

## Removal of Pb (II) from aqueous solution with Reactive Red 198 and carbonization of sugar beet pulp with citric acid

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### Abstract

In this study, a high carbon activated carbon was obtained from the sugar beet pulp by carbonizing with concentrated citric acid. The main purpose of this study is to evaluate the obtained catalyst for heavy metal removal in waste water. The adsorption studies of a basic dye Red 198 on the sugar beet pulp (SBP) were investigated. Balance and kinetic studies, carbon adsorbents obtained from sugar beet pulp were used for balance and kinetic studies. With carbonization of sugar beet pulp with citric acid was studied to remove Pb (II) from Reactive Red 198 dye aqueous solutions. Adsorption in research were performed to investigate the effects of solution concentration (20-30-40 mg/L), temperature (25,35,45°C) and pH (3-6-9) on the separation of Reactive Red 198 aqueous solution from beet pulp. Carbon balance adsorbent obtained from sugar beet pulp was done in equilibrium and kinetic studies. Langmuir and Freundlich isotherm models work were done to be appropriate for Reactive Red 198 adsorption. The maximal lead adsorption capacity obtained from Langmuir isotherm at pH 8 and 25°C 837.53 mg/g. Adsorption of Pb (II) is an endothermic process. The results showed that Reactive Red 198 from the beet pulp is an inexpensive and efficient adsorbent for the removal of aqueous solutions.

**Keywords:** Sugar beet pulp (SBP), Carbonization, Pb (II) adsorption, Citric acid, Reactive Red 198

### Introduction

In the plastic, textile, dyeing and finishing processes, dyeing, coloring matter, leather, cosmetics, pharmaceuticals, food and paper manufacturing industry consume very big amounts of multicolored waste water at distinct stages (Yao et al., 2010). The use of paints in these industries constitutes an important class of pollutants (Hussien et al., 2016). The basic use of dyes is to change the color properties of different substrates such as paper, fabric, leather, and others. It has been determined that dyes affect photosynthetic activity to a great extent (Inastopoulos et al., 2014; Ferreira et al., 2014). In industry like this, it is seen that multicolored wastewater is discharged to water bodies such as streams and rivers and causes serious environmental effects. It also inhibits color light penetration, delays photosynthetic activity, and inhibits biota growth. Heavy metals in living life are considered to be one of the most toxic groups. In the food chain, it is possible to access

wastes to water receptors or landfills. Heavy metals have been found to cause toxic effects, cancer and muscle-nerve diseases as they are very difficult to remove from the body (Petrović et al., 2014; Milojković et al., 2014).

SBP, a byproduct of sugar beet behind sugar extraction, produces approximate 14 million tons on dried matter basis every year in the European Economic Community, whichever consisting firstly of hemicellulose, pectin and cellulose (Gerente et al., 2000). This product is generally supplied to farmers for use as animal feed, but it is also used in various methods to increase the value of paper production (Vaccari et al., 1994; Vaccari et al., 1997), such as detergents (Petiti et al., 1993), fibers (Bertin et al., 1988; Michel et al., 1988) and pectins (Arslan 1995; Levigne et al., 2002). Removal of diverse organic and inorganic pollutants in industrial wastewater is insignificant practice in adsorption processes using appropriate adsorbents. Nowadays, attention has been paid to the manufacture of

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lowcost sorbents from agricultural waste or products. Cellulosic products grown in agriculture are used in carbon production in the separation and purification stages of industrial processes. Sugar beet produces wastewater, emissions, odors, and solid waste and causes environmental pollution to a large extent. Plant material and sludge washed in large mills are broken down in freshwater bodies, absorbing available oxygen (Gopal et al., 2009; Clay., 2013). The transformation of SBP to activated carbon is an economically inexpensive alternative. Since the beet pulp contains carboxyl groups and high metal cations, pH, presence, and concentration of organic and inorganic ligands which absorb nature are important factors Violante et al., 2010; Moubarik et al., 2015). In addition, the cationic species of polysaccharides and their modified products are effective in adsorbing aqueous solutions. Therefore, SBP is used in heavy metal adsorption to prevent environmental pollution (Sharma et al., 1994; Altundogan, 2005).

