The European Journal of Mineral Processing and Environmental Protection
Vol.5, No.1, 1303-0868, 2005, pp. 26-34

Cu(II) adsorption from aqueous solutions on sugar beet pulp carbon

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Accepted 4 September 2004

ABSTRACT

Sugar beet pulp was carbonized at different temperature to convert into an effective adsorbent, namely, sugar beet pulp carbon (SBPC) to be used for the removal of Cu(II) ions from aqueous solutions. The parameters affected the adsorption yield such as pH, contact time, adsorbent dosage and initial Cu(II) concentration were optimised. The optimum pH was determined as 5.5, and it was found that the adsorption reached equilibrium state in 120 min. Equilibrium data were fitted to both Langmuir and Freundlich adsorption models in the concentration range studied. The maximum adsorption capacities of Cu(II) ions were 12.24, 13.44 and 14.81mg/g for SBPCs obtained at 300, 400 and 500°C, respectively. The adsorption of Cu(II) ions were analysed using Lagergren pseudo first-order kinetic model. The results showed that the Cu(II) adsorption process followed the first order rate expression and the adsorption rate constants decreased with increasing carbonization temperature. © 2005 SDU. All rights reserved.

Keywords: Sugar beet pulp; Carbonization; Adsorption; Cu(II) ions; Isotherms

1. INTRODUCTION

The primary sources of copper pollution in aquatic environment are wastewaters of mining and plating industry and agricultural sources such as fertilisers, fungicidal sprays and animal wastes (Sittig, 1973; Kabata-Pendias and Pendias, 1992). Like all toxic metals, copper cannot be destroyed in the natural environment by biological degradation and accumulates in living organism, resulting in liver damage and acute poisoning to human body. Before the industrial wastewaters containing toxic metals are discharged into natural water sources, the concentration of these metals should be reduced under the tolerance limits proposed by water quality standards. For that reason, various treatment methods that can remove and recover copper from waste solutions are needed. The most widely used treatment method for copper removal from wastewaters involves chemical precipitation as hydroxide at alkaline pH or sometimes as sulphide (Sittig, 1973; Birdgwater and Mumford, 1979). A major problem with this type of treatment is the solubilisation of deposited precipitants (hydroxide and sulphide) as a potential source of pollution for the metal ions. The other treatment methods used to remove Cu(II) from aqueous wastes are ion exchange, evaporation and electrolytic recovery processes (Sittig, 1973). On the other hand, in the recent years adsorption techniques that use inexpensive adsorbents for the removal of heavy metals from waste streams have been considered. Many materials that can be used to produce low cost adsorbents are available in large quantities either as industrial waste and agricultural by products or as natural sources. These type of adsorbents can be assumed as low cost since they require little processing and are abundant in nature (Baily *et al.*, 1999). The agricultural by products investigated for Cu(II) adsorption include coirpith (Kadirvelu *et al.*, 2000), coirpith carbon (Namasivayam and Kadirvelu, 1997), peanut hull carbon (Periasamy and Namasivayam, 1996), waste fruit residues (orange, apple, pineapple, dates, grapes) (Senthilkumaar *et al.*, 2000) and baggase fly ash (Gupta and Ali, 2000) from sugar industry. It has been reported that carbonaceous materials such as sawdust (Ajmal *et al.*, 1998; Yu *et al.*, 2000) and pine bark (Al-Asheh *et al.*, 2000) have the capacity to uptake Cu(II) ions from aqueous solutions.

Sugar beet pulp is a carbonaceous material that mainly consists of cellulose and its derivatives and lignin. Some researchers used sugar beet pulp as an adsorbent in their studies (Dronnet *et al.*, 1999; Reddad *et al.*, 2002). It has been reported that the activated carbon from sugar beet is an effective adsorbent for the

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removal of Cd(II) (Ozer *et al.*, 1998) and Cu(II) (Tanyildizi *et al.*, 1999) ions from aqueous solutions. The main objective of this study was to investigate the use of an alternative adsorbent obtained by direct carbonization of sugar beet pulp, a by-product of sugar industry, without activation in the removal of Cu(II) ions from aqueous solutions.

