USING OF DATE PALM LEAF MIDRIB BIOCHAR AS AN ADSORBANT IN WATER TREATMENT

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ABSTRACT

Date palm leaf mid-rib (DPLM) biochar has been examined as an adsorbent in comparison with another two adsorbents (Corn stovers biochar and local commercial coal) to test for its physical & chemical adsorption properties. DPLM and Corn Stover biochars have been prepared in a muffle by carbonization at 450 ^{0}C for 2 hrs, then activation by sulphuric acid 10% at 40-50 ^{0}C, then washing well with distilled water until no excess sulphate ions are longer present. Different adsorption experiments have been done to the adsorbents complete with the commercial coal. Methylene blue dye was used for the adsorption experiments. The results shows that the DPLM activated biochar at alkaline pH gave the maximum adsorption (99.7%). The physical characterization experiments done to the DPLM revealed that it has a large surface area, and very large pore volume.

Key words: Biochar, Dyes, Methylene blue, adsorption, Date palm Leaf Mid rib.

INTRODUCTION

Biochar is a carbon-rich solid material derived by heating biomass, under limited or no oxygen (pyrolysis or carbonization) conditions (Sohi, 2012), The production utilizes organic materials such as wood (Schimmelpfennig & Glaser, 2012; Liu *et al.,* 2010), animal manure (Park *et al*., 2011) and agricultural waste (Demirbas, 2004). Most of these starting materials are readily available and quite

often the choice of biomass will depend on the most abundant one in a given area. Biochars are used as environmentally benign adsorbents for removal of contaminants from water (Tan *et al*., 2015). This is attributed to their high surface area, presence of numerous functional groups, porous network and their mineral components which imbues the materials with high adsorptive properties. These properties make biochar a potentially low-cost and effective adsorbent and a suitable alternative to activated carbons which are widely applied in conventional wastewater treatment regimes worldwide. Concerning the capital costs involved, activated carbons require high preparation temperatures including activation steps whereas biochars require relatively lower temperatures hence reduced energy and infrastructural requirements besides being a renewable resource (Ahmad *et al.,* 2012; Lu *et al.,* 2012). Pyrolysis with (or without) activation converts organic wastes into material with beneficial properties which can serve as an adsorbent and a biofilm carrier for wastewater treatment. Agricultural wastes are better than other adsorbents because the agricultural wastes are usually used without (or with) a minimum of processing (washing, drying, grinding) and thus reduce production costs by using a cheap raw material and eliminating energy costs associated with thermal treatment (Franca *et al.*, 2009). Furthermore, biochar synthesis adds economic value to these waste materials and would potentially eliminate their negative environmental impacts. Many industries like the textile industry used dyes to color their products and thus produce wastewater containing organics with a strong color, where in the dyeing processes the percentage of dye lost wastewater is 50% of the dye because of the low levels of dye-fiber fixation (Mohan *et al.,* 2007). Discharge of these dyes into effluents affects the people

who may use these effluents for living purposes such as washing, bathing and drinking (Sharma, and Sobti, 2000). Also dyes may impart toxicity to aquatic life and may be mutagenic, carcinogenic and may cause severe damage to human beings, such as dysfunction of the kidneys, reproductive system, liver, brain and central nervous system (Kadirvelu *et al*., 2003; Dinçer *et al.,* 2007; Shen *et al.* 2009).

The removal of color from waste effluents becomes environmentally important because even a small quantity of dye in water can be toxic and highly visible (Chiou *et al.,* 2004). Since the removal of dyes from wastewater is considered an environmental challenge and government legislation requires textile wastewater to be treated, therefore there is a constant need to have an effective process that can efficiently remove these dyes (Lee and Choi 2006). Therefore adsorption by agricultural by-products used recently as an economical and realistic method for removal of different pollutants has proved to be an efficient at removing many types of pollutants such as heavy metals [Argun *et al.,* 2007, Aroua *et al.,* 2008], COD [El-Naas *et al.,* 2010, Ahmad and Hameed, 2009), and dyes [Weng *et al.,* 2009, El-Halwany 2010, Leechart *et al.,* 2009).

