

## Boric Acid Removal from Water with Alginate Based Beads and Films as Adsorbents

Kübra MOD<sup>1</sup>, Güler HASIRCI<sup>2</sup>, Nilüfer HİLMİOĞLU<sup>3\*</sup>

<sup>1</sup> Kocaeli University, Chemical Engineering Department, Kocaeli, Türkiye

<sup>2</sup> Kocaeli University, Chemical Engineering Department, Kocaeli, Türkiye

<sup>3</sup> Kocaeli University, Chemical Engineering Department, Kocaeli, Türkiye

Kübra MOD ORCID No: 0009-0003-2103-1244

Güler HASIRCI ORCID No: 0000-0001-7435-8118

Nilüfer HİLMİOĞLU ORCID No: 0000-0002-2627-8890

\*Corresponding author: niluferh@kocaeli.edu.tr

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### Keywords

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nanotube

**Abstract:** High levels of boron cause harmful effects on humans, animals and plants. While boron is an important auxiliary nutrient for plants, excessive amounts cause toxic effects. The removal of boron in wastewater from chemical processes, industrial processes and agriculture is an important field of study. One of the most widely used methods for boron removal is adsorption method due to its low cost and ease of processing. In this study, adsorption method and sodium alginate microcapsules and sodium alginate-carbon nanotube film adsorbents were used as adsorbents for boric acid removal. In adsorption studies, sodium alginate-carbon nanotube film adsorbents with 42.11% boron removal were more efficient than sodium alginate microcapsules. As a result of kinetic studies, it was found that the study was consistent with the pseudo-first order kinetic model.

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## Adsorban Olarak Aljinat Bazlı Küreler ve Filmler ile Sudan Borik Asit Giderimi

### Anahtar

### Kelimeler

Borik Asit,  
Mikrokapsül,  
Adsorpsiyon,  
Sodyum  
aljinat,  
Film  
adsorbanlar,  
Karbon  
nanotüp

**Öz:** İnsan, hayvan ve bitkiler üzerinde borun yüksek değerlerde olması zararlı etkilere sebep olmaktadır. Bitkiler için bor önemli bir yardımcı besin iken fazla miktarda bulunması toksik etkilere yol açmaktadır. Kimyasal proseslerin, endüstriyel çalışmaların ve tarım sonucunda açığa çıkan atık suların içerisindeki borun giderilmesi önemli çalışma alanlarından biridir. Borun giderilmesi için en çok kullanılan yöntemlerden biri düşük maliyet ve işlem kolaylığı sebebiyle adsorpsiyon yöntemidir. Bu çalışmada, borik asidin giderilmesi için adsorpsiyon yöntemi ve adsorban olarak sodyum aljinat mikrokapsül küreler ve sodyum aljinat-karbon nanotüp film adsorbanlar kullanılmıştır. Adsorpsiyon çalışmalarında sodyum aljinat-karbon nanotüp film adsorbanlarla, % 42.11 giderim ile sodyum aljinat mikrokapsüllerden daha verimli bor giderimi elde edilmiştir. Kinetik çalışmalar sonucunda çalışmanın yalancı birinci derece kinetik model ile uyumlu olduğu bulunmuştur.

## 1. INTRODUCTION

Water problem is one of the biggest problems of our age. The increasing number of population, industrial zones and increasing demands, agriculture and water use are important reasons for the occurrence of water problems. In order to overcome the water problem, studies are being developed on new solutions through natural resources. For example, trying to obtain efficient clean water by separating sea water. While these and similar studies are carried out, metal products such as boron are seen in the

water obtained. In order to remove these metals from water, research is being carried out for studies that are the least harmful to nature and can achieve high efficiency. Scientists should continue these studies with the least damage to natural resources [1]. Many heavy metals are seen in wastewater as a result of chemical processes, especially in industry and agriculture. Heavy metals cause harmful effects when they penetrate into the human body or other living organisms. Boron, one of the heavy metals, is an important product for the health of humans, plants, animals and other living species. However, its high levels have a harmful effect on living things [2]. Boron, which

