The Use of Modified Beetroot Fibers by Sodium Dodecyl Sulfate (SDS) Cleaning Water Contaminated by Organic and Inorganic Compounds

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Abstract

The beetroot fibers were used to decontaminate water polluted by methylene blue dye (MB), to remove heavy metals from wastewater and to soften hard water. In order to improve the adsorbent performance and to determine the optimum conditions of industrial wastewater cleaning, the effect of fiber particle sizes, initial concentrations of pollutants, pH of aqueous solutions and effluent rates on the cleaning procedure were investigated. Data showed that the efficiency of cleaning increased when fiber particle size decreased (from mm to μm scale). Optimum pH value for adsorption was 6 to 6.5. Maximum metal cations retention or hardness of modified fibers by sodium dodecyl sulfate (SDS) was estimated at 70 mg per gram of fiber; while the maximum retention of methylene blue was found to be 300 mg of dye per gram of fiber. Chemical modification of fibers by an anionic surfactant such as sodium dodecyl sulfate increased the efficiency of the dye elimination by 2-fold when compared to unmodified fibers. The adsorption parameters were determined using Langmuir and Freundlich isotherms.

Keywords: modified beetroot fibers, sodium dodecyl sulfate, heavy metals, hard water softening, methylene blue, adsorption isotherm

1. Introduction

Dyes have been extensively used in dyeing paper and pulp, textiles, plastics, leather, cosmetics and food industries (Gulnaz, 2004). The colored compounds are not only visually displeasing when present in water streams but they can also inhibit sunlight penetration into the stream and affect aquatic ecosystems (Tsai, 2001). These dyes such as methylene blue (MB) are also harmful to aquatic life. MB dye causes eye burns, which may be responsible for permanent injury to the eyes of human and animals. By inhalation, it can give rise to short periods of rapid or difficult breathing, while ingestion through the mouth produces a burning sensation and may cause nausea, vomiting, profuse sweating, mental confusion, painful micturition, and methemoglobinemia (Ghosh, 2002; Feng, 2012).

Removing color from wastewater can be done via several traditional biological and physico-chemical methods (Addaou, 2012; Chern, 2001; Robinson, 2002; Gupta, 2000; Janos, 2003). Chemical methods use coagulation or flocculation (Orfao, 2006), combined with flotation and filtration. Precipitation - flocculation, electroflotation, electrokinetic coagulation and ozonation to remove color (Maurya, 2008). Several alternative biological methods have also been used for the adsorption of dyes from aqueous solutions. For instance, the invasive brown seaweed *Sargassum muticum algae* (Eugenia, 2005), the marine green Ulva When research is driven by the desire to resolve controversial issues, all sides in the debate should be *lactuca algae* (Mikati, 2013), and *Chara aspera algae* (Low 1994), have been tested for removal of methylene blue from aqueous solutions.

Various treatment systems have been developed using activated carbon as adsorbent (Hatt, 2013). Researchers have studied the production of activated carbon from palm-tree cobs (Feng, 2012), plum kernels (Tseng, 2003), cassava peel (RajeshWarisivaraj, 2001), bagasse (Tsai, 2001), jute fiber (Senthilkumaar, 2005), rice husks (Yalcin, 2000), olive stones (El-Sheikh, 2009), date pits (Girgis, 2002), fruit stones and nutshells (Aygun, 2003), and also bamboo activated with potassium hydroxide (KOH) and carbon dioxide (CO_2) (Tan, 2008). As for the

heavy metals removal from wastewater, several studies were carried out and highlighted that the biosorption of metal cations mainly depends on the interactions of metals with specific groups displayed on the biosorbent surface (Kratochvil, 1998; Bai, 2001).

In the present work, the efficiency of a new beet fiber based biofilter modified with hydrophobic chains of sodium dodecyl sulfate (SDS) was evaluated contaminated water. A dual role of the modified filter was studied; the first was its effectiveness in eliminating heavy metals and softening hard water via positive and negative charges of chemical functions and the second lies in its utility in removing organic compounds, such as dyes, by the hydrophobic chains of SDS.