2. EXPERIMENTAL

2.1. Material

Sugar beet pulp was provided from a local sugar factory at Elazig, Turkey. The sugar beet pulp was dried to reduce its moisture content and then stored in polyethylene bags until use. The carbonization of sugar beet pulp was carried out as;

1- Approximately 30g of dried pulp was placed in a aluminium foliage pillow in cylindrical shape with diameter of 3.5cm and height of 13cm.
2- A few holes were opened on the pillow to allow for removing gaseous components released from sugar beet pulp during heating.
3- These pillows were subjected to heat in an oven at a heating rate of 15°C/min up to final temperatures of 300, 400 and 500°C, respectively, and held at these temperatures for two hours.

Afterwards, the resulting char named as sugar beet pulp carbon (SBPC) was cooled to room temperature. The SBPC samples were ground and sieved to obtain -150μm (100 mesh) fractions. The fractions were stored in a desiccator until use in adsorption experiments.

2.2. Preparation of solution

A stock solution of Cu(II) ions was prepared by dissolving the pure copper wire. To do this, firstly, the copper wire was treated with nitric acid solution to remove oxide films and then dried. One gram portion of dried wire was dissolved in 5ml of concentrated nitric acid in a 1000ml flask and distilled water was added up to the mark. The stock solution was diluted as required to obtain standard and working solutions containing various amount of Cu(II) ions. The solution pH was adjusted to the required value by using 0.1M NaOH and 0.1M HNO_3 solution. All reagents used were of analytical grade.

2.3. Adsorption experiments for Cu(II) ions

All working solutions were prepared in a 50ml glass flask into which distilled water, NaOH or HNO_3 solution and a required amount of stock solution of Cu(II) ions were placed to obtain different initial concentrations and pHs. These solutions were transferred to a conical flask (150ml) containing a certain amount of SBPC. The flasks were capped tightly and shaken at a rate of 300 cycle/min using a flask shaker (Stuart Scientific, SF1 model) equipped with a temperature controlled water bath for contact time varying from 15 to 300min. At the end of predetermined shaking period, aqueous phase were separated by filtration and Cu(II) ions concentration in supernatants were determined by atomic absorption spectrophotometer (Perkin Elmer, 370 model). The removal percentage and the adsorption density, q(mg/g), of Cu(II) ions was calculated by using equations 1 and 2, respectively.

$$\text{Removal (\%)} = (C_o - C)\frac{100}{C_o} \qquad (1)$$

$$q\ (\text{mg/g}) = [(C_o - C) x V]/m \qquad (2)$$

Here, C_o and C are the concentration of the Cu(II) ions in aqueous phase before and after the predetermined contact period, respectively (mg/l); V is the volume of aqueous phase (ml) and m (g) is the amount of SBPC used. All experiments were performed at 25°C in duplicate and average values are presented. The results were found to vary within ±5%.

3. RESULTS AND DISCUSSION

3.1. Effect of solution pH

It is well known that the solution pH is one of the most important variables affecting adsorption yield of metal ions because the surface charge of an adsorbent can be modified by changing the pH of solution. The effect of solution pH on Cu(II) adsorption by SBPC was investigated by varying the pH of Cu(II) solution-SBPC suspensions from 1.5 to 8.4. The results are shown in Figure 1. In all cases, the adsorption of Cu(II) ions did not occur at the pH less than 1.5. With increase in the pH of the solution from 1.5 to 5.5 the adsorption yield increased from zero to 45.3%, 52.4% and 61.0% at 25°C for SBPCs obtained at 300, 400 and 500°C, respectively. At pH values between 5.5 and 8.4, a little increase was observed up to 49.5%, 55.1% and 67.0%. The adsorption capacities of SBPC obtained at higher temperatures are higher than that of low temperature carbonization products. The weight loss increase with temperature due to the removal of volatile substances from sugar beet pulp and this situation causes the carbonization products to have a larger surface area and more suitable adsorption site.