This study is aimed at investigating the possibilities of the use of some local readily available agricultural waste product in Egypt for the removal of cationic dye (Methylene blue) from aqueous solution. The system variables studied include sorbent dose, initial concentration of the dye and pH.

Experimental

Plants Residuals: To produce the biochar, we selected two types of residuals (agriculture wastes) that are abundant in the Egyptian environment (Corn Stover (CS)) and (Date Palm Leaf Mid rib (DP)). Corn stovers were brought from El-Giza rulers' & DPLM was brought from El Menia). In addition, another adsorbent was used which is the commercial coal. It was purchased from the local market.

Chemicals: Sulphuric acid used was obtained from DOP Organik Kimya – Ankara-Turkey. Methylene Blue dye was obtained from El-Nasr for chemical industries- Abo Zaable - Cairo- Egypt. Barium Chloride (used as indicator) was brought from Fisher Scientific UK.

Preparation of the adsorbents:

Corn Stovers Biochar (Bcs) & DPLM Biochar (Bdp) were developed from corn Stover CS & date palm-leaf midrib (DP) respectively by using slow pyrolysis method. The CS and DP, were cleaned, crushed carefully into smaller-size particles. These were put into canister to produce Corn Stover Biochar (Bcn) and DPLM Biochar (Bdp), respectively.

Canisters were made form iron steel, cylinder shape having a length of 35 cm and diameter of 7.5 cm, having two (3 mm) opening from the top to exit the exhaust of the pyrolysis. It was placed in an electric muffle furnace Type Thermolyne Furnatrol III Type 19300 controller, its heat range from 50^0C up to 1200 ^{0}C. The two biomasses biochar were produced by slow pyrolysis at 450 ^{0}C method, which was chosen from previous studies. (Gerçel *et al.*, 2007), (Taha *et al.*, 2014), (Joan J Manya 2012), (Mohan *et al.*, 2014) .It was started by raising the temperature up gradually to 100 ^{0}C for one hour for drying. After this period, the pyrolysis started by raising the temperature to 450 ^{0}C by the rate of (10^0C/

min), kept at the same temperature for 2 hours. Samples were cooled to room temperature; biochar was then collected and weighed again to determine the product / raw material percent. The resulted Biochar was then grinded using mortar and pestle, sieved using laboratory sieve with mesh size 250 – 500 µm. and then stored in suitable containers.

The charcoal (CHc) was brought from the Egyptian local market, grinded using mortar and pestle , sieved using laboratory sieve to the mesh size 250 – 500 µm. (Kavitha and Namasivayam, 2007).

Chemical activation of the biochar: Productions of treated biochars were done using a solution of 10% conc. H_2SO_4. Bcn, Bdp, CHc, were used to produce TBcn, TBdp and TCHc respectively. They were prepared by soaking 20 gram of each Biochar in 100 ml H_2SO_4 of (10%) solution for one hour at 40-50 ^{0}C. Then the samples were cooled down, separated from the sulphuric acid and washed several times with distilled water to remove any remaining sulphate ions. To ensure that all the sulphate ions have been removed away, $BaCl_2$ was used to test the filtrate solution, until there is no white precipitate formed in the washing solution. The treated products are then well-dried in the oven at 70 ^{0}C overnight, and transferred to glass dishes to be stored.

Characterization of the biochar: Different characterization techniques were used to study the chemical and morphology of the biochar. Infrared spectroscopy was carried out using Nicolet iS50 FT-IR spectrophotometer, to determine the main functional groups. Different adsorbents surface morphology was examined by scanning electron microscopy (SEM) to and was performed with SEM Model Quanta FEG250, USA with accelerating voltage 30 kV. The crystalline phase of

the biochar was detected using X-ray diffraction (XRD) Analysis on a Shimadzu XRD-6000 diffractometer with Cu Ka (λ= 1.54056 Å).