Processed agricultural materials have been shown to increase absorption capacities for metal ions of poly acids (such as citric, tartaric, phosphoric) at slightly high temperatures. With these processes, some extra acidic groups may form an ester linkage in the material. (Wong et al., 2003; Lehrfeldt 1997).

Adsorption of Pb (II) from aqueous solution with SBP was investigated by Pehlivan et al (2008).It is stated that the adsorption process is relatively fast and a 70-75% removal of Pb metal is achieved in 70 minutes.It is stated that adsorption density is pH dependent, 43.5 mg / g at pH 5 for Pb (II) and removal occurs in the form of ion exchange, physical adsorptionand chemical adsorption.In differentwork conducted by the similar researcher, sugar beet pulp was used for adsorption of Zn (II)

from aqueous solutions (Pehlivan et al., 2006).In the study, it is stated that the process reaches equilibrium in 60 minutes and maximum adsorption concentrations are determined as 35.6 mg / g at pH 6.0 for Zn (II).

In this work, sugar beet meal was treated with citric acid to obtain a substance with a high carbon content. The main purpose of this work is to evaporate solid products or auxiliary products obtained during carbonization of divalent lead in gas, liquid and aqueous solutions. This purpose, the impact of pH, contact time, solution concentration and solution temperature wereresearched. Experimental data were associated with properties of binding capacity, kinetic and thermodynamic were discussed. The hexavalent lead adsorption properties of the obtained carbonaceous material were determined.

## Materials and Methods

### Materials

SBP was supplied from Uşak sugar endustry. The SBP was dehumidified by blowing air for 20 hours. It was dried in an at 55 °C for 22 hours. The dough was milled in a mixer. These-milled material was sifted in sieve shaker and The samples to be used in the experiment are divided into micro grain sizes. 120 mesh (125µm) samples were used in each step of the experiment.

The chemical structure of Reactive Red 198 (MW=984.2 g/mol) dyestuff is given in Figure 1 and was chosen as the adsorbate. Firstly, a wavelength scan was performed for this dye-stuff and the highest absorbance value was found to be 519 nm. In the following studies, measurements were made by keeping this value constant.

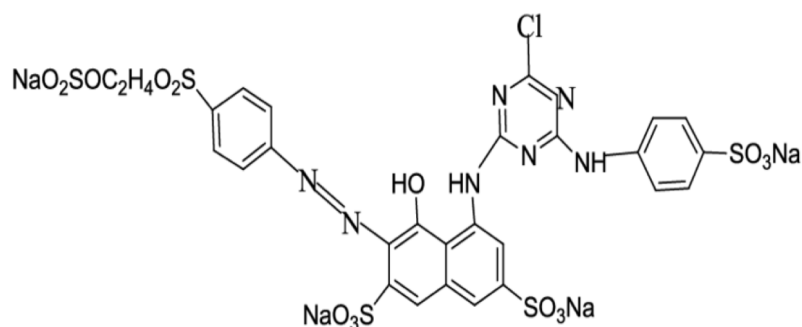


Figure 1. Chemical structure of Reagent Red 198 (Tunali et al., 2009)

### Preparation of sugar beet pulp

The SBP was esterified with citric acid solution. For this purpose, 30 g beet pulp samples were mixed with 0.7 M citric acid ( $C_6H_8O_7 \cdot H_2O$ ; Merck 100242; 99%) solution in a glass bowl at a rate of 8 times their weight. This mixture was mixed with the aid of a glass baguette to allow the citric acid solution to penetrate well into the beet pulp. After the solid had absorbed the solution, the mixture was left in the oven at 55 °C for 28 hours. The weight of the obtained material was determined. It was then heated in an oven at 110 °C. For 100 minutes to remove the water formed from the esterification with the internal water. The weight of the material was determined again. The resulting material was rinsed with distilled water at

a rate of 45 times its weight each time for 1.5 hours until no citric acid was left in the wash water, and at the end of this time the filtrates were removed. To determine the presence of citric acid in the wash water, 15 mL of 0.25 M  $Pb(NO_3)_2$  solution was added to 15 mL of wash water and precipitation (lead citrate precipitate) was observed. In the washing step where no precipitation was observed, it was concluded that citric acid did not pass into the washing water anymore and the washing process was terminated. This washed product was spread by thin film to remove coarse moisture by means of a fan and then dried to constant weighing (~ 24 hours) in an oven at 110°C. Once the final weight was determined, it was stored in a closed container for use in the experiments.