2. Method

2.1 Preparation of Fibers

Beetroot fibers used were collected from different agricultural areas of Lebanon, processed, purified and used to synthesize the modified biofilter. The collected fibers were cut into small pieces, air-dried and powdered in a grinder. The samples obtained were first sieved through a 100 μm sieve and then through a 50 μm sieve, then soaked in distilled water for 24 hours at room temperature. Once washed by distilled water, fiber powder was air-dried with T_{air} of 100 ºC, and then stored until use. Double distilled water was used in all solutions. Modified fibers were prepared as follows: twenty grams of fibers were transferred into a beaker 500 ml containing 100 ml of aqueous SDS (Fischer Scientific Co.) salt solution with 2.9 g/l (~0.01 mol/l). The mixture was then heated at 50 ºC and stirred for 4 hours. After filtration through a whatman 0.45 μm filter, fibers were washed by distilled water several times then air-dried with T_{air} of 100 ºC for 24 hours.

2.2 Column Procedures to Remove Dyes, Total Dissolved Solid (TDS) and Heavy Metals

The column consisted of a plexiglass tube (30 cm height, 4.0 cm of diameter) perforated at the bottom and connected to a pumping system. In order to assure homogeneity and reproducibility of the results, the column was filled with 20 g of fibers and compressed until getting a density of 0.4 g/cm^3. The flow rate of liquid was adjusted to 4 GPM (gallons per minute) at a pressure of 15 PSI. 50 ml of polluted water was transferred into the biomass filter. The same operation was repeated ten times using the same initial solution in the objective to reach the saturation of fibers by pollutants. Solutions of methylene blue (Sigma Aldrich) with different concentrations (1000 ppm, 300 ppm, 200 ppm, 100 ppm and 50 ppm) and heavy metal solutions ($CuSO_4 \cdot 5H_2O$, $NiSO_4 \cdot 6H_2O$, $Pb(NO_3)_2$, $ZnCl_2$ (Prolabo)) prepared at different concentrations (1000 ppm, 200 ppm and 100 ppm) were transferred into the filter and tested. The pH values were adjusted to an ideal pH of 5-6 corresponding to the maximum of retention as shown in the figure 1 by the addition of aqueous HCl (Prolabo).

2.3 Instruments and Methodology

Metals were analyzed by atomic absorption spectrometry (Perkin Elmer 5000 and Shimadzu A-6800), where the corresponding lamp (Zn, Cu and Pb) was selected for each metal. TDS were measured using Oakton CON 11 handheld conductivity TDS meter. UV absorption measurements were performed on a Shimadzu using 10 mm quartz cuvette cells. Concentrations of MB in solutions were determined, before and after adsorption by a double beam UV spectrophotometer (Shimadzu, Japan) at 665 nm. The Infra Red (IR) spectrum was recorded on a Fourrier Transform Infra Red (FTIR) spectrometer (UNICAM). The surface area of the polymer samples was determined by nitrogen physisorption measurements. Nitrogen adsorption-desorption isotherms were obtained at 77 K, over a wide relative pressure range from 0.01 to 0.999, with a volumetric adsorption analyzer TRISTAR 3000 manufactured by Micromeritics. The samples were degassed under vacuum for several hours at 60 °C before nitrogen adsorption measurements.

3. Results

3.1 Beetroot Chemical Composition

The average percentage of different components of beetroot fibers are respectively 25% of cellulose, 5% of lignin, 30% pectin, and 40% hemicelluloses (Dinand 1996). The materials contain several chemical functional groups like hydroxyl, carboxylic, aldehyde, ketone, C=N. Functional groups played an important role in grafting SDS molecules and also in adsorbing and complexing heavy metals on fibers surface.

3.2 Grafting SDS Chains on the Surfaces of Beetroot Fibers

Modified fibers were prepared as follows: twenty grams of fibers were transferred into a beaker 500 ml containing 100 ml of aqueous SDS (Fischer Scientific Co.) salt solution with 2.9 g/l (~ 0.01 mol l^{-1}). The mixture was then heated at 50 ºC and stirred for 4 hours. After filtration through a whatman 0.45 μm, fibers were washed by distilled water several times then air dried with T_{air} of 100 ºC for 24 hours. The new materials obtained were

analyzed by FTIR spectrometer. The changes were attributed to grafted SDS chains on the surface of fibers. (Table 1).