Adsorption experiments: Methylene blue solutions (50 ml of constant concentration of 0.0001 Molar) in which the bio-sorbent was suspended (0.25 g of biochar), were agitated at 150 rpm for overnight . Dye concentrations before and after adsorption were measured using a UV/Vis spectrophotometer. The adsorption parameters were optimized in terms of pH, contact time, and bio-sorbent dosage.

Removal efficiencies were calculated using equations 1.

$$RE = \frac{(C_0 - C_t)}{C_0} x\ 100 \qquad (1)$$

where c_0 is the initial concentration in mg/L, c_t dye concentration at time t

Optimization experiments have been made to test for the best adsorption conditions, the following are the experiments done to:

A- Effect of contact time to detect the best adsorption time to make the highest adsorption rate before turning to be stable, at 25 ^{0}C when 50 ml of 0.0001 M of the dye solution was agitated with 0.25 g of TBdp at pH 9.00 The flasks were shaken at 150 rpm overnight and the equilibrium concentration of the remaining dye was determined spectrophotometrically.

B- Effect of sorbent dose at constant initial concentrations equilibrium uptake of dye was investigated with sorbent masses of (0.05, 0.10, 0.25, 0.35, 0.50 gram) of TBdp, in contact with dye solutions of a constant concentration of 0.0001 Molar and pH 9.00. The flasks were shaken at 150 rpm and room temperature for 4 h and the equilibrium concentration of the remaining dye was determined spectrophotometrically.

C- Effect of initial dye concentration was investigated by adding 0.25 g of TBdp biochar in contact with volume of 50 ml dye solution of concentrations ranging from (0.0001,0.0005, 0.0010, 0.0015 and 0.0020) molar and at pH 9.00. The flasks were shaken at 150 rpm and room temperature for 4h and the equilibrium concentration of the remaining dye was determined spectrophotometrically.

RESULTS AND DISCUSSION

The Weight of each biomass was registered before and after the pyrolysis and it is shown in the Table 1.

Table 1: pyrolysis yield results

It.	Biomass	Weight before pyrolysis	Weight before pyrolysis	yield
1.	Corn Stover	1435 gram	488 gram	34 %
2.	Date Palm Midrib	1574 gram	490 gram	31 %

This is due evaporation of all the Humidity inside the plants residual, and all the volatile gases, e.g. VOCs and NOXs and cellulose and hemicellulose in the plant waste.

Physical characterization of the biochars: (for the treated biochars)

FTIR spectroscopy: The FTIR spectra of the three biochars are illustrated in Fig. 1. The intensity of a broad bond at the range of 3675 – 3000 cm^{-1} in the biochars decreased or almost disappeared in the biochar produced at 450°C. This bond represents the stretching vibration of hydroxyl groups and suggesting that organic OH groups are very unstable at elevated temperatures and also attributed to acceleration in dehydration reaction of biomass (Chen *et al.*,2012).

For corn cob (Stovers) biochar , the main functional groups and respective regions of absorption were identified: axial deformation of the O-H group (3459 cm^{-1}) associated with the functional groups of phenols, alcohols and carboxylic acids; aliphatic C-H axial deformation (2943 cm^{-1}); stretching of C=O (1743 cm^{-1}); C=C stretching of aromatic groups among other groups (1659 cm^{-1}); C-O stretch of polysaccharides (1044 cm^{-1}, whose intensity is related to the amount of cellulose) (Ibrahim *et al.* 2013, Park *et al.*, 2012, Sevilla *et al.*, 2011).

The decrease of the peaks in this region confirms the degradation of the hemicelluloses and cellulose from the original biomass after the pyrolysis. The peak around 1611 cm^{-1} in the charcoals corresponds to the vibration of the aromatic ring of the lignin indicating the presence of lignin even using a temperature of 400 °C in pyrolysis.

