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Қ. И. Сәтпаев атындағы Қазақ ұлттық техникалық зерттеу университеті

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ГЕОЛОГИЯ ЖӘНЕ ТЕХНИКАЛЫҚ ҒЫЛЫМДАР СЕРИЯСЫ

♦

СЕРИЯ ГЕОЛОГИИ И ТЕХНИЧЕСКИХ НАУК

♦

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INTERPHASE DISTRIBUTION OF BORIC ACID BETWEEN AQUEOUS SOLUTIONS AND MODIFIED CELLULOSE

Abstract. Regarding the need for processing of hydromineral raw materials, many researchers have an increased focus on core and applied problems of producing organic sorbents based on natural materials, in particular, different kinds of cellulose modified by mercerization methods (treatment with alkali solutions) and chemical change of functional groups.

The research addresses the interfacial distribution of boric acid on a mercerized, then chemically modified crushed kernel of common apricot, Prunus armeniaca (apricot).

It was found that the sorbent material has selective properties for boric acid, extracting it from the aqueous phase and concentrating H_3BO_3 in the solid phase of the sorbent (modified cellulose). On the basis of carried out elemental chemical and mass spectrometric analyzes, along with IR spectrum of sorbents, the structure of sorbents was identified, and a mechanism for the absorption of boric acid by the phase of modified cellulose (CM) was proposed.

Keywords: boric acid; modification; interphase distribution.

Nomenclature of user. In this work rational nomenclature for naming organic compounds was applied. The extra-system units (% and the amount of the solute in a liter of solution) were used to specify the concentration and the rate of extraction. Abbreviations were spelled out in full at their first occurrence in the text.

Introduction. Spectrophotometric analysis and acid-base titration in the presence of polyhydric alcohols of mannitol, xylitol, sorbitol, etc. were applied in order to analyze the amount of boron (the relative error of the analysis is ±3.5%) [1-3].

The literature describes the use of cellulose and polyhydric alcohols as components for the synthesis of various sorbents. For example, the works [4-6] describe methods for preparing composite sorbents using cellulose fibrillated fibers together with compounds of iron and calcium to extract inorganic ions from aqueous solutions, but do not consider the extraction of boric acid.

In the invention [7], it is proposed to obtain an effective sorbent for wastewater treatment from metal ions by immobilizing finely dispersed $MgCO_3$ and $Mg(OH)_2$ onto fibrillated cellulose fibers.

In the patent of the Russian Federation No. 2528696 [8] in order to extract silver and phosphorus it is proposed to obtain a sorbent by treating fibrillated cellulose fibers in a salt solution of zinc and sodium sulfide.

According to the patent of the Russian Federation No. 2520457 [9] a composite sorbent consisting of polyvinyl alcohol and nanophasic oxyhydroxide separated from waste from groundwater deironing stations is used to purify aqueous environment from arsenic.

In the work [10], carboxymethylcellulose (CMC) containing layered double hydroxides was synthesized by the ion exchange method [10]. Experiments have shown that on such CMC, the adsorption of boron increases with increasing contact time, boron concentration and pH.

A study of the removal of boron from simulated and real water systems in the northern part of Chile using ultrafiltration reinforced with a polymer in water, for example, polyglycidyl methacrylate-N-methyl-D-glucamines, which was used to form complexes with boron, is presented in the work [11]. The retention capacity of the polymer membrane was maintained at a level of 2-4 mg per gram of polymer.

For producing adsorbents obtained from natural polymer [12] two forms (powder and fiber) of cellulose derivatives and N-methylglucamine [12] were synthesized. The sorption capacity of the cellulose derivatives by boron was the same as that of commercially available polystyrene resin with N-methyl-glucamine type groups. It was found that cellulose derivatives are superior to a polystyrene resin as the adsorbent for boron (III) in the processing of large amounts of wastewater.

It should be noted that the general shortage of sorbents obtained using glucomine is due to a significant biodegradation of glucomine, since the latter is a nutrient for bacteria.

Thus, there is no information in the literature on neither on chemical modification of mercerized cellulose, by "sewing" of chlorinated derivatives of polyhydric alcohols to its polymeric carbon skeleton (matrix), nor the structure and sorption properties of these compounds. Nevertheless, the interest of researchers in the study of cellulose derivatives as sorbents remains [3].

The purpose of this research was to study the redistribution of the initial boron content in the form of H_3BO_3 between the phases of the aqueous solution and the sorbent in time, on chemically modified cellulose (CM).