is a particularly vital supplementary food for plants, turns into a harmful effect in excessive amounts [3]. The World Health Organization (WHO) has announced the limit values for boron and set this value as  $2.4 \text{ mg L}^{-1}$  [4]. Boron has many uses. It has a wide range of uses such as hygiene materials, fire extinguishing products, medical products, personal care products, textiles, ceramics [5]. Boron does not exist alone in nature. It forms borate together with oxygen. Borax, kernite, colemanite, ulexite are borate minerals with high industrial value [6]. Although boron is a mineral found in soil and plants, it is found in high amounts only in certain countries. Turkey is the first among these countries. However, although we have high boron values, we are not ranked first in the world boron production ranking [7]. One of the compound forms of boron is boric acid. Boric acid shows weak acidic properties [8]. Boric acid is the most preferred compound among the usage areas of boron [9]. Boron removal from wastewater constitutes a wide area of study due to its high usage area. Many different techniques are used for the separation of boron from aqueous solutions. Among these methods, chemical precipitation, reverse osmosis, ion exchange and ultrafiltration are inadequate for low-value concentrations and also show high-cost characteristics. However, adsorption method, which is one of the other methods, is used more than other methods due to its ease of use and low cost [4]. Adsorption technique is the binding of multiple molecules with the ability to dissolve in different media such as gas or liquid to a solid substrate. The solid ground is the adsorbent and the substance adhering to the ground is the adsorbate. The reverse of the adsorption process is called desorption process. There are many parameters to be considered in the adsorption process such as concentration, pH, temperature, time. But one of the most important parameters is the type of adsorbent selected. Selecting the most suitable adsorbent is important to increase the efficiency of the study. When selecting the adsorbent, attention should be paid to the fact that adsorption should be at high values, it should be affordable and adsorption should take place quickly [9]. In this study, experiments were carried out separate adsorbents were used: sodium alginate beads and sodium alginate-carbon nanotube films. The adsorption study using sodium alginate microcapsules was aimed to be made more efficient by adding carbon nanotube.

Alginate obtained from algae contains mannuronic acid and guluronic acid. The reason for using sodium alginate microcapsules is that sodium alginate is a biopolymer and an environmentally friendly adsorbent. Studies in the field of nanotechnology also contribute to improving the properties of materials. Buckyballs" (spherical molecules) are formed by bonding carbon atoms in clusters of 60. When a certain number of cobalt or nickel atoms are added to the buckyballs, their form changes and they become chemically stable "nanotubes" with a wall thickness of one nanometer. Carbon nanotubes (CNTs) are characterized by their weightlessness, high stretch coefficient and being the most resistant fiber. Many studies have been carried out to obtain carbon nanotube synthesis and as a result, certain methods have emerged. These methods are carbon nanotubes obtained by synthesizing from solid carbon and gaseous carbon. Apart from these methods, other synthesis methods are being

tried for different carbon nanotubes to be obtained with different environments [10]. The carbon nanotube used in the study was preferred as an adsorbent due to the free available void spaces on its surface, its use in the literature as a good adsorbent in aqueous solutions and its low cost [11].

## 2. MATERIAL AND METHOD

### 2.1. Material

The solutions used for boron adsorption experiments in our study were obtained with boric acid ( $\text{H}_3\text{BO}_3$ ) distilled water. Sodium alginate and carbon nanotubes were used to obtain adsorbents.  $\text{CaCl}_2$  was used as cross-linker of alginate. The pH of the solutions we used during the experiment was adjusted with the help of NaOH and HCl. NaOH, D-mannitol, a few drops of phenolphthalein were added to determine the boric acid concentration by titration. Figure 1. shows the titrator used for boron adsorption and the prepared solutions.