Along with the earlier observations, an elevating pyrolysis temperature from 350 to 500 °C generated a decrease in several characteristic bands, for example, at 3280 cm^{-1} (O-H stretching vibrations of hydrogen bonded hydroxyl groups), at 1700 cm^{-1}, C=O ((carboxyl C=O stretching mode) and at C-O-C (stretching for cellulose, hemicelluloses and lignin), while there are number of bands whose intensity increased, for instance, the 832 and 700 cm^{-1} bands (aromatic C-H deformation modes), the 1570-1600 cm^{-1} band (aromatic C=C stretching and C=O stretching modes of conjugated ketones and quinones), and the 1400 cm^{-1} band (C-C stretching vibrations in the aromatic ring) For the three biochars, the FT-IR spectrum exhibit a peak at 1577 cm^{-1} which could be assigned to C=C-C stretch characteristic of the aromatic ring stretch. An absorption band at 1700 cm^{-1} due to the stretching vibration by the carbonyl groups of the carboxyl groups and the peak at 1100 cm^{-1} is evidence of a C-O-C stretch associated with

lignin. These IR absorption bands have also been observed for other biochars based on other biomasses (Lee *et al.*, 2010, Smidt and Meissl 2007).

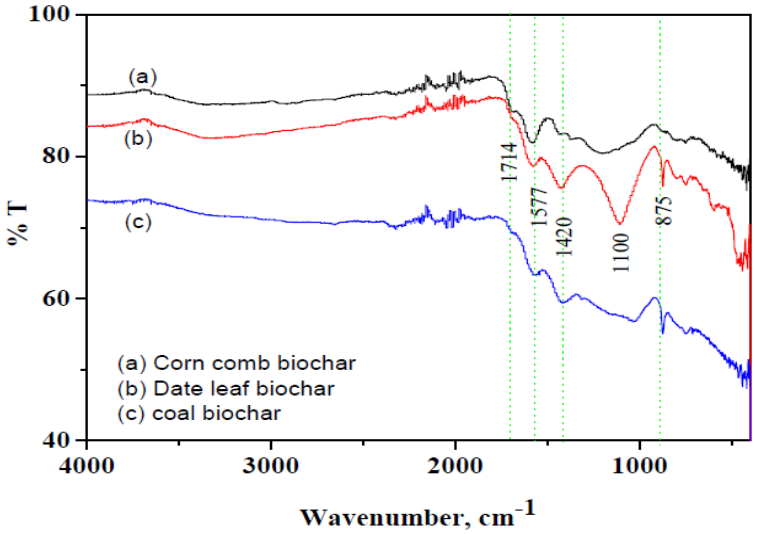

Fig 1: FT-IR Spectra of treated agriculture based biochar.

The inorganic sulphate ions absorbs strongly at 1125 - 1080 cm^{-1} due to asymmetric SO_4^{--} stretching. The symmetric stretch is normally forbidden symmetry but may occasionally be seen as a very weak sharp band at 1000 cm^{-1}. This statement agrees with the value of 1015.5 cm^{-1} (KHSO4) 989.3 cm^{-1} ($NaHSO_4$).

X –Ray Diffraction: X –Ray Diffraction have been also made to detect the shape of the activated biochars. XRD patterns of the produced biochar show two broad peaks (Figure 2) at 2θ of about 23° and 43° corresponding to the (002) and (100) plane reflection, and reveal the amorphous nature of as-prepared carbon materials. This can be explained by the rupture of multiple bonds C-C (the aromatic rings) and formations of group and functions on the surface. The crystallites are formed by two or more of these plates being stacked one above the other. Notable the absence of sharp peaks which indicate it is a is a largely amorphous structure (Kennedy, *et al.*, 2007).