Table 1. Summary of wavelengths from FTIR spectra of modified and unmodified beetroot fibers

N◦ Of lines	Unmodified beetroot fibers wavenumbers (cm^{-1})	modified beetroot fibers wavenumbers (cm^{-1})
1	3523	3523
2	3397	3364
3	3282	3282
4	2920	2926
5	1701	1731
6	1638	1627
7	1616	------
8	1430	1430
9	1243	1320
10	1239	1239
11	1106	1139
12	-----	1101
13	1057	1046

3.3 Effect of pH on the Retention Efficiency of Cations and Dyes

In order to study the effect of pH on the retention efficiency of fibers, we prepared for each metallic cation Pb, Cu and Zn, triplicate solutions at the same concentration (200 ppm) with pH values varying from 1.5 to 6.5. Furthermore, we tested the retention efficiency for TDS (total dissolved solid) Figure 1 shows that metallic cations are more easily removed with the pH increase. The percentage of retention regularly increased to reach a maximum at pH 6.5 with the following values: 100% for lead, 99% for zinc, 98% for copper and 99% for TDS of hard water.
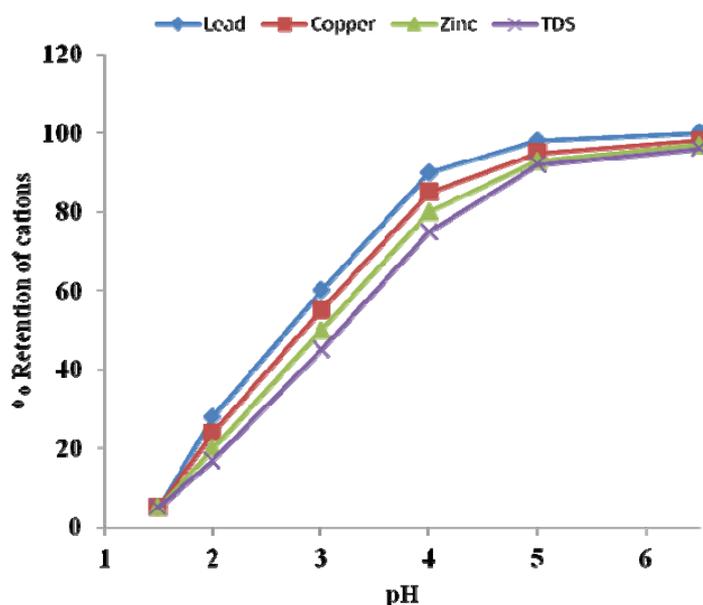
Figure 1. Effect of pH on removal efficiency of heavy metals (measurements were performed in triplicates)

While, to study the influence of pH on the efficiency of beetroot fibers in removing dyes, we filtered solutions of methylene blue (100ppm) through the fibers at variable pH ranging from 1.5 to 7. Solutions were filtered through biofilters under the same conditions regarding mass of the filter and same volume of the methylene blue solutions. Optimum pH for the maximum of removal was 6.5