It is often suggested that the adsorption of metal ions at adsorbent-solution interface is not governed by the free metal ion concentration. Free metal ions may be hydrolysed in aqueous solution, giving various hydrolysis products depending upon the pH of solution, represented by:

$$M_{aq}^{z+} + 2nH_2O \Leftrightarrow M(OH)_n^{z-n} + nH_3O^+ \quad (3)$$

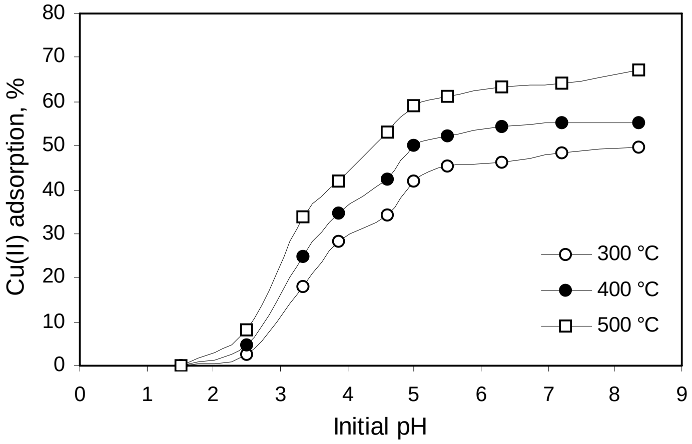

Figure 1. Effect of initial pH on the adsorption Cu(II) ions by SBPCs (Conditions: 50ml 100mg/l Cu(II) solution; 0.25g SBPC; contact time120 min; contact temperature 25°C)

As suggested by Elliott and Huang (1981) various species of copper can be formed as a function of solution pH (Figure 2). As seen from the figure, the hydrolysis extent of Cu(II) ions is unimportant over the pH 6.4. On the other hand, the solubility product for $Cu(OH)_2$ is given as 2.0×10^{-19} (Manahan, 1986), and the value of theoretical pH, which is a measure of starting point for precipitation, is 6.05 for initial concentration of 100mg-Cu(II)/l. For that reason, over the pH 6.05, Cu(II) ions can be precipitated as hydroxide and adsorption of ions is masked by precipitation. At around pH 5.5, there are three species exist; Cu^{2+} and $Cu(OH)_2$ in small quantity and $CuOH^+$ in large quantity (Figure 2). The optimum pH (5.5) where the effective removal take places seems to be related to the concentration of $CuOH^+$ which formed to a large extent in the pH range 4.5 to 6.5. Optimum pH for Cu(II) ions adsorption from aqueous solution by activated carbons from almond shells, olive and peach stone has been reported as 5.0 (Ferro-Garcia *et al.*, 1988). In other study using coirpith carbon, maximum removal was attained in the pH range 4.0 to 5.0 (Namasivayam and Kadirvelu, 1997). It has been reported that the adsorption of Cu(II) by peanut hull carbon increased with increase in pH and reached a maximum value at pH 6.0 (Periasamy and Namasivayam, 1996). Yu *et al.* (2000) have reported that the greatest increase in sorption rate of Cu(II) ions on sawdust was observed in a pH range 4.0 to 8.0. In another study performed using sawdust to remove Cu(II) ions from industrial wastewater, maximum removal percentage was observed at pH 6.0 (Ajmal *et al.*, 1998). These results are in a good agreement with this study, indicating that the optimum adsorption of Cu(II) ions from aqueous solutions took place at pH around 5.5.

Cu(II) ions adsorption mechanisms can be explained on the basis of changes on the carbon surface charge at different pHs. At strong acidic conditions, copper would be predominantly present as Cu^{2+} ion. At these conditions there would be competition between H^+ and Cu^{2+} due to electrostatic repulsion between these cations for adsorptive sites on surface of SBPC, which leads to decrease the yield of adsorption. As the pH increases, the Cu^{2+} and $CuOH^+$ formed by the hydrolysis reaction will replace H^+ from the surface of SBPC, hence, the adsorption yield will increase. As a result, the adsorption extent of Cu(II) is, to a large extent, dependent on the solution pH, active sites on the adsorbent used, and the behaviour of Cu(II) ions in the solution.