**Figure 1.** Titrator used for the titration process in the study, solution prepared for boron determination (left), titrated boron solution (right).

### 2.2. Preparation of Alginate Microcapsule Beads

Sodium alginate was weighed and added to 100 ml, resulting in a 5% solution. The sodium alginate solution was dropped into the 0.05 M  $\text{CaCl}_2$  solution for crosslinking. It was left in a magnetic stirrer overnight to complete the crosslinking reaction. After the mixing of the microcapsules was completed, they were passed through distilled water to remove  $\text{CaCl}_2$ . The resulting sodium alginate microcapsules were allowed to dry at room temperature. Figure 2. shows the final form of sodium alginate microcapsules.



Figure 2. Sodium alginate microcapsule beads

### 2.3. Preparation Of Sodium Alginate-Carbon Nanotube Films

To make boric acid adsorption more efficient, sodium alginate was mixed with carbon nanotubes. During the construction of sodium alginate-carbon nanotube film adsorbents, 0,7 g carbon nanotubes were added to 2% sodium alginate solution of the amount of sodium. The sodium alginate-carbon nanotube mixture was kept in a magnetic stirrer overnight. The resulting mixed solution was dropped into 0,01 M  $\text{CaCl}_2$  for crosslinking. The sodium alginate-carbon nanotube film adsorbents were washed with distilled water to remove  $\text{CaCl}_2$  and allowed to dry at an oven for a short time. Figure 3. shows the final sodium alginate-carbon nanotube film adsorbents.



Figure 3. Sodium alginate-carbon nanotube film particles

### 2.4. Boric Acid Determination

Since it is weakly acidic in boric acid solutions, it cannot be directly titrated with base solution. For this reason, mannitol or glycerin should be added to the solution before titration to convert boric acid into a strong monovalent acid form [13]. In our study, a stock solution was prepared for boric acid and solutions at other concentrations were obtained by diluting the stock solution. The stock solution of boron was obtained by

measuring 5.730 g of boric acid ( $\text{H}_3\text{BO}_3$ ) and completing it with pure water. Stock solution concentration is  $1000 \text{ mg L}^{-1}$ . Boric acid concentration was titrated with 0.1 N NaOH by adding 1 mL boric acid solution, D-mannitol and 1-2 drops of phenolphthalein indicator until a color change occurred.

### 2.5. pH Effect on Boric Acid Adsorption

HCl and NaOH solutions were used to adjust the pH value of boric acid solution in adsorption studies. One of the most important parameters for boric acid solution to provide more efficient results is the pH value. Boron ions have the ability to transform into different ionic structures at various pH values. Boron removal is due to the pH-controllable tetrahydroxyborate and  $\text{B}(\text{OH})_3$  structures of boric acid.  $\text{B}(\text{OH})_3$  is rarely observed as  $\text{OH}^-$  and tetrahydroxyborate at low pH values. Therefore, for low pH, boron removal occurs at lower trace fractions due to the lower affinity of  $\text{B}(\text{OH})_3$  [12].

### 2.6. Boron Removal in the Literature

Table 1. Literature studies on boron removal.

ADSORBENT	WORKING CONDITIONS	YIELD	REFERENCE
EGGSHELL MEMBRANE (ESM)	For adsorption results, values in the range of Ph 2-10 were analyzed. Adsorption reached high values at 35°C. Modified eggshell membrane (MESM) was also used as an adsorbent in the study. Concentration increase was seen as an important parameter.	According to the adsorption results, the highest value is 96.96% at pH 8.	[1]
ALGINATE BEADS	The adsorption study was carried out at the lowest pH 3 and the highest pH 12, and at temperatures of 20 °C and 35 °C.	50% boron adsorption was obtained.	[14]
ZIF-67	The experiment was carried out at pH 4 and temperature 25 °C, ZIF-67, boron concentration value is 0.5 mol L <sup>-1</sup> .	As a result of the study, a high value of 579.80 mg g <sup>-1</sup> was reached.	[15]