In order to achieve this goal, the following tasks were set:

– chemical processing of cellulose in order to improve its operational, physical and chemical properties;

– establishment of the mechanism of the sorption process on (CM);

– examination of regularities of interphase distribution of boron in the solution-solid phase system for the development of practical recommendations.

Experimental part. To improve the capacity of the CM sorbent, it was additionally modified by chemically "sewn" molecules of hexahydrolic alcohol (sorbitol). The synthesis was carried out in two stages.

The first stage is the synthesis of 1-chloro or 1.6-dichlorosorbite. Six-atom alcohol-sorbitol is treated with hydrogen chloride at 95 °C in toluene during 6 hours.

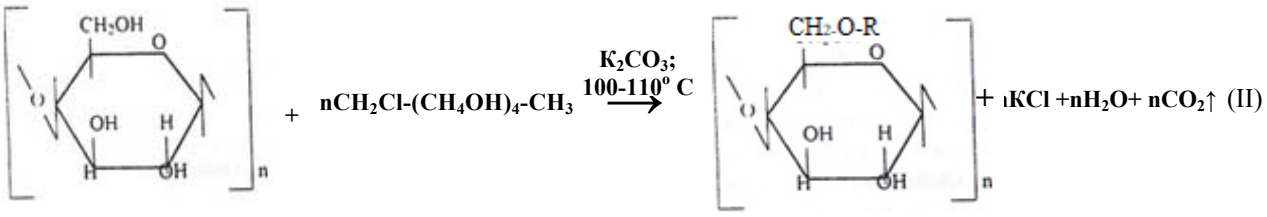
$$2CH_2OH\text{-}(CHOH)_4\text{-}CH_2OH + 3HCl \xrightarrow[\text{toluene}]{95^\circ C} Cl\text{-}CH_2\text{-}(CHOH)_4\text{-}CH_2Cl + 2H_2O \quad (I)$$

$$Cl\text{-}CH_2\text{-}(CHOH)_4\text{-}CH_2OH + H_2O$$

Chlorination in reaction (I) proceeds by the mechanism of nucleophilic substitution. The reaction product is treated with a solution of sodium carbonate until neutral, and then washed with water. After separation of the aqueous layer, the mixture of the chlorine derivatives of sorbitol is purified by distillation.

The chlorinating agent – hydrochloric acid was prepared by the known reaction of a mixture of sulfuric acid and sodium chloride when heated.

II stage – chlorinated sorbitol is "sewn" chemically to mercerized cellulose at 100-110 °C by 10% solution of K_2CO_3 during 5 hours:



where R is the residue of a polyhydric alcohol "sewn" to the cellulose monomer.

The reaction product is filtered off, washed with water, alcohol and again with water until neutral, then it is brought to an air-dry state. The composition of the substance was determined by independent IR spectroscopy, elemental chemical and mass spectrometric analyzes. A chromatography-mass spectrometer Clarus SQ 8 was used for this work.

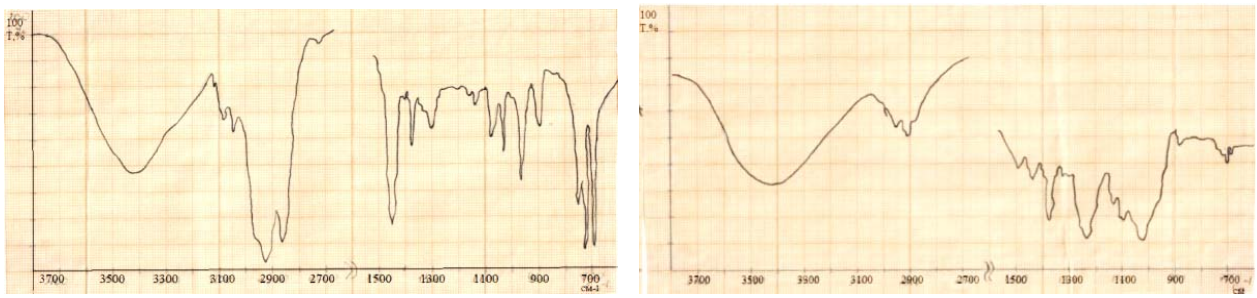
IR spectra of absorption of reaction products are recorded on an IR-20B spectrophotometer in potassium bromide tablets in the range of 600-3800 cm^{-1}.