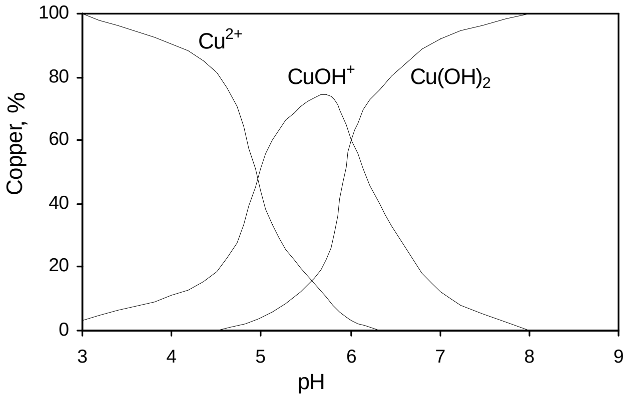

Figure 2. Speciation of Cu(II) as a function of pH (Elliott and Huang, 1981)

3.2. Effect of contact time

Figure 3 illustrates the removal percentage of Cu(II) ions by SBPC obtained at different carbonization temperatures. These experiments were carried out for different contact times with a fixed adsorbent dosage of 5g/l at initial pH 5.5. As seen from the figure, the trend of curve showed that adsorption yield is higher at the beginning for all SBPC samples because of the presence of free adsorption sites. Adsorption yield of the samples obtained at elevated temperatures are higher than those of low carbonization temperature-samples. The removal percentage of Cu(II) ions increased with contact time and attained equilibrium at 120min for SBPC samples. It was observed that 45.3%, 52.4% and 61.0% of the total Cu(II) ions were removed from the solution by using SBPC obtained at 300, 400 and 500°C within equilibrium time of 120min. After equilibration time, there was a small increase in the removal yield.

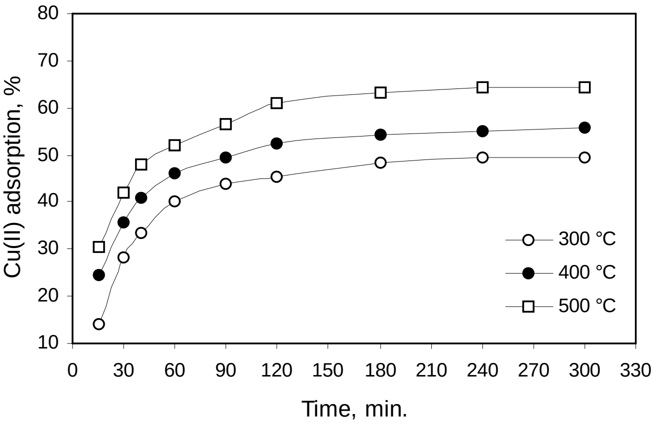

Figure 3. Effect of contact time on the adsorption Cu(II) ions by SBPCs (Conditions: 50ml 100mg/l Cu(II) solution; 0.25g SBPC; initial pH 5.5; contact temperature 25°C)

The equilibrium times of Cu(II) ions for different adsorbents have been reported in a wide range of contact times. For example, Periasamy and Namasivayam (1996) investigated the adsorption of Cu(II) by peanut hull carbon and GAC and reported that the equilibrium times were 120 and 300min, respectively. The equilibrium time for low-rank Turkish coal has been reported to be 20min (Karabulut *et al.*, 2000). Gupta and Ali (2000) have reported that the equilibration time for Cu(II) ions removal was 60min in a study carried out by bagasse fly ash. In a study that carried out by coirpith carbon, equilibrium time was 25min (Namasivayam and Kadirvelu, 1997). The equilibrium time for fruit residues and phosphated fruit residues was 360min (Senthilkumaar *et al.*, 2000). It must be noted that there are several parameters affecting the equilibrium time such as stirring rate of suspension, physical and chemical properties of adsorbent (porosity, surface area, the structure of functional groups, active adsorption sites), the amount of adsorbent and the properties of adsorbate (atomic and ionic radius, polarity and electronic charge). Therefore the equilibrium times for the adsorbent used are different, however, it can be said that SBPC would require intermediate time for equilibrium.