3.4 Retention Efficiency of Beetroot Fibers for Cationic Model Solutions

The modified fibers were first tested to remove some metallic cations from a model solution, a mixture of lead, zinc, nickel and copper cations. The total concentration was 200 ppm (50 ppm of each). Figure 2 shows that after 10 passages, the retention efficiency of the modified fiber varied from: 100% to 96% for lead, 99% to 96% for zinc, 99% to 85% for copper and from 99% to 77% for nickel. The beetroot fibers have proven their ability to remove various heavy metals. Additional tests were conducted for softening hard water, which showed a removal efficiency of the hardness exceeding 97% for TDS = 400 ppm. Since modified fibers were prepared to improve the removal capacities for the organic compounds, it will be applied on the removal of dyes from wastewater. In addition, effect of the sizes of beetroot fibers will be studied in order to optimize the best conditions for wastewater treatment.
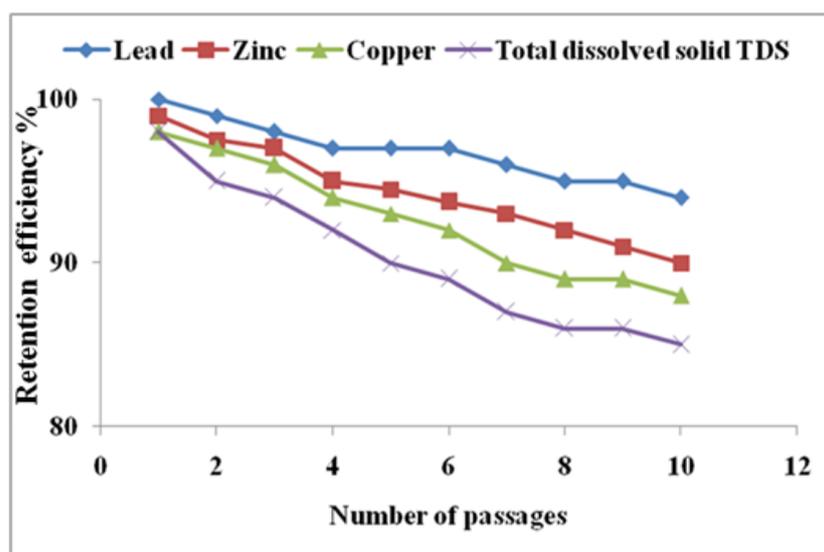
Figure 2. Retention efficiency of the modified fiber for lead, zinc, copper and TDS with the number of passages

3.5 UV-visible Absorption Spectra of Methylene Blue Solution (100 Ppm), Filtered by Unmodified and Modified Beetroot Fiber

The efficiency of the modified fibers was tested by filtration of water polluted by methylene blue at the concentration of 100 ppm and comparison of the results between modified and unmodified fibers. The UV Spectrum of the initial concentration shows a maximum of absorption at λ_{max} = 665 nm and an absorbance A = 1.11. Fifty ml of methylene blue were filtered through 20 g of beetroot fibers (50 μm) modified and unmodified and UV absorption spectra were recorded before and after the filtration of solutions. Figure 3 presents the effect of modified fibers on the removal efficiency of methylene blue. As shown, the unmodified fibers reduced 85% of methylene blue from solution at 100 ppm of concentration, while the modified fiber was able to remove more than 99%.
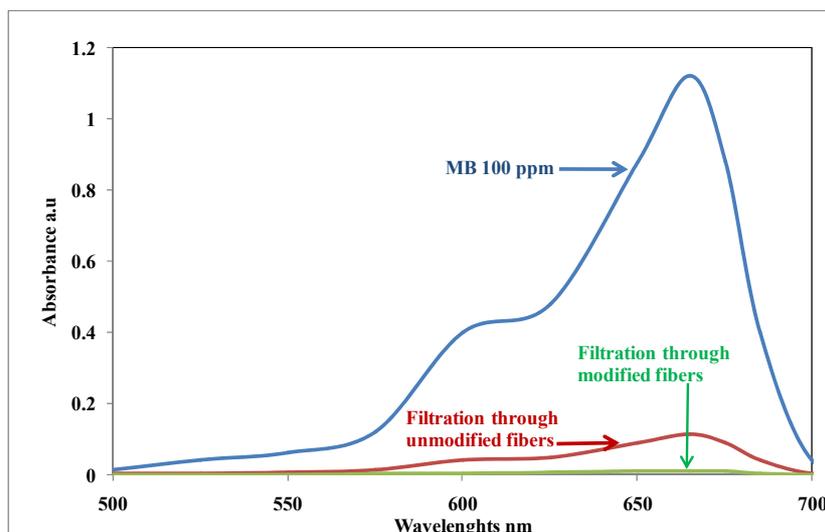
Figure 3. Effect of SDS chain on the efficiency of modified fibers