### Adsorption study

Adsorption studies were performed at 25 °C by adding a constant quantity of adsorbent (2.0 g) into an amount of 250 ml of glass containing 150 ml of solution at distinct starting concentrations (20, 30 and 40 mg / L). The pH (3, 6 and 9) of this dye solution was determined using 0.12 M NaOH and 0.12 M HCl solutions and measurements were made using a pH meter. The test tubes were placed on a rotary shaker (150 rpm). An equilibrium was maintained for 200 minutes. These samples were puted in a centrifuge tube and centrifuged at 4000 rpm for 8 minutes. The concentration of Reactive Red 198 in the supernatant solution was investigated by measurement of the optical density at 519 nm using a spectrophotometer.

The quantity of lead adsorbed was detected according to the measured lead concentration before and after equilibration. The amount of adsorption at equilibrium  $q_e$  (mg/g) was provided by the following equation:

$$q_e = \frac{V}{m} (C_i - C_e) \quad (1)$$

where  $C_i$  and  $C_e$  are the initial and equilibrium liquid phase concentrations (mg/L),  $V$  the volume of the solution (L) and  $m$  is the weight of the sugar beet pulp used (g).

### Isotherm analysis

Adsorption isotherm explain the coaction among the solution and is used to determine the adsorbent and highest adsorption. The dried sugar beet meal reveals the binding capacity to Reactive Red 198. Results obtained were investigated by applying the model Freundlich (Freundlich, 1906) ve Langmuir (Langmuir, 1916). Freundlich isotherm is the most widely used model for defining an adsorption method of heavy metal ions in aqueous solution. Freundlich isotherm occurs by the accumulation of metal ions with heterogeneous surface through multi-layer adsorption. The quantity of adsorbed increased as a result of increasing concentration. Equilibrium data were investigated with the Freundlich equation:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (2)$$

$C_e$  (mg/L) and  $q_e$  (mg/g) equilibrium concentrations and the quantity of dye adsorbed at equilibrium,  $K_f$  adsorption capacity and  $n$  adsorption intensity are indicative of Freundlich constants (Kong et al., 2014).  $K_f$  and  $1/n$  can be calculated from  $\ln q_e$  and  $\ln C_e$  graph.

The best of all sorption isotherms applicable to sorption processes is the Langmuir model. In this model, the amount of absorption on the surface of each sorbate molecule has equal absorption activation energy and can be calculated to define sorption of an ionic species of Pb (II) from an solution (Ed. 2).

$$\frac{C_e}{q_e} = \frac{1}{bq_{max}} + \frac{C_e}{q_{max}} \quad (3)$$

where  $C_e$  is equilibrium concentration of Pb (II) (mg/L),  $q_e$  the amount of Pb (II) adsorbed per unit mass of sorbent (mg/g) and  $q_{max}$  is the highest sorption capacity (mg/g).  $b$  (L/mg) is a constant connected to the concern of binding sites or bonding energy. If the value gives a straight slope with respect to  $C_e/q_e$  intersects  $1/q_{max}$  and  $1/bq_{max}$  and shows sorption in the Langmuir model.

### Results and Discussion

Some properties of SBP treated with citric acid are summarized in Table 1. In the experimental stage with citric acid, it is seen that the capacity to bind some extra carboxyl groups of cellulose made strong in independent first alcohol groups is increased. Depending on the solubility of some organic materials for the extracted example, a measure of organic pollution may show a stabilization of a lower chemical oxygen demand value. The results show that a basis processing followed by a citric acid alteration stabilizes the sugar beet pulp because of crosslinking and increases the cation uptake resulting from carboxylic groups introduced into the molecules.