ZIF-8	Adsorption study was carried out at 45°C, lowest pH 2 and highest pH 12.	191 mg g <sup>-1</sup> is the highest value found in the study.	[16]
3,4-DIHYDROXY BENZALDEHYDE-MODIFIED CHITOSAN	The boric acid concentration used in the study was 400 mg L <sup>-1</sup> , pH 9.0 for DMAK and pH 5.0 for MEK. It was observed that the adsorption capacity increased with temperature and decreased with increasing temperature for MEK.	The adsorption capacity of the modified chitosan was obtained as 36.77 mg g <sup>-1</sup> . This result is about 4 times higher than the boron adsorption capacity of unmodified chitosan (9.3 mg g <sup>-1</sup> ).	[17]
ACTIVATED CARBON	The most efficient adsorption was observed at pH 8.5. For the experiments, 0.5 g adsorbent amount was used, 30 mg B (dm <sup>3</sup> ) <sup>-1</sup> boron concentration was carried out with 0.025 or 0.05 dm <sup>3</sup> boron solution.	Activated carbon modified with Filtrasorb 400 (F400) has a high adsorption capacity with 51.3% removal efficiency and 0.319 mg g <sup>-1</sup> adsorption capacity.	[18]
CARBON NANOTUBE IMPROVED WITH TARTARIC ACID	The highest values were obtained at pH 6. The ideal adsorbent amount was determined as 0.4 g L <sup>-1</sup> . Adsorption studies were performed at concentrations of 20 mg L <sup>-1</sup> .	As a result of the study, an adsorption capacity of 1.97 mg g <sup>-1</sup> was obtained.	[19]

### 2.7. Adsorption Studies for Sodium Alginate Beads and Sodium Alginate-Carbon Nanotube Film Adsorbents

Adsorption studies were carried out with two different adsorbent microcapsules obtained with 2% sodium alginate adsorbent microcapsules and 10% carbon nanotubes added to 2% sodium alginate.

In the adsorption studies for boric acid removal with the obtained adsorbent microcapsules, Equation 1 (q) was used to calculate the adsorption percentage and Equation 2 was used to obtain the adsorption capacity [9].

$$q_e = ((C_o - C_e)/m) \times V \quad (1)$$

C<sub>o</sub> (mg L<sup>-1</sup>) indicates the initial concentration for boric acid, C<sub>e</sub> (mg L<sup>-1</sup>) indicates the equilibrium concentration, q (mg g<sup>-1</sup>) indicates the adsorption capacity, m (g) indicates the amount of adsorbent and V (L) indicates the solution volume.

Adsorption removal % was calculated from Equation 2 [9].

$$(\%) = ((C_o - C_e)/C_o) \times 100 \quad (2)$$

### 2.8. Adsorption Kinetic Studies for Sodium Alginate Beads and Sodium Alginate-Carbon Nanotube Film Adsorbents

Kinetic models were calculated for sodium alginate microcapsules and sodium alginate-carbon nanotube film adsorbents. Calculations for the pseudo-first-order kinetic model were obtained with the equation used by Lagergren in Equation 3., and the pseudo-second-order kinetic model was obtained with the equation shown in Equation 4 [9].

$$\log(q_e - q_t) = \log(q_e) - (k_1/2.303)t \quad (3)$$

q<sub>e</sub>: Amount of adsorbed substance per gram of adsorbent at equilibrium (mg g<sup>-1</sup>), q<sub>t</sub>: Amount of adsorbed substance per gram of adsorbent at any time t (mg g<sup>-1</sup>), t: Time (h), k<sub>1</sub>: pseudo-first-order kinetic constant (h<sup>-1</sup>).

The pseudo-quadratic kinetic equation is calculated using equation 4 [9].