3.3. Effect of SBPC dosage

The effect of SBPC dosage on the removal of Cu(II) ions was studied by varying the dosage from 2 to 20g/l at pH 5.5 for 120min. The adsorption densities, q(mg/g), and removal percentages of Cu(II) ions were plotted versus adsorbent dosage (Figure 4).The adsorption density increased with increasing in the dosage to a maximum value and then declined. 5g SBPC per litre of solution is suitable dosage for this process, to give a maximum adsorption density corresponding to 9.06, 10.48 and 12.20mg/g for SBPCs obtained at 300, 400 and 500°C, respectively. On the other hand, it is apparent that the removal percentage of Cu(II) ions increased with increase in the dosage of SBPC due to the greater availability of the exchangeable site or surface area at higher dosage of the adsorbent. When the dosage of 20g/l were applied for SBPCs obtained at 400 and 500°C, Cu(II) ions in the solution (100mg/l) were completely removed.

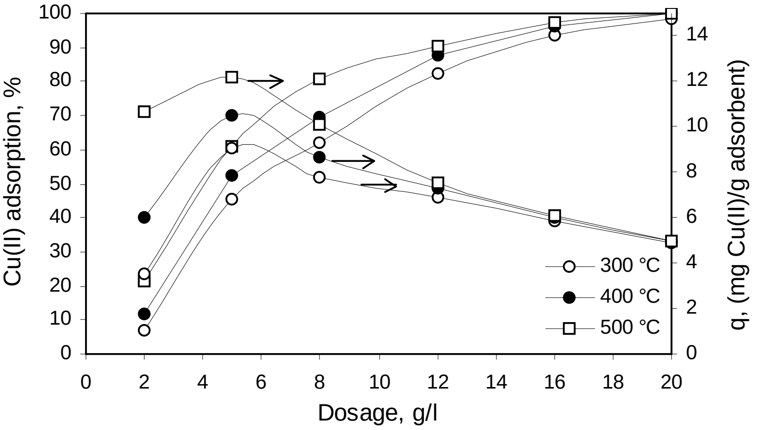

Figure 4. Effect of the dosage on the adsorption Cu(II) ions by SBPCs (Conditions: 50ml 100mg/l Cu(II) solution; contact time 120min; initial pH 5.5; contact temperature 25°C)

3.4. Adsorption isotherms

The equilibrium isotherms for the adsorption of Cu(II) ions on SBPC samples were determined by contacting 0.25g SBPC with 50ml of Cu(II) solution of various concentration (25-400mg/l). SBPC-Cu(II) suspensions were agitated in a constant temperature (25°C) at pH 5.5. Langmuir and Freundlich adsorption models were used to describe the equilibrium between adsorbed Cu(II) ions on the SBPC and Cu(II) ions in solution.

Langmuir isotherm which is valid for monolayer adsorption is applied to adsorption on completely homogenous surface with negligible interaction between adsorbed species (Atkins, 1994). The linear form of the equation can be written as

$$\frac{C_e}{q_e} = \frac{1}{b\,q_{max}} + \frac{C_e}{q_{max}} \tag{4}$$

Where C_e is equilibrium concentration of the adsorbate (mg/l), q_e is the amount adsorbed per unit mass of adsorbent (mg/g), q_{max} is the maximum surface density or adsorption capacity and has same units with q_e. b (l/mg) is a constant related to the affinity of binding sites or bonding energy. q_{max} represents a practical limiting adsorption capacity when the surface of adsorbent is completely covered with adsorbate. When C_e/q_e was plotted versus C_e , straight lines with a slope $1/q_{max}$ and intercepts $1/bq_{max}$ were obtained (Figure 5), indicating that the adsorption Cu(II) ion on SBPC follows the Langmuir model. The Langmuir constants b and q_{max} were calculated and their values for three SBPC samples obtained different temperatures are given in Table 1.