3.6 Effect of Initial Concentration on the Removal Efficiency of Methylene Blue From Wastewater Using Modified Fibers

A series of filters consisting each of 20 g of modified fiber by SDS, were ground to 50 µm, and packed into plexiglass cylinders. 50 ml of methylene blue solutions from a mother solution 1000 ppm were transferred into each of those cylinders to perform the filtration. Concentrations of methylene blue solutions used for this test varied between 50 ppm and 1000 ppm. The absorption spectra of UV-Vis solutions for all solutions before and after filtration were determined to evaluate the influence of initial concentrations on the retention capacities of the filtering system. Figure 4 shows that the removal efficiency was significantly remarkable, and it is estimated to be more than 99% for methylene blue having concentrations less than 750 ppm and 96% for concentration of 1000 ppm. Similar experiments were repeated with unmodified fibers in order to provide evidence on the effect of the hydrophobic SDS chains attached to fibers in removal of dyes. The results showed undoubtedly that the unmodified fibers used to clean polluted waters by the methylene blue were less efficient in cleaning of polluted water.
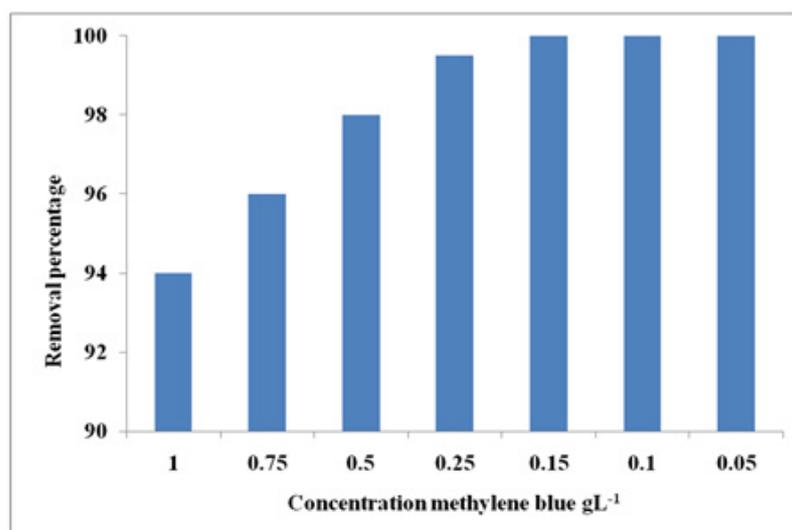
Figure 4. Effect of initial concentration on the removal efficiency of methylene blue

3.7 Effect of Fibers Size on Methylene Blue Removal Efficiency

For this objective, solutions of 1000 ppm methylene blue have been passed through similar filters differing only by sizes. Modified beetroot with dimension in mm scale (XL mm, 0.5-1 mm), 100 µm and 50 µm were tested to

determine the fibers saturation in function of the loaded volume. Results of retention efficiency in function of fibers with different sizes are shown in Figure 5. Data showed that the retention efficiency was inversely proportional to fiber size. Elimination of 1000 ppm methylene blue was 85% with XL (mm) and reached 97% with the 100 μm fibers.
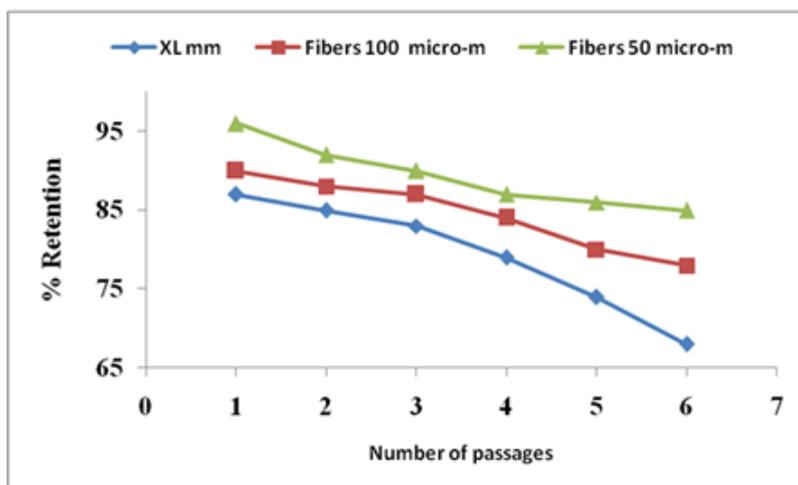
Figure 5. Retention efficiency in function of fibers with different sizes

Table 2 illustrates the retention efficiency of the modified fibers according to the loaded volume and the size of fibers.