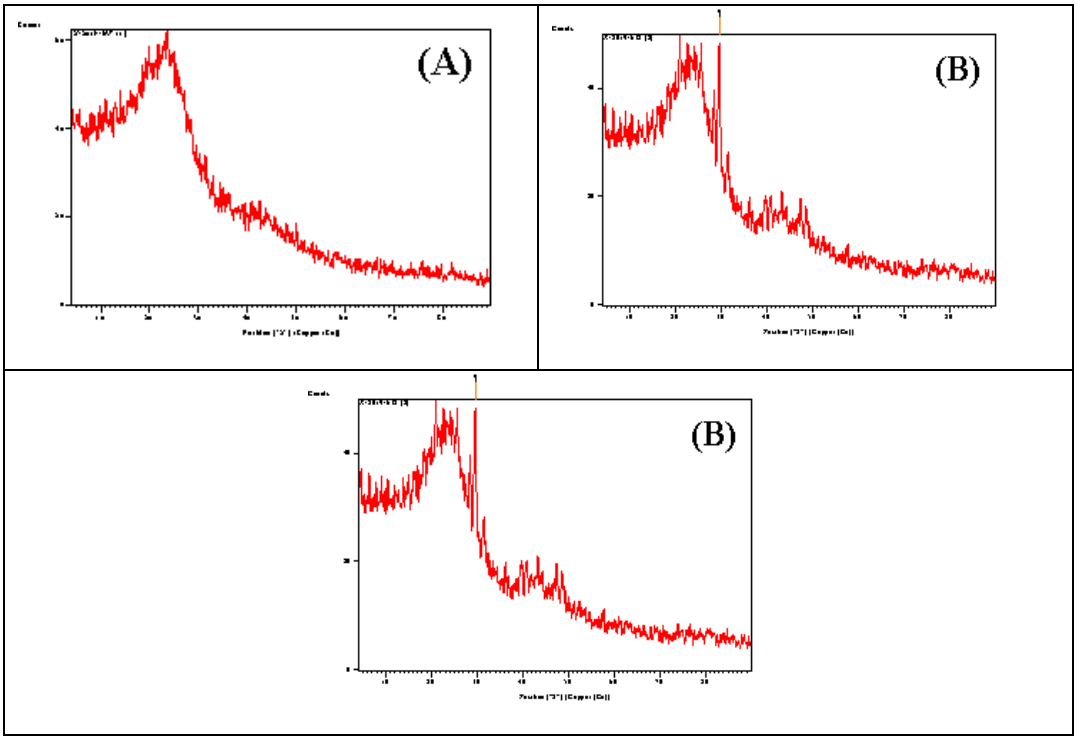

Figure 2: XRD of a) Corn b) Date c) Coal

Scan Electron Microscope: SEM photos were taken to different sections in the three activated adsorbents, and the results were as the following:

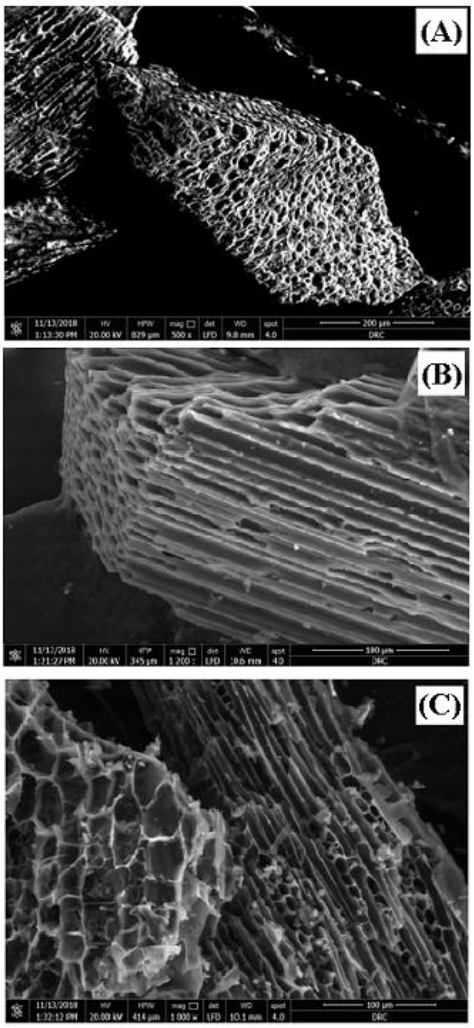

Fig 3: SEM Images of activated: *a) Corn biochar b) Date biochar and c) Coal*

SEM Images (Fig 3) revealed the presence of high porosity overall the adsorbent which means more efficiency in adsorbing different organics including industrial dyes.