Table 1. Contents of SBP and citric acid-treated SBP

Parameter	Sugar beet pulp	Citric acid-treated sugar beet pulp
Water retention capacity (g/g)	8.21	5.07
Swelling capacity (ml/g)	7.24	4.13
Moisture content (%)	6.52	6.11
Ash content (%)	4.58	5.79
Bulk density (g/ mL)	0.302	0.373
Conductivity <sup>b</sup> (µs/cm)	156	242
pH <sup>1</sup>	4	8
Chemical oxygen demand <sup>1</sup> (mg-O <sub>2</sub> /L)	169.7	72
Cation exchange capacity <sup>2</sup> (mequiv./g)	0.91	4.27

<sup>1</sup>Measurements in the liquid obtained by equilibrating 1g of substance with 100 mL of water for 24 hours

<sup>2</sup>On dry basis (at 110 °C)

### Effect of contact time at Pb(II) adsorption

Different experiments were kept for equilibration with initial Red 198 dye concentration. Figure 2 shows the adsorption kinetics of the dye holding capacity of Reactive red 198 at 25, 35 and 45°C over time. The amount of dye getting adsorbed

on the surface of activated carbon increases with increasing contact time till a tableland is achieved. It represents the state of dynamic equilibrium in which the amount of dye adsorbed onto the adsorbent is in equilibrium with that of the present in solution. Adsorption studies were performed for 8 hours

and It is seen from Figure 2 that the adsorption capacity increases with increasing temperature and time. More dye was removed during the initial 100 minutes contact time. Finally afast adsorption for all studied temperatures, the equilibrium was established in 130 minutes. After an equilibrium time of 150 minutes, the graph now shows that Reactive Red 198 is very well adsorbed.

The attainment of equilibrium takes a bit longer time due to a complex mechanism involved in the adsorption of dyes on macro and micro pores of activated carbon. The mechanism entails the confrontation of dye molecules to the boundary layer before diffusing onto the surface of adsorbent and then finally entering into the porous structure of carbon.

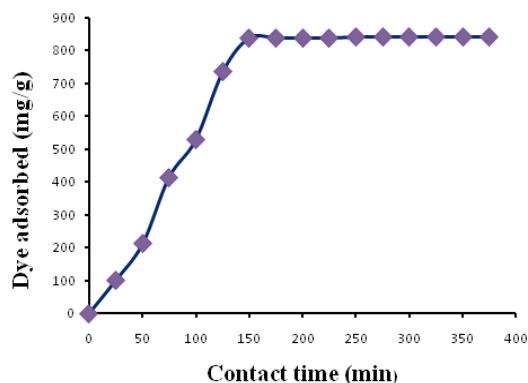


Figure 2. Influence of contact time on the removal of Pb(II) by using of citric acid-treated SBP (pH 8.0; adsorbent dosage: 5 g/l; initial Pb(II) concentration: 25–450 mg/L; temperature: 25 °C)

In most of the relevant studies published in the literature, it has been found that pH is an significant be effective component for dye adsorption. Experiments were implemented in the pH range from 1.0 to 10.0 to find a appropriate pH value for influence adsorption of the Reactive Red 198 dye by citric acid-treated sugar beet pulp. The increase in Pb(II) adsorption from 1 to 6 was due to increasing number of coordinate bonds between Pb(II) and nitrogen atoms of amine groups on SBP hydrogel. The decrease in adsorption from pH 8 to 10 was attributed to the gradual precipitation of Pb (II) ions to from  $Pb(OH)_2$ . Red 198 dye adsorption decreased with increasing pH which was due to the gradual deprotonation of  $NH_3^+$  on SBP hydrogel to hydrophobic  $NH_2$  thereby increasing electrostatic repulsion between the hydrogel and Red 198 dye molecules (Akiode et al., 2015). The change in equilibrium dye uptake with initial pH is shown in Figure 3. The highest equilibrium uptake value was obtained to be 837.53 mg/g at

pH 8.0.