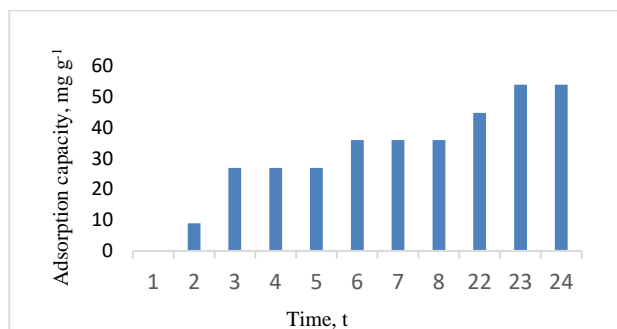
$$t/q_t = 1/(k_2 q_e^2) + t/q_e \quad (4)$$

q<sub>e</sub>: Adsorption capacity at equilibrium (mg g<sup>-1</sup>), q<sub>t</sub>: Adsorption capacity at time (mg g<sup>-1</sup>), k<sub>2</sub>: Pseudo second order kinetic constant (g.mg<sup>-1</sup> h<sup>-1</sup>), t: Time (h).

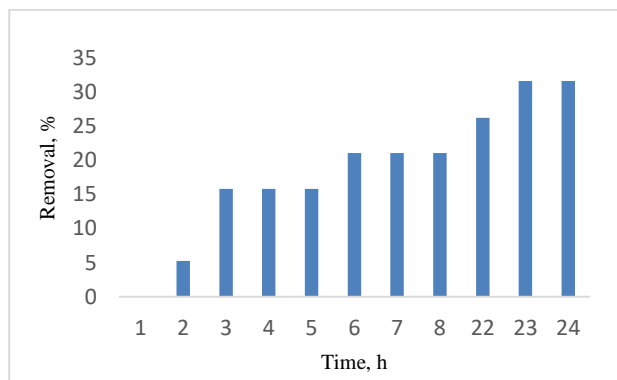
## 3. RESULTS

### 3.1. Adsorption Results for Sodium Alginate Microcapsule Beads

In line with the results, as seen in Figure 4., the highest efficient adsorption capacity q was obtained as 54 mg g<sup>-1</sup> at the 24th hour. According to the data obtained as seen in Figure 5., 31.58% removal was calculated in the adsorption using sodium alginate microcapsules. Looking at Figure 4., it can be seen that adsorption did not occur in the first hour, but adsorption occurred from the 2nd hour. The highest expenditure was observed in the 23rd and 24th hours.



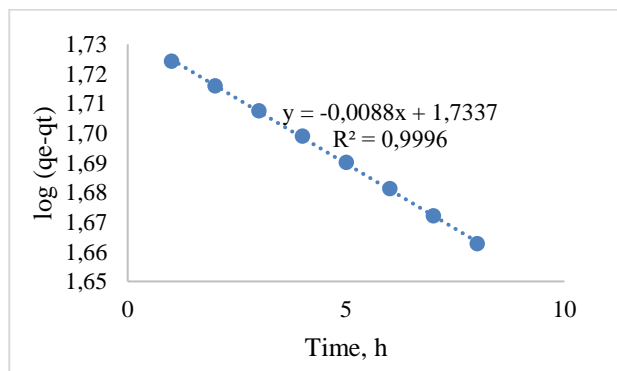
**Figure 4.** Adsorption capacity of sodium alginate microcapsules (experimental conditions: initial boron concentration: 1000 mg/L, adsorbent dose: 0.36 g pH: 9.66).