Preliminary experiments to select the best adsorbent: The absorbents have tested to determine the best adsorbent according to its removal ability. In the same time, the adsorption experiments were performed at three different pH to determine which pH is the best for most high adsorbance of the tested dye. (Table 2)

Table 2: Removal % of the biochars at different pH

It.	**Adsorbent**	**Removal %**		
		pH=3	**pH = 6**	**pH = 10**
1	Untreated Corn	45.3	63.5	96.028
2	Treated Corn	48.7	65.3	98.062
3	Untreated (DPLM)	48.8	64.8	96.949
4	Treated (DPLM)	47.5	65.5	99.777
5	Untreated Coal	47.9	63.4	95.456
6	Treated Coal	49.1	65.9	98.507

From Table 2, it is clear that the highest absorbance was obtained by the Treated Date in the alkaline medium. The removal % was 99.77% .From this result, treated date in alkaline medium was selected to perform the optimization adsorption experiments.

Adsorption using Date Palm Leaf Midrib:

Effect of contact time: Contact time is an important factor in the sizing of an adsorption plant. The results for the effect of contact time on adsorption capacity are shown in Figure 4. From the diagram, it can be observed that a rise in the removal efficiency in the first 4 hours up to a maximum of 94.1%. No much

improvement in the removal efficiency was observed after this time as it reaches 96.6% after 24 hours.

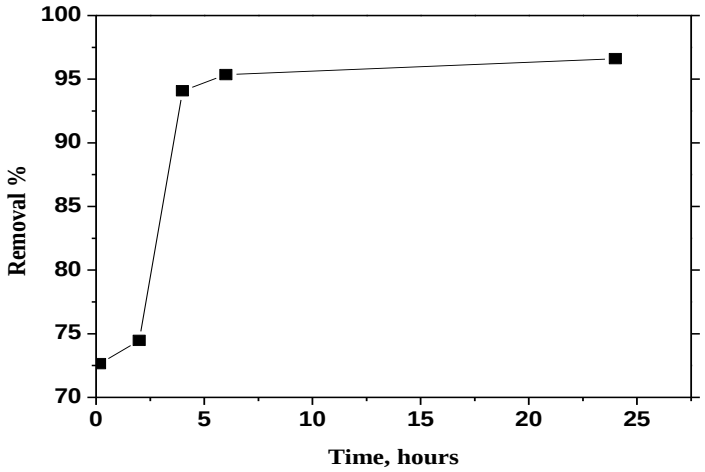

Figure 4: Effect of contact time (pH = 9, C_0 = 32 mg/L, TBdp = 0.25g/50ml (5 g/L)

Sorbent dose effect: Effect of adsorbent dosage is illustrated in Figure 5. It can be observed that removal efficiency increased sharply between bio-sorbent dosage of 0.25 g (97.1%) due to an increase in surface area and hence the number of active, and slightly increased at 0.35g (79.5%) while further increase to 0.5g the removal efficiency slightly decreased (96.6%) and so 0.25 g was considered as the optimum dose.

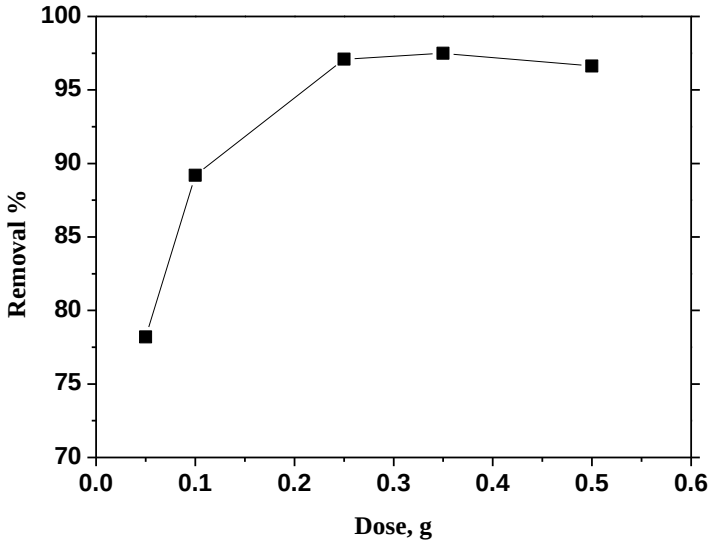

Figure 5: Effect of adsorbent dose (pH = 9, C_0 = 32 mg/L, TBdp = 0.25g/50ml (2 g/L)