Table 2. Removal efficiency of methylene blue (1000 ppm) by modified and unmodified fibers at different sizes (XL mm, 100 μm, and 50 μm)

Number of passages	% Removal of dye by modified fibers (XL mm)	% Removal of dye by modified fibers (100 μm)	% Removal of dye by modified fibers (50 μm)
1	95	97	99
2	93	92	98.5
3	92	90	98
4	90	84	97.5
5	85	87	97
6	83	90	96

3.8 Effect of Residence Time on the Retention Effectiveness

Two methods of filtration were performed using modified fibers with a mass equal to 20 g and sizes of 100 μm. A volume of 50 ml of 1000 ppm methylene blue mother solution was filtered through the filter described below, either by free filtration $\tau_{free\ time}$ = 20 min or by forced filtration using a special pump $\tau_{forced\ time}$ = 1 minute. The UV-Vis spectra of solutions were analyzed before and after filtration in both systems. Figure 6 shows that the retention efficiency of methylene blue was remarkably higher when the filtration was a free time operation.
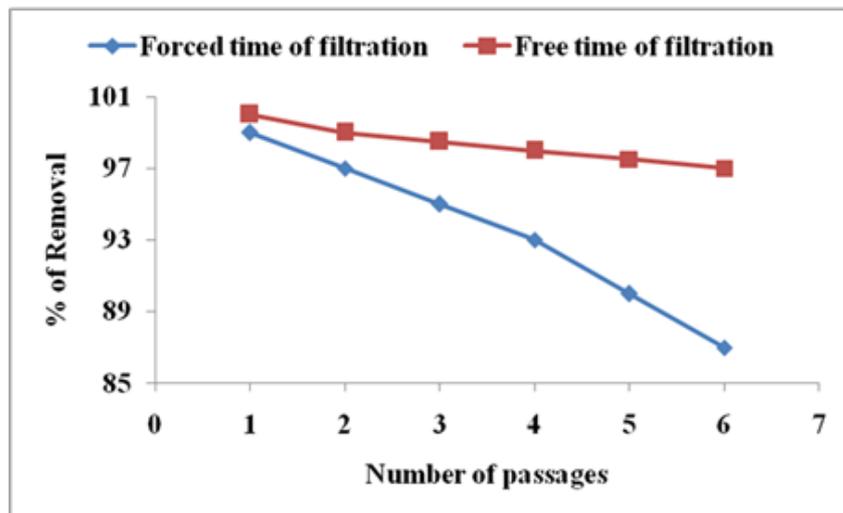
Figure 6. Effect of residence time on the retention effectiveness

3.9 Nitrogen Adsorption-Desorption

Nitrogen adsorption-desorption isotherms of the two samples (modified and unmodified beetroot fibers) (See supporting information S3). The specific surface area S_{BET} and the constant C_{BET} determined from the nitrogen adsorption data (BET method) are listed in table 3. Isotherms are not affected by the polymer nature since shapes of the two isotherms are quite similar. Nitrogen experiments performed on the two samples produced the same isotherms which could categorize samples into one group according to the IUPAC classification (Sing, 1985). Resulting adsorption-desorption isotherms had no hysteresis loop and exhibited "Type III" isotherm. The reversible type III isotherms have the representative form of isotherms obtained with nonporous or macroporous solids. In addition, they characterize weak gas/solid interactions, i.e. the surface interacts weakly with the adsorbent molecules. For this reason, the nitrogen. uptake remained low at a low relative pressure. Then, once a molecule is adsorbed, the adsorbate/adsorbate interactions will promote the adsorption of further molecules. Consequently, the resulting isotherms convert into convex form to the pressure axis.

Table 3. Nitrogen physisorption data of the samples. C_{BET} is a constant related to the difference between the molar free energy of adsorption of the first layer and the liquefaction one

Samples	BET surface area; S_{BET} (m^2/g)	C_{BET}
Unmodified fibers	2.6	< 2
Modified fibers	3.8	< 2

The BET surface areas of the two materials are reported in Table 3. No significant change was detected in-BET surface area between the unmodified fibers (S_{BET} = 2-3 m^2/g) and the modified (S_{BET} = 4 m^2/g). The surface areas remained very low, thus the two samples are nonporous or, at least, macroporous. Obtained surface area values need to be compared with those of other catalyst materials. Typically, activated carbons should have a BET surface area higher than 500 m^2/g (Garcia-Garcia 2003; Sricharoenchaikul, 2008; Rima, 2013), because as standard catalysts (or photocatalysts) used in water treatment display a surface area of the order of 200 m^2/g (Blin, 2012; Zimny, 2012).