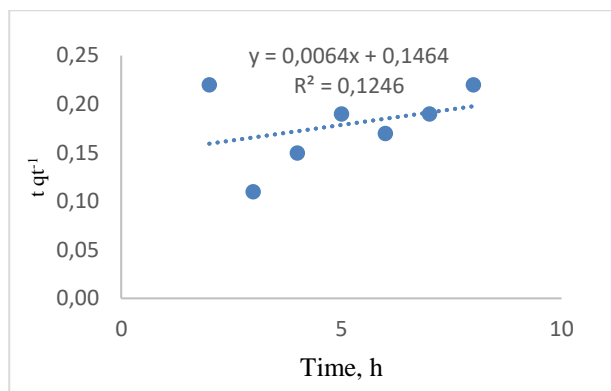
**Figure 5.** Boron removal % of sodium alginate microcapsules (experimental conditions: initial boron concentration: 1000 mg/L, adsorbent dose: 0.36 g pH: 9.66).

### 3.2. Adsorption Kinetics for Sodium Alginate Microcapsule Beads

The relationship of adsorption study with kinetic models is discussed. The graphs of the pseudo-first-order kinetic model are given in Figure 6., and the graphs of the pseudo-second-order model are given in Figure 7. It can be seen that the  $R^2$  value of the pseudo-first order model is higher. For this reason, sodium alginate beads were found to be more suitable for the pseudo-first-order kinetic model among the two models.



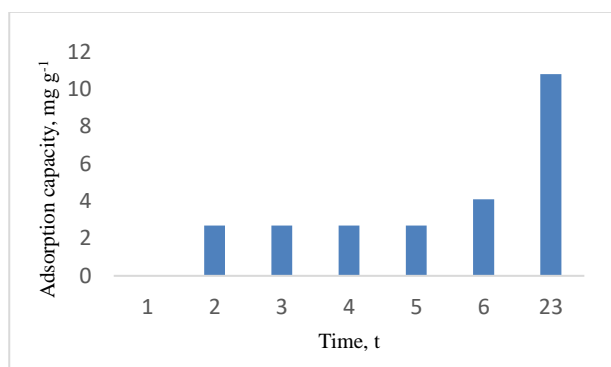
**Figure 6.** Pseudo-first-order kinetic model for sodium alginate microcapsule beads. Experimental conditions: 1000 mg L<sup>-1</sup> concentration, 0.36 g adsorbent dosage, pH 9.66.



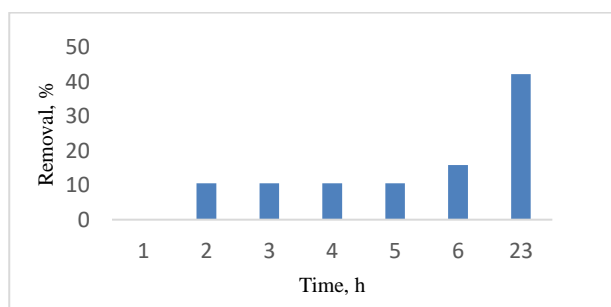
**Figure 7.** Pseudo- second order kinetic model for sodium alginate beads. Experimental conditions: 1000 mg L<sup>-1</sup> concentration, 0.36 g adsorbent dosage, pH 9.66.

### 3.3. Adsorption Results of Sodium Alginate-Carbon Nanotube Film Adsorbents

As a result of the studies, it was observed that sodium alginate-carbon nanotube films reached high adsorption values. Figure 8. shows the results of adsorption capacity for sodium alginate-carbon nanotube films, and Figure 9. shows the adsorption percentage obtained. It was observed that adsorption progressed slowly in the first hours but reached the maximum value at the 23rd hour. A more effective adsorption result was obtained compared to adsorption with sodium alginate microcapsule beads.