The percentage of carbon and hydrogen was equal: C - 78.86%, H - 9.66%. According to these data, the calculated ratio of carbon to hydrogen atoms is 1: 1.41 and it practically coincides with their theoretical ratio in a monomer.

The calculation was made using the well-known method for determining the formula of a chemical compound based on its percentage and element composition:

$$C: H = 78.85 / 12.0: 9.65 / 1.0 = 6.57: 9.65 = 1: 1.47$$

In figures 1 and 2, IR spectra of chlorinated sorbitol and modified cellulose are presented for comparison.



a) b)

Figure 1 – IR spectra of a) chlorinated sorbitol, b) modified cellulose

In the IR spectra in the range of 3400-3200 cm^{-1}, there is an intense wide band of valence vibrations ὺ that arise when intermolecular hydrogen bonds-polyassociates are formed.

In the spectra of chlorinated sorbitol, based on the significantly increasing of the intensity of the vibrational bands with the participation of the bond (C-Cl), the substitution of two primary (OH) groups for chlorine atoms can be suggested.

Sewing chlorinated sorbitol proceeds quite fully, which is proved by the minimization of the band of stretching vibrations $ὺ_{Cl}$в in the range of 700 cm^{-1}.

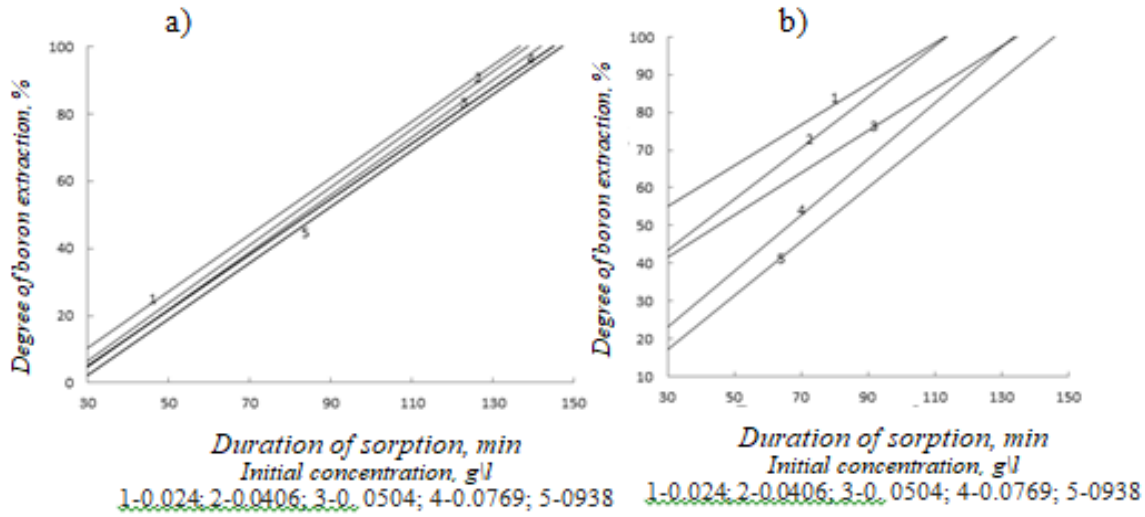


Figure 2 – The influence of time on the amount of boron sorption
a) unmodified cellulose b) modified cellulose

In the range of 970-900 cm^{-1}, average low-density absorption bands characteristic of deformation vibrations of methane groups (CH) are observed.

In the 750-700 cm^{-1} range, intense narrow bands characteristic of the bond (C-CI) appear both in the mixture of the chlorinated derivatives of sorbitol and in the finished product.

Results and discussions. The extraction of boron from water in both types of CM is studied depending on the initial boron concentration, contact time and phase ratio (S: L).

Under static conditions, in a state of equilibrium on a modified CM the degree of extraction reaches 95-97%. The modified CM is more efficient compared to the unmodified one. The sorption capacity of the CM reaches more than 6mg per gram of sorbent, which is at the level of the best existing boron selective ion exchangers. In order to obtain more information, the data of the experiments were processed using the program Excel Microsoft (figures 3, 4). Figures contain a quantitative characteristic of the influence of time and boron concentration on the degree of its sorption.

The regression equations for the dependence of the degree of boron extraction on the time factor on unmodified and modified cellulose at different initial concentrations of boron in the solution were approximated by linear dependence for computer processing (table).