It was at the lowest level in pH 2 in dye adsorption, severely rised at pH 4 and reached its highest level at pH 8. Since weakest acidic groups are low in PH 2, the total surface charge on the SBPhappens less negative by being surrounded by hydronium ions. This reduces the interaction of the dye cations with the larger driving forces with the binding sites of the citric acid-treated SBP and decreases the attractiveness of positively charged cations (Pehlivan et al., 2008). This pH values greater than 4, the carboxyl groups of citric acid-treated sugar beet pulp are deprotonated and charged negatively. Therefore, it can be assumed that the maximum adsorption level is achieved that all carboxyl functions of citric acid-treated sugar beet pulp are deprotonated and possibly neutralized by electrostatic interaction between the negatively charged adsorbent and Reactive Red 198 molecules.

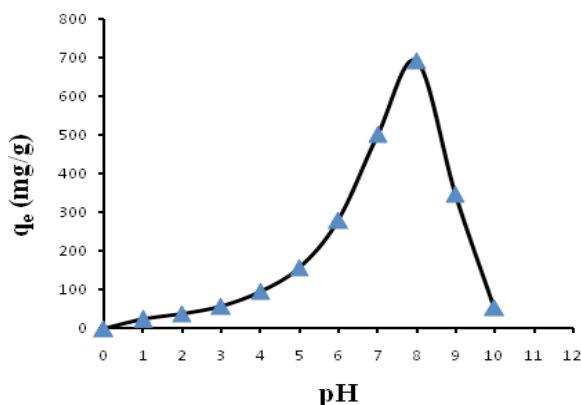


Figure 3. Influence of pH on the Reactive Red 198 adsorption ( $C_0 = 50$  mg/L,  $T = 25^\circ C$ )

### Effect of temperature on adsorption

Temperature effects rate and degree of adsorption. In addition, For a contact period on the effect of temperature Pb (II) adsorption on citric acid-treated SBP, the range of 25-35-45°C was studied (Table 2).

As shown in Table 2, the equilibrium adsorption ( $q_e$ ) rised with the rise of the initial Reactive Red 198 concentration. Temperature effects the ratio and degree of adsorption. In addition, the temperature dependency of adsorption ensures thermodynamic and mechanical knowledge regarding the sorption process. The influence of temperature on the effect of Pb (II) adsorption on citric acid-treated SBP was studied at 25-45 °C for a 6 hour contact period in solutions with initial concentrations ranging from 22 to 40 mg/L.

The highest yield is seen in Table 2, where the removal of the dye increases to 837.53 mg/g at 25 ° C. The increase in temperature, the increase in the number of active surface areas for adsorption on the surface of the adsorbent may be related to the increase in the pore volume of the adsorbent. It can be said that the increase in adsorption may be caused by the tempera-

ture drop in the thickness of the layer surrounding the sorbent. Therefore, the mass transfer in the adsorbate layer causes a decrease in resistance. At the same time, the increase in kinetic energy increases the activity of the dye molecule. The increase in dye concentration caused a decrease in the adsorption movement. At low concentrations, the dye had a low absorption rate and higher biosorption yields. At higher concentrations, the existing adsorption sites were reduced and saturation of the sorption sites appeared. Therefore, a decrease was observed in adsorption efficiency values (Table 2).

### Adsorption equilibrium studies

Adsorption experiments were performed under agitation using activated carbon and aqueous Red 198 dye at 25, 35 and 45 °C. The adsorption experiments were implemented to work effects of diversity initial Red 198 concentration, SBP amount and temperature (25, 35 and 45 °C). The concentration of dye was monitored after fixed interval of time (50-100 min) using UV-Visible spectrophotometer at absorption maxima of 519 nm. The unit adsorption capacity are shown in Table 2.

