2420,343 cm⁻¹, which corresponds to NH or OH (Yi-min et al, 2012), (Brateka et al, 2013), (Gonzalez et al, 2006) and (Ferro et al, 2011), while the bands located at 1460 cm⁻¹ and 1100 cm⁻¹ are attributed to C=C vibration and CO respectively, in relation to 2350 cm⁻¹ which corresponds to CN nitrile. The spectrum of Activated Carbon from Buffing dust, there are also peaks at 594-870 cm⁻¹ corresponding to CS and aromatic CH respectively (Ferro et al, 2011), (Carpenter et al, 2013), (El Nemr, 2011) and (Kanagaraj et al, 2006).



SEM; 1 micrographs of the surfaces of AC3 SL.

SEM; 2 micrographs of the surfaces of AC6 BD

Figure 2: The SEM micrographs of activated carbon, (A) activated carbons obtained from Leather Shaving after physical activation at 850°C during 4 hours AC3 and (B) activated carbon obtained from Buffing Dust after physical activation at 750°C 4 hours AC6.

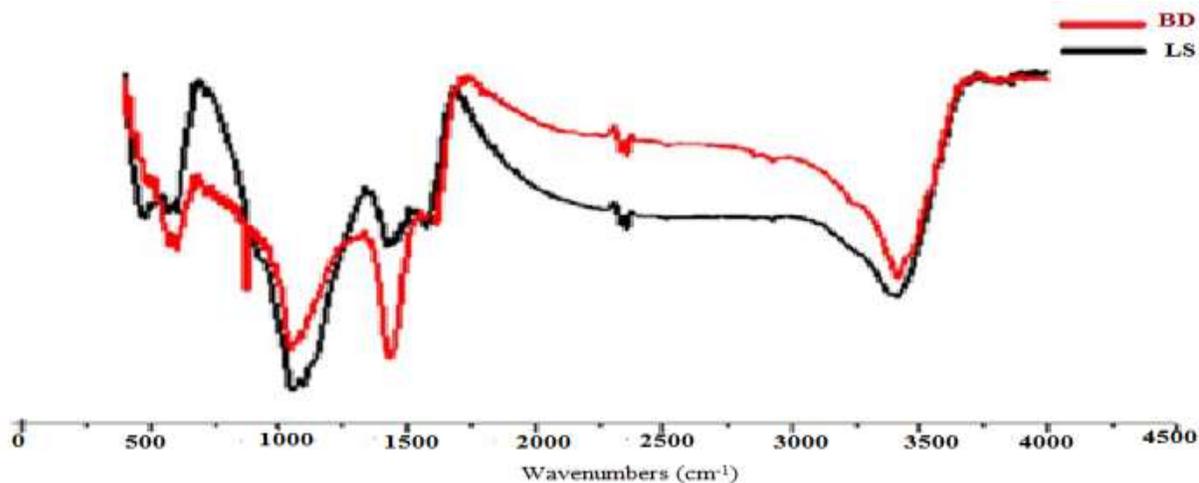


Figure 3: The FT-IR spectrum of activated carbons obtained from Leather Shaving after physical activation at 850°C during 4 hours AC3 and activated carbon obtained from Buffing Dust after physical activation at 750°C during 4 hours AC6 before Cr(VI) sorption

Adsorption Cr(VI)

The curves given in figure 3 represent the change in the adsorbed amount (mg/g) as a function of pH. It can be noticed that the adsorbed amount increases with pH until a maximum value for the pH = 3 (57.71 mg/g for the activated carbons from Leather Shaving and 50.8 mg/g for the activated carbons from the Buffing Dust) and then declines at pH = 5, because the degree of protonation of the surface is reduced gradually, leading to the reduction of adsorption. In acidic medium, the surface of the adsorbent is highly protonated and promotes the adsorption of Cr(VI) in its anionic form. When the pH = 3 we find that the adsorption of the predominant form $\text{Cr}_2\text{O}_7^{2-}$ is important. This suggests that the adsorption cannot be by

electrostatic forces (physisorption) but rather by chemical forces via an electron exchange between the activated carbon and $\text{Cr}_2\text{O}_7^{2-}$ ion (Kantarli et al, 2009), (Kantarli et al, 2010), (Vengosh et al, 2018) and (RAO et al, 2002). This leads according to a reduction of chromium (VI) to chromium (III) by the catalytic action of carbon. Below pH = 3, we noticed a decrease in the amount adsorbed to a minimum value at pH = 1; the adsorption capacity was decreased by about 8 times, with the decrease of pH from 3-1. This decrease was attributed to the various complexes that Cr(VI) can form in aqueous solution. Under the acidic conditions (pH < 1.6), chromium is transformed in its neutral form H_2CrO_4 which decreases its adsorption capacity on the activated carbons.

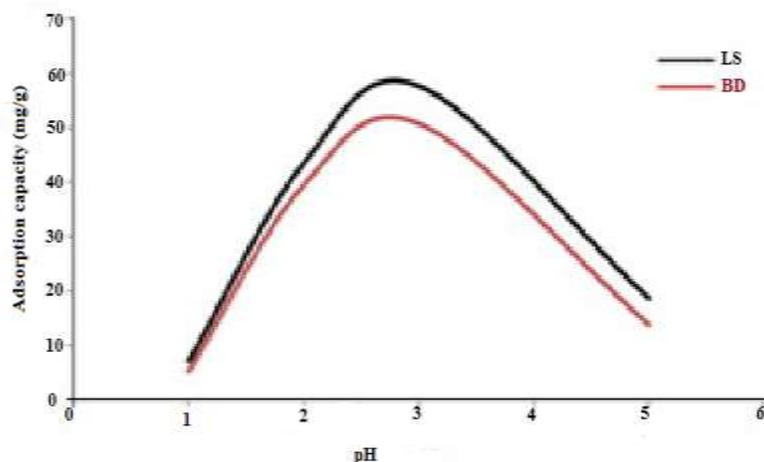


Figure 4: Effect of pH for the adsorption of Cr(VI) onto activated carbon prepared from Leather Shaving and Buffing Dust. Conditions: during 4 hours, 0.01 g/250 mL, at 30°C.

Adsorption of Cr(III) from the Tannery Effluent

The achievement of the adsorption tests on the solution of tannery effluent after treatment and oxidation of chromium (III) to chromium (VI), allowed us to find a concentration of 3.215 g/l with the spectrophotometer. This tannery effluent is discharged into the waste water after each chromium tanning operation. In order to reduce the concentration of chromium in the effluent from tanneries, we realized the Procedure shown in figure 4. After treatment we got the adsorbed quantity and elimination rates. In the prevailing condition pH = 6, chromium is present in Cr(OH)₂ form (Sreeram et al, 2003). The latter is adsorbed on the activated carbon from Leather Shaving which leads to a removal rate of 76%, and for Activated Carbon from Buffing Dust to a removal rate of 73%.

CONCLUSION

The adsorption tests showed that activated carbons prepared from the tannery wastes, are good adsorbents for methylene blue, iodine and Cr(VI). The procedure that we adapted for the removal of chromium (III) in the tannery effluent with the prepared activated carbon, has allowed us to remove 76% of the concentration of chromium (III) in the tannery effluent, this with the activated carbon based on the shaving leather, and 73% with the activated carbon based on the buffing dust, with a surface that exposes various functional groups responsible for the adsorption. Thus these tannery wastes can be valued by decreasing their accumulations in nature as pollutants, and also by transforming them into activated carbons in order to remove the pollutants or recover the metals accumulated in the wastewaters.

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