Dependence of the degree of boron extraction on the initial concentration

Unmodified cellulose			Modified cellulose		
initial concentration of boron, g/l	y= ax+b type of equations	coefficient of approximation R^2 (correlation)	initial concentration of boron, g/l	y= ax+b type of equations	coefficient of approximation R^2 (correlation)
0.024	y = 0.8433x – 15.08	0.8845	0.024	y = 0.5437x + 38.7	0.8643
0.0406	y = 0.862x – 19.3	0.8668	0.0406	y = 0.678x + 23.2	0.9018
0.0504	y = 0.853x – 20.87	0.8524	0.0504	y = 0.5597x + 24.91	0.8884
0.0769	y = 0.853x – 20.87	0.8524	0.0769	y = 0.7453x + 0.62	0.8804
0.0938	y = 0.8363x – 22.82	0.8485	0.0938	y = 0.7173x – 4.48	0.8785
Note: a – tangent of the slope of the line to the axis of the abscissa, b – the segment cut off by the straight line along the ordinate axis.					

It can be seen from the table that the coefficient of approximation achieves values greater than 0.85, which indicates the reliability of the results obtained.

Figure 3 shows the information obtained by calculation, describing the influence of the time factor on the amount of boron sorption on two types of cellulose.

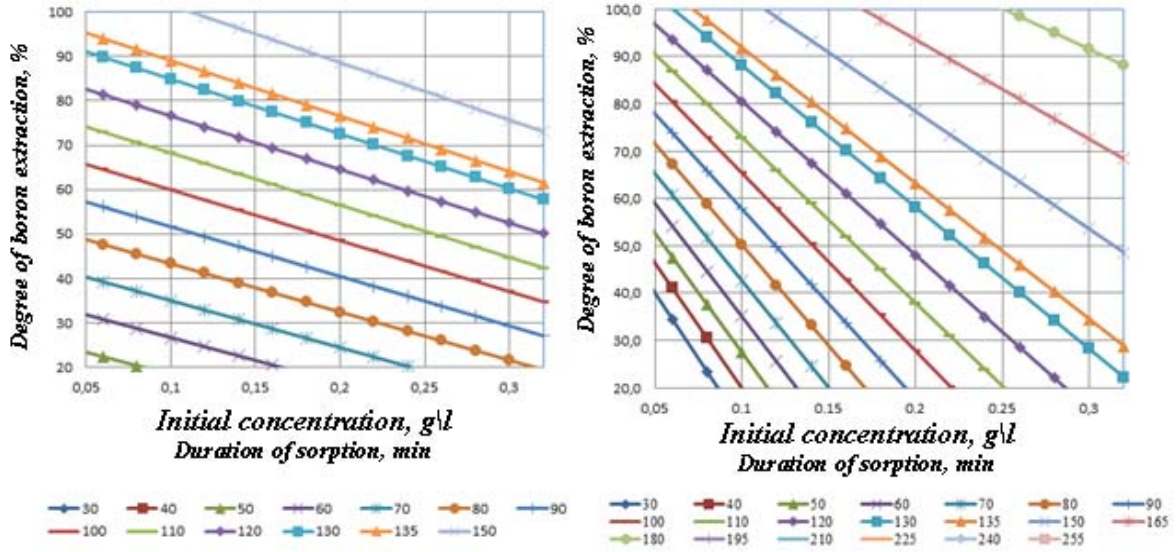


Figure 3 – The diagram of the dependence of boron sorption on the initial concentration and time of sorption on a) unmodified cellulose, b) modified cellulose

It can be seen from the graph (figure 3) that as the time and concentration increase, the degree of the extraction rate of H_3BO_3 decreases. This is due to the gradual saturation of the sorbent phase with boron, which leads to an increase in diffusion difficulties in the transportation of H_3BO_3 molecules in nano- and micro-dimensional pores of sorbents.

A sufficiently complete extraction occurs in all cases with a sorption time of more than 130 minutes, and the modified sorbent is much more efficient than the unmodified one.

Based on the Microsoft Excel program calculation a combined empirical equation is obtained, that relates the dependence of the influence of the initial boron concentration and sorption time on the extraction value:

$$Y = (0.86\text{-}0.3 \cdot X) \cdot \tau\text{-}85.2 \cdot X + 14.7 \quad (1)$$

where: Y is the boron sorption, (% of the initial content), X is the initial concentration in the solution, g / l; τ is the time of the sorption process, min.