The equilibrium data for Cu(II) ions were also fitted into Freundlich model. This model deals with multilayer adsorption of the substances on the adsorbents. The logarithmic form of this model is given by the following equation.

$$\ln q_e = \ln K_f + \frac{1}{n}\ln C_e \tag{5}$$

Where K_f is the measure of adsorption capacity, n is adsorption intensity and other parameters have been defined as in Langmuir model. A plot of $\ln q_e$ against $\ln C_e$ should be a straight line, the slope and intercept of which correspond to 1/n and $\ln K_f$, respectively. The values of K_f and n were calculated from the slopes and intercepts of the plots in Figure 6 and presented in Table 1.

The data related to the equilibrium fitted the Langmuir model better than Freundlich as seen from the values of regression coefficients are shown in Table 1. The values of q_{max} and b calculated from Langmuir plots were found to be 12.24mg/g and 0.042L/mg for SBPC obtained at 300°C. The values of both q_{max} and b increased with a rise in carbonization temperature, but increase in the values of b is more outstanding compared to those of q_{max}. A comparison of the values of adsorption capacity (q_{max}) of SBPCs with those various adsorbents reported in the literature is given in Table 2. It must be noted that direct comparison of SBPC with the other reported adsorbents is difficult due to different experimental conditions applied in the studies cited. The adsorption capacities of SBPCs are higher than some of the adsorbents reported. But the adsorption capacity of for chitosan (Juang *et al.*, 1999), coirpith carbon (Namasivayam and Kadirvelu, 1997), washed (Reddad *et al.*, 2002) and modified sugar beet pulp (Dronnet *et al.*, 1999), activated carbon from sugar beet pulp (Tanyildizi *et al.*, 1999) and peanut hull carbon (Periasamy and Namasivayam, 1996) are higher than SBPC (Table 2).

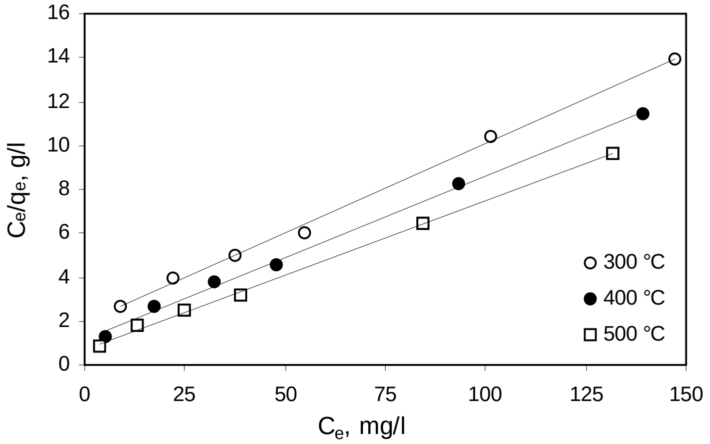

Figure 5. Langmuir plot of Cu(II) ions adsorption by SBPCs (Conditions: contact time 120min; 0.25g SBPC; initial pH 5.5; contact temperature 25°C)