Furthermore, it is interesting to note that the hydrophobic behavior of the samples was highlighted by the very low value of the C_{BET} constant (C_{BET} < 2). In fact, the C_{BET} parameter gives an indication on the adsorbate/adsorbant interaction according to

$$C_{BET} = \exp\left(\frac{E_{ads} - E_{liq}}{kT}\right) \qquad (1)$$

Where E_{ads} is the energy of a molecule adsorbed in the first layer, whereas E_{liq} is the adsorption energy in the other layers identified to the liquefaction energy. Determination of the C_{BET} value has been proposed recently as a method to assess a surface polarity. The parameter C_{BET} is high for polar surfaces and low for non-polar surfaces (Brunel, 2000; Trens, 2004). In the present study, the very low value of the parameter C_{BET} emphasizes the large hydrophobic character of used materials. Specific surfaces of the samples of modified and unmodified fibers were determined by measurements of nitrogen physisorption. Low values of specific surfaces indicated that removal efficiency for heavy metals from-hard water TDS and dyes cannot be attributed to a specific surface

3.10 Adsorption Isotherms

Two important physiochemical aspects for the evaluation of the adsorption process as a unit operation are the equilibrium of the adsorption and the kinetics. Equilibrium studies evaluate the capacity of the adsorbent. The equilibrium relationships between adsorbent and adsorbate are described by adsorption isotherms, usually equivalent to the ratio of the quantity adsorbed to the one remaining in solution at a fixed temperature at equilibrium. There are two types of adsorption isotherms: Langmuir adsorption isotherms and Freundlich adsorption isotherms.

(a) Langmuir isotherm. The Langmuir adsorption isotherm is often used for adsorption of a solute from a liquid solution. The Langmuir adsorption isotherm is often expressed as

$$Q_e = XmKC_e/(1+KC_e)$$

Where, Q_e is the adsorption density at the equilibrium solute concentration C_e (mg of adsorbate per g of adsorbent). C_e is the concentration of adsorbate in solution (mg/L). Xm is the maximum adsorption capacity corresponding to complete monolayer coverage (mg of solute adsorbed per g of adsorbent). K is the Langmuir constant related to energy of adsorption (L of adsorbent per mg of adsorbate). The above equation can be rearranged to the following linear form

$$C_e/Q_e = 1/X_mK + C_e/X_m$$

The linear form can be used for linearization of experimental data by plotting C_e/Q_e against C_e. Langmuir constants X_m and K can be evaluated from the slope and intercept of linear equation.

(b) Freundlich isotherm. Freundlich isotherm is the relationship describing the adsorption equation and is often expressed as

$$Q_e = K_f C_e^{1/n}$$

where, Q_e is the adsorption density (mg of adsorbate per g of adsorbent). C_e is the concentration of adsorbate in solution (mg/L). K_f and n are the empirical constants dependent on several environmental factors and n is greater than one. This equation is conveniently used in the linear form by taking the logarithmic of both sides as:

$$LnQ_e = LnK_f + 1/n \; LnC_e$$

A plot of LnC_e against LnQ_e yielding a straight line indicates the confirmation of the Freundlich isotherm for adsorption. The constants can be determined from the slope and the intercept.

3.10.1 Method Used for Adsorption Test

The method used for the adsorption tests for different methylene blue concentrations is as follows: (1) 20 g of fibers as an adsorbent were transferred into a column and different concentrations of methylene blue were filtered through the column. (2) For each concentration, a volume of 500 mL of methylene was filtered through the modified fiber several times until complete saturation of the filter. The concentrations that has been tested were 1000, 850, 650, 500, 300 and 200 ppm. (3) Q_e was determined and C_e/Q_e vs C_e and LnQ_e vs LnC_e were plotted. The experimental results of retention capacities obtained for different sizes of fibers modified and unmodified and the captured mass by gram of fibers were calculated and summarized in Table 4.