Table 2. Equilibrium uptake capacities and adsorption yields obtained at different concentrations and temperatures

$C_0$ (mg/L)	25°C		35°C		45°C	
	$q_{eq}$ (mg/g)	Adsorption (%)	$q_{eq}$ (mg/g)	Adsorption (%)	$q_{eq}$ (mg/g)	Adsorption (%)
25	837.53	94.12	794.71	83.73	516.18	61.80
35	802.47	90.18	739.5	77.91	493.62	59.09
45	673.57	72.48	557.38	58.73	286.49	34.30

### Isotherm study

Adsorption isotherm is the work of equilibrium relationship between adsorbate adsorbed on the surface of adsorbent and adsorbate remained in solution, if they are kept in contact with each other for longer periods of time. The data obtained in the adsorption experiment at distinct temperatures were investigated by placing Freundlich and Langmuir isotherm models (Langmuir, 1918; Freundlich, 1907). The Langmuir model is the most accepted one for the application of activated carbon in waste water treatment. This model takes on the monolayer formation on the surface of adsorbent which applies the more specific binding of adsorbate. All sites on the surface of adsorbent are equivalent and there occurs no interaction between adsorbate and adsorbent molecules. Freundlich isotherm is used in the adsorption of ions in heavy metal solutions since it assumes that the collection of metal ions occurs on a heterogeneous surface through multilayer adsorption and that the amount of adsorbate adsorbed rises infinitely. The best of all adsorption isotherms in adsorption processes is the Langmuir model. In this model, each sorbate molecule had the same absorption ac-

tivation energy as the absorption capacity of the surface of the molecule. Langmuir adsorption isotherm can be used to define the absorption of an ionic species such as Pb (II) from aqueous solutions. Experimental data investigated at 25 °C for pH 3-9 and Reactive Red 198 solutions, the concentration of 20-40 mg/L, equilibrium values of the liquid phase concentration and the values corresponding to the amount of adsorbed material were used to generate the adsorption isotherms of the concentration of the dye. Freundlich and Langmuir equations give the coefficients in Table 3. The isotherm models to the adsorption work is evaluated by the coefficient of determination ( $R^2$ ) of each surface. It is seen from the data that Freundlich and Langmuir isotherm models provide a well-suitable fit in all investigated pH values. The values of correlation coefficient were almost ideal ( $R^2 > 0.999$ ) for pseudo second order kinetic model, which shows a good agreement in theoretical and experimental results as shown in Table 3. In addition to, a good correlation was observed between  $q_e$  values, calculated using pseudo second order model and the experimental observation.

Table 3. Isotherms constants for Reactive Red 198 adsorbed on citric acid-treated Sugar beet pulp (Eq. 1 and 2)

T (°C)	Langmuir model			Freundlich model		
	$q_{max}$	$K_b$	$R^2$	$K_F$	n	$R^2$
25	838.01	2.96	0.999	207.83	57.61	0.987
35	793.14	2.79	0.946	196.71	2.94	0.857
45	531.80	1.87	0.634	131.89	1.97	0.564

## Conclusion

This article presents the improvements in the use of waste materials from the sugar industry as adsorbents and their performance in eliminating various water pollutants. The study found that surface modification with chemicals greatly improves the efficiency of favorable kinetic sequencing and adsorption yields and the extraction of adsorbents from sugar beet waste. Adsorbents' performance depends largely on pollutant types and experimental conditions. Most of the studies are aimed at determining maximum adsorption capacities using a single synthetic contaminant solution.

In this study, modified sugar beet pulp was investigated by treatment of lead absorption with citric acid. Characterization tests of the changed sugar beet pulp (SBP) showed improved cation exchange capacity, equilibrate hydration and dissolution properties. Adsorption studies were performed to investigate the effectiveness of modified sugar beet pulp in lead binding. The surface chemistry of carbons also change on varying the type of treatment. Langmuir and Freundlich isotherm models were analyzed according to temperature. The Langmuir model best correlates experimental data. Kinetic evaluations show that lead absorption follows the second order kinetic model. According to the results of the experiment, dye adsorption is exothermic depending on the temperature. The maximum adsorption capacity of the sugar beet pulp for Reactive Red 198 was 837.53 mg/g at 25 °C, pH 8. In general, this study showed that there may be a simple sorbent from SBP with high lead removal capacity.

The present work demonstrates a viable approach for the preparation of tailor made carbon from the sugar beet pulp (SBP) and their potential in removing Red 198 from aqueous streams.

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