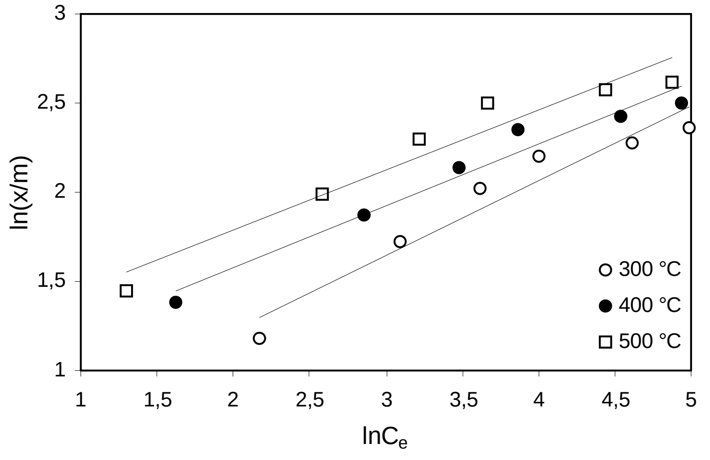

Figure 6. Freundlich plot of Cu(II) ions adsorption by SBPCs (Conditions: contact time 120min; 0.25g SBPC; initial pH 5.5; contact temperature 25°C)

The effect of isotherm shape on whether adsorption is “favourable or unfavourable” has been considered. The essential characteristic of Langmuir isotherm can be expressed in terms of dimensionless constant separation factor or equilibrium parameter defined by Weber and Chakravorti (1974) as follows:

$R_L = 1/(1+bC_o)$ (6)

Where b is Langmuir constant and C_o is initial concentration Cu(II) ions. R_L value indicates the type of isotherm. If R_L value is between 0 and 1, the adsorption process is favourable. The RL values were found to be less than 1 and greater than zero, in the Cu(II) concentration range 25 to 200mg/l for all SPBC samples. These results show that Cu(II) ions adsorption on SBPC is favourable.

Table 1
Langmuir and Freundlich constants related to the adsorption of Cu(II) ions on SBPC obtained at different temperatures

Carbonization temp.	Langmuir constants			Freundlich constants		
(°C)	q_{max} (mg/g)	b (l/mg)	R^2	K_f	n	R^2
300	12.24	0.042	0.998	1.49	2.40	0.934
400	13.44	0.063	0.997	2.42	2.98	0.963
500	14.81	0.092	0.998	3.08	2.99	0.934

Table 2
Cu(II) adsorption capacity of some reported adsorbents

Adsorbent	q_{max} (mg/g)	References
Activated carbon	8.85	Ferro-Garcia *et al.*, 1988
Activated carbon	9.21	Ferro-Garcia *et al.*, 1988
Activated carbon	7.21	Ferro-Garcia *et al.*, 1988
Bagasse fly ash	2.26	Gupta and Ali, 2000
Low-rank Turkish coals	1.62	Karabulut *et al.*, 2000
Coirpith carbon	39.7	Namasivayam and Kadirvelu, 1997
Peanut hull carbon	65.57	Periasamy and Namasivayam,1996
Chitosan	174.8	Juang *et al.*, 1999
Pine bark	9.47	Al-Asheh *et al.*, 2000
Sawdust	1.79	Yu *et al.*, 2000
Washed sugar beet pulp	21.1	Reddad *et al.*, 2002
Modified sugar beet pulp	17.1	Dronnet *et al.*, 1997
Activated carbon	63.69	Tanyildizi *et al.*, 1999
Sphagnum moss peat	5.75, 8.87, 12.44	Ho and McKay, 2000
SBPC (300°C)*	12.24	This study
SBPC (400°C)*	13.44	This study
SBPC (500°C)*	14.81	This study

* Shows carbonization temperature of sugar beet pulp.

The values of K_f, which is a measure of adsorption capacity, increased from 1.49 to 3.07 with rise in carbonization temperature from 300 to 400°C. In all cases, the values of n are higher than 1 and increased with carbonization temperature. The situation n>1 is most common and may be due to a distribution of surface sites or any factor that causes a decrease in adsorbent-adsorbate interaction with increasing surface density (Reed and Matsumoto, 1993). According to McKay *et al.* (1981) the values of n in the range of 2-10 represent good adsorption.