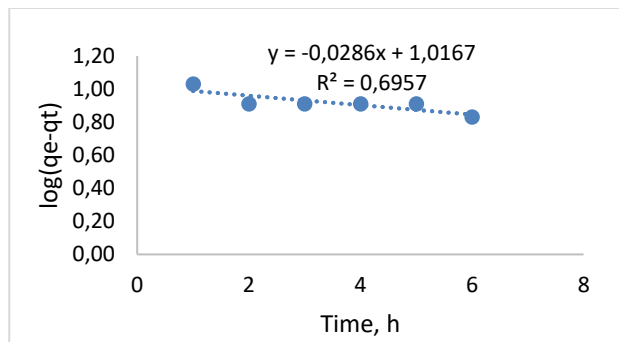
**Figure 8.** Adsorption capacity of sodium alginate-carbon nanotube film adsorbents (experimental conditions: initial boron concentration: 1000 mg/L, adsorbent dose: 0.36 g pH: 9.66 carbon nanotube amount: 0.7 g).



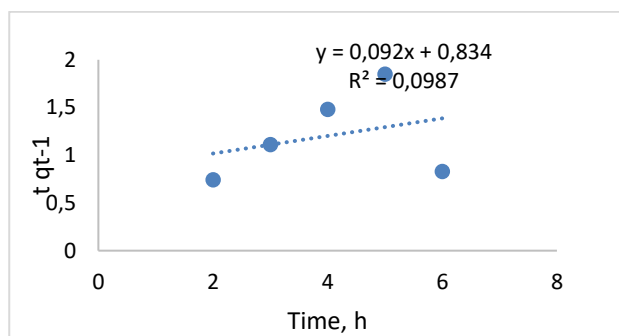
**Figure 9.** Boron removal % of sodium alginate-carbon nanotube film adsorbents (experimental conditions: initial boron concentration: 1000 mg/L, adsorbent dose: 0.36 g pH: 9.66 carbon nanotube amount: 0.7 g).

### 3.4. Adsorption Kinetics for Sodium Alginate-Carbon Nanotube Film Adsorbents

The fit of sodium alginate-carbon nanotube microcapsules with boric acid adsorption kinetic models was examined. Looking at Figure 10. and Figure 11. for the pseudo-models, it was observed that the adsorption kinetics of sodium alginate-carbon nanotube films were in agreement with the pseudo-first order kinetic model, because the pseudo-first order model that has a high R<sup>2</sup> value.



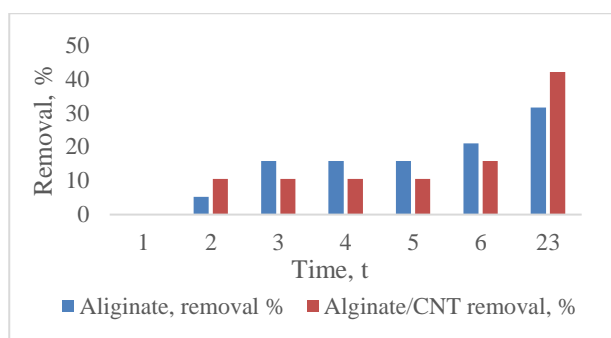
**Figure 10.** Pseudo-first-order kinetic model for sodium alginate /CNT film adsorbents. Experimental conditions: 1000 mg L<sup>-1</sup> concentration, 0.36 g adsorbent dosage, pH 9.66, carbon nanotube amount: 0.7 g.



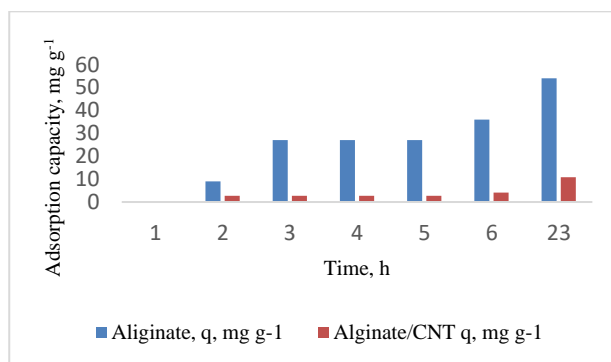
**Figure 11.** Pseudo-second-order kinetic model for sodium alginate /CNT film adsorbents. Experimental conditions: 1000 mg L<sup>-1</sup> concentration, 0.36 g adsorbent dosage, pH 9.66, carbon nanotube amount: 0.7 g.