Effect of initial Dye concentration: From the removal efficiency as illustrated in Figure 6, one can observe a sharp decrease in the removal efficiency from an initial dye concentration of 31.9 mg/L to 400 mg/L. The trend may be due to the limitation of active sites with increase of initial dye concentration.

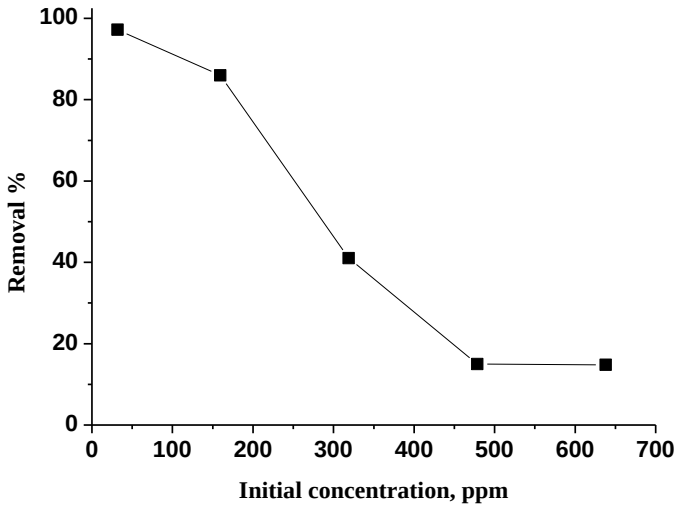

Figure 6: Effect of initial dye concentration.

CONCLUSION

Date Palm Leaf Mid rib (DPLM) can be used as a good adsorbent due to containing large quantity of phenolic compounds. It is abundant in environment & can easily be converted to a biochar and further more to activated carbon. It can be used widely in water treatment field either in simple or complicated treatment plants. Its nature makes it unique adsorbent for organic compounds especially the dyes under alkaline mediums.

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المستخلص

تم اختبار الكربون البيولوجى (البيوشار) لجريد النخيل كمادة إدمصاص ومقارنتها بمادتين مازتين أخريتين وهما فحم قوالح الذرة والفحم العادى التجارى الموجود بالأسواق. وقد تم إجراء الإختبارات الكيميائيه والفيزيائيه لتحديد الفوارق التركيبيه لهم. تم تحضير الكربون داخل فرن كهربي على درجه حرارة ٤٥٠ درجه مئويه، ثم عمل تنشيط لجزء منه. تم التنشيط باستخدام حمض الكبرتيك المخفف ١٠% لمدة ساعتين، ثم الغسيل باستخدام مياه مقطرة حتى تمام التأكد من خلو مياه الغسيل من أيونات الكبريتات. تم عمل اختبارات الإدمصاص المختلفه وذلك لتحديد أي بيوشار هو الأفضل من حيث قدرته على إدمصاص صبغة أزرق الميثلين، وقد كان الفحم المنشط المصنع من جريد النخل فى وسط قلوي pH 9 هو أقوى بيوشار من حيث كفاءة الإدمصاص، حيث قدرته على إدمصاص ٩٩,٧% من الصبغة الموجوده طبقاً لظروف التجربه. كما كشفت الاختبارات الفيزيائيه التى تمت على سطحه، أن السطح كان به فجوات كبيره وذو مساحة سطح كبيرة.

الكلمات الدالة: بيوشار، كربون بيولوجى، الصبغات، الميثلين الأزرق، الإدمصاص، جريد النخيل.