3.5. Adsorption kinetics

In order to determine the adsorption rate constant, pseudo-first order kinetic model of Lagergren was applied to the experimental data. This model was the first rate equation for the adsorption of liquid/solid system based on the solid capacity (Ho and McKay, 1999), and generally is expressed as follows:

$$\frac{dq_t}{dt} = k(q_e - q_t) \tag{7}$$

Where q_e (mg/g) is the amount of adsorbate at equilibrium, q_t is amount of adsorbate at any time t, k (1/min) is the rate constant of pseudo first-order adsorption. Integrating of the equation 7 for the boundary conditions t=0 to t=t and q_t=0 to q_t=q_t, gives:

$$\log(q_e - q_t) = \log(q_e) - \frac{k}{2.303}t \tag{8}$$

Linear plots of $\log(q_e\text{-}q_t)$ versus t show the applicability of the equation 8 for the adsorption of Cu(II) ions by SBPC (Figure 7). It can be seen that the data follow Lagergren kinetic model to great extent, since the correlation coefficients (R^2) were found to be higher than 0.986. k values calculated from the slopes of the plots were 0.042, 0.030 and 0.024L/min for SBPC samples obtained at 300, 400 and 500°C, respectively.

4. CONCLUSIONS

This study indicated that the sugar beet pulp carbon (SBPC) can be used as a an effective adsorbent for the removal of Cu(II) ions from aqueous solutions. The adsorption yield was solution pH dependent and maximum adsorption capacities were found to be 12.24, 13.44 and 14.81mg/g for the adsorbents obtained by carbonising sugar beet pulp at 300, 400 and 500°C, respectively. The maximum adsorption capacities of SBPCs were seen to be relatively higher compared to those of many adsorbents reported in the literature. Cu(II) ions in the solution (100mg/l) were adsorbed by the developed adsorbents nearly up to 100% at a pH of 5.5 with the dosage of 20g/l. Ion exchange may be the dominant mechanism in the adsorption process and $CuOH^+$ ions formed by hydrolysing Cu(II) ions seem to play the most important role. The results related to adsorption isotherms showed that the equilibrium data fitted the Langmuir and Freundlich model. The suitability of the first order kinetic model for the adsorption Cu(II) ions was also examined by using Lagergern's first-order rate expression.

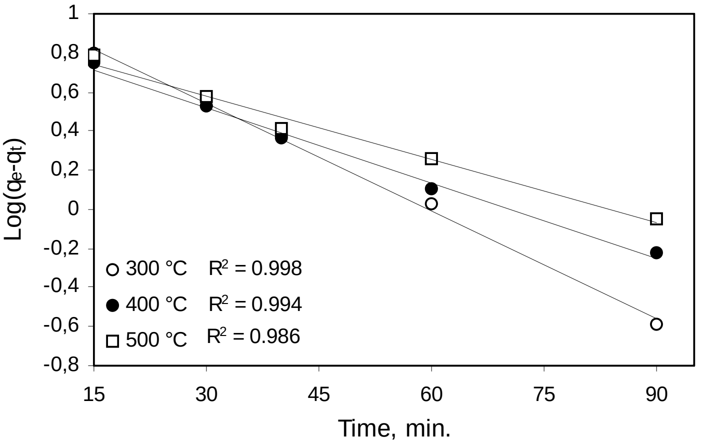

Figure 7. Lagergren plot of Cu(II) ions adsorption by SBPCs (Conditions: 50ml 100mg/l Cu(II) solution; 0.25g SBPC; initial pH 5.5; contact temperature 25°C)

ACKNOWLEDGEMENT

This study was supported by the State Planning Organisation (DPT) under project no 97K120990, Turkey.

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NOMENCLATURE

b : Langmuir constant, l/mg
C : Final concentration of adsorbate in solution, mg/l
C_e : Equilibrium concentration of adsorbate in solution, mg/l
C_o : İnitial concentration of adsorbate in solution, mg/l
k : Adsorption rate consatant of pseudo first-order, 1/min
K_f : Freundlich constant
M : Amount of adsorbent, g
n : Freundlich constant
R : Regression cofficient
R_L : Dimensionless constan separation factor
t : Time, min
V : Volume of solution, l
q_e : Amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium, mg/g
q_{max} : Maximum adsorption capacity, mg/g
q_t : Amount of adsorbate adsorbed per unit mass of adsorbent at any time, mg/g