### 3.5. Comparison Of Sodium Alginate Beads And Sodium Alginate-Carbon Nanotube Films

The adsorption study showed that sodium alginate-carbon nanotube films reached higher values than sodium alginate microcapsules. As can be seen in Figure 12., 42% removal was achieved in 23 hours in adsorption with sodium alginate-carbon nanotube films. The chemical, thermal and moisture stability and large pore structures of carbon nanotube adsorbents are effective in providing efficient results. Figure 13. shows that sodium alginate microcapsules have higher q values than sodium alginate-carbon nanotube microcapsules. The reason for this is that the sodium alginate content in sodium alginate microcapsules is 5% and the sodium alginate content in sodium alginate-carbon nanotube films is 2% [11]. As a result of this study, all the results obtained with sodium alginate beads and sodium alginate-carbon nanotube films adsorbent are shown in Table 2.



**Figure 12.** Comparison of adsorption removal % of sodium alginate microcapsules and sodium alginate-carbon nanotube film adsorbents (experimental conditions: initial boron concentration: 1000 mg/L, adsorbent dose: 0.36 g pH: 9.66 carbon nanotube amount: 0.7 g).



**Figure 13.** Comparison of adsorption capacities of sodium alginate microcapsules and sodium alginate-carbon nanotube film adsorbents (experimental conditions: initial boron concentration: 1000 mg/L, adsorbent dose: 0.36 g pH: 9.66 carbon nanotube amount: 0.7 g).

**Table 2.** Adsorption results for sodium alginate microcapsule beads and sodium alginate-carbon nanotube film adsorbents.

	Adsorption Removal (%)	Adsorption Capacity (mg/g)	Pseudo first order kinetic model data
<b>Sodium Alginate Microcapsule Beads</b>	31.58	54	$k_1 : 0,02026 \text{ h}^{-1}$ $R^2 : 0,9996$ $q_{e \text{ theoretical}} : 54,16$ $q_{e \text{ experimental}} : 54$
<b>Sodium Alginate-Carbon Nanotube Film Adsorbents</b>	42.11	10.8	$k_1 : 0,0658 \text{ h}^{-1}$ $R^2 : 0,6957$ $q_{e \text{ theoretical}} : 10,39$ $q_{e \text{ experimental}} : 10,8$

#### 4. DISCUSSION AND CONCLUSION

This study is quite different from our previous adsorption studies [20, 21]. It is also seen in the literature that boron adsorption that has the low removal value is a difficult type of adsorption that takes a very long time. In our study, boric acid removal from water was studied with the help of two adsorbents. Sodium alginate microcapsule beads and sodium alginate-carbon nanotube film adsorbents were used to remove boric acid from water. As a result of the sodium alginate microcapsules and sodium alginate-carbon nanotube film adsorbents used in the study, more efficient results were obtained with adsorption studies with the adsorbent obtained by adding carbon nanotube. Maximum adsorption efficiency of 42% and adsorption capacity of 10.8 mg g<sup>-1</sup> were obtained using sodium alginate-carbon nanotube film adsorbents. As a result of the kinetic studies for sodium alginate-carbon nanotube film adsorbents, the pseudo-first order kinetic model was found to be appropriate. The importance of ambient conditions for boric acid removal from water has drawn attention. It was concluded that higher pH values have better absorption results in boron removal and as the pH value of the boric acid solution increases, boron removal increases. In conclusion, compared to the studies in the literature, pH value is an important parameter for boron removal and the percentage of boron removal obtained by using sodium alginate-carbon nanotube film adsorbent is similar to the results in the literature. Sodium alginate and carbon nanotube are suitable adsorbent candidates for the removal of boron from aqueous media as they are environmentally friendly materials since alginate is a biopolymer and carbon nanotube provides high adsorption values.

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