Table 4. Experimental results of retention capacities obtained for different sizes of modified and unmodified fibers. Removed methylene blue (mg) by

Passages	XL fibers (mm) each passage	100 μm fibers each passage	by 50 μm fibers each passage	modifed XL fibers (mm) each passage	modified fibers 100 μm each passage	modifed fibers 50 μm each passage
1	48	49	49.5	47.5	50	50
2	43	47	48	46.5	49	49.75
3	35	46	47.5	46	48.5	49.65
4	30	44	45	44	48.5	49.6
5	28	42	44.5	35	48	49.5
6	22	42.5	43.5	31	47	49.5
7	18	40	30	26	30	47
8	15	28	25	16	20	45
9	12	20	22	16	15	37
10	10	15	20	12	15	35
11	10	15	20	10	15	35
Total removed mass	271	356.5	395	330	381	500
Total removed mass per g of fibers	13.5	17.8	19.75	16.5	19.05	25

Langmuir and Freundlich plots are presented in Figure 7a and 7b respectively. For both isotherms, the estimation of adsorbed quantities of methylene blue are fitted. In the two models Freundlich and Langmuir, the amount of dye absorbed by fibers provides acceptable linearity which confirms the accuracy of our experimental results.
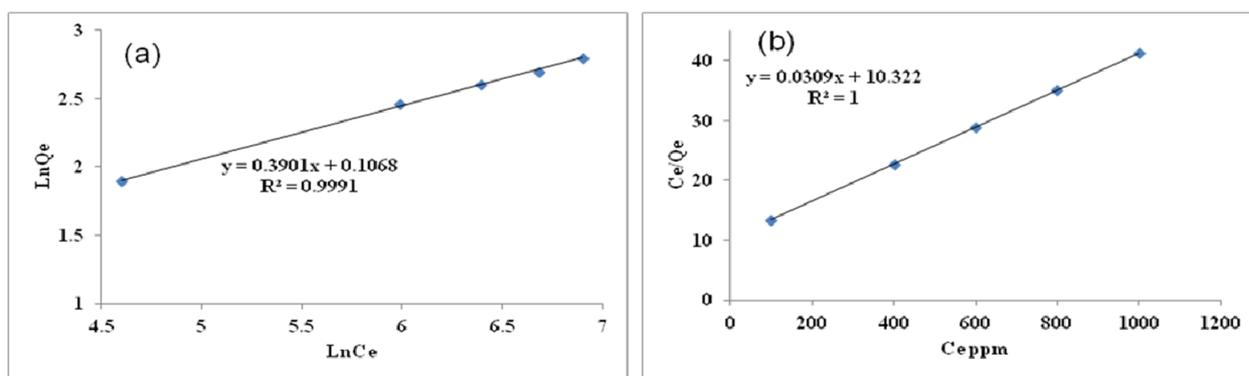
Figure 7. (a) Adsorption Freundlich isotherm modified fibers 50 microns, (b) adsorption Langmuir modified fibers 50 microns (the values 850 ppm and 750 ppm were averaged by the value of 800 ppm)

4. Discussion

Georgiev et al. (2012) has found that the removal of Cu(II), Zn(II), Cd(II) and Pb(II) ions in aqueous solutions through synthetic zeolite was lower than 20 mg g^{-1} even after 20 minutes of contact time. Moreover, Murugesan 2006, has found that the capacity of removal of Arsenic III and Arsenic V by fungal biomass were 1.11 and 4.95 mg/g respectively. The modification of fibers by SDS showed an enhancement of the removal capacity compared to unmodified fibers and previopusly reported work (Murugesan, 2006; Tangahu, 2011; Georgiev, 2012). The removal mechanism likely involves complexation by chemical functions of fibers and adsorption by electrostatic

Vander Waals interactions. The new aspect of the reported method here is its capability to have a dual role in the removal of heavy metals by complexation mechanism, and removal of oganic pollutants through the hydrophobicity of the SDS chains. Removal of methylene blue from polluted water changed with the pH solution, and the optimum removal efficiency was obtained at pH 6.5. Furthermore, removal efficiency increased significantly with the decrease in fiber size. Freundlich and Langmuir isotherms were established and described the removal process indicating favorable elimination of methylene blue from polluted water.

Declaration of Interest

Authors have no conflict of interest, in particular no financial, consulting and personal relationships with other people or organizations that could influence (bias) the author's work.

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