In a graphical form, this dependence is shown in Figure 3a in the form of a diagram.

As can be seen from the diagram of the combined dependence of the degree of sorption on the initial boron concentration for different sorption times (30 to 150 min or more), the degree of increase of the target substance decreases with an increase of the initial boron concentration and a decrease of the phase contact time. As noted earlier, this type of dependence is associated with an increase in the degree of boron saturation of the sorbent phase and diffusion difficulties.

Since the obtained dependences are linear, this allows extrapolating the results of sorption to other values of the variables: the time of the process and the initial content of the extracted substance, without conducting new experiments.

Experiments were carried out using modified mercerized cellulose with "residues" of chlorine derivatives of polyhydric alcohols sewn to it (The results are given in figure 2b).

As it is shown on this figure in comparison with unmodified cellulose, the recovery of H_3BO_3 on this sorbent is also more effective.

The degree of sorption of boron on modified cellulose is described by the empirical equation, which we obtained using the above described calculation method.

$$Y = (2.54\ X + 0.5) \cdot \tau\text{-}630.2 \cdot X + 52.6 \quad (2)$$

From the analysis of the diagram obtained by visualizing this equation (Figure 3b) it follows that the general pattern of the change in the magnitude of the dependence of sorption on the variable factors will remain the same as for the unmodified sorbent. However, the efficiency and speed of extraction is much higher. This is confirmed by a very significant relative increase of the tangent in the slope of the majority of straight lines (about 0.60-0.75 percent of extraction per minute). The tangent of the slope of the straight line to the abscissa axis graphically represents the speed of the process, which was used to estimate the speed of the process.

Figure 2b shows that in the case of long process duration and a large percentage of boron extraction, irrespective of its initial concentration, the straight lines converge at one point. This is obviously due to the almost complete saturation of the sorbent with boron over time. The saturation of the sorbent for all solutions occurs in this case with a shorter sorption time of about 100 minutes.

It is convenient to use the diagram (figure 3) proposed by us to determine the efficiency of sorption of a substance at given concentrations of boron and the sorption time, for example at a boron concentration of 0.2 g / l in 165 minutes 95% of the substance is extracted and 150 min less than 80% (figure 3 b). The proposed methodology for drawing up such diagrams can be used to visualize a wide range of other processes of interphase redistribution and concentration of matter in heterogeneous systems.

Thus, it can be concluded that the mercerization of the studied cellulose species and the subsequent "sewing" of chlorine derivatives of polyhydric alcohols to it led to a significant increase in the sorption capacity of cellulose.

With an increase in the background salt, the extraction of the substance is significantly improved as a result of the salting out effect. For example, it was found that, other things being equal, an increase in the NaCl concentration from 0.005 N to 1.00 N increases the coefficient of interphase distribution of boron

(Kc) 226 times between the sorbent phase and the aqueous solution from 3350 to 7640, respectively, (initial concentration of 1.40 g / l, S: L = 1: 50). The values of Kc for different concentrations of salting out agent (NaCl) are given below:

Kc	3350	4800	5515	6230	7640
[NaCl]н	0.005	0.050	0.250	0.500	1.00

The free energy of the isochoric process ΔF is related to Kc, the equilibrium constant of the chemical reaction or the chemisorption process by the following formula:

$$-\Delta F^{o}_{298} = 2{,}3 \cdot RT \cdot \lg K_c$$

where: T is the absolute temperature, K; R is the universal gas constant.

Calculation using this formula using the values of Ks for the smallest and largest concentration of the salting out agent NaCl (0.005 and 1.00 N) showed that the free energy of sorption for a more concentrated salting out solution with respect to the less concentrated salting out solution increases 1.31 times (almost 30%). This is due to a decrease in water activity with an increase in the salt background as a result of the binding of water molecules to hydrated ion shells.

Further, the influence of the ratio of the solid and liquid phases (S: L) on the process was studied. Studies have shown that under comparable conditions, a change in S: L ÷ 1: 100; 1:75; 1:50; 1:25 significantly increases the extraction of boron in modified cellulose by (30-50%) compared to unmodified cellulose.

Data on the sorption capacity of sorbents allow us to conclude that the CM exhibits a static capacity of 6.02 mg/g dry sorbent, i.e. at the level of synthetic boron selective anion exchangers (ANB).

We have noticed that CM is not susceptible to biocorrosion, unlike ANB since it does not contain glucose derivatives in its structure.

Practical interest is the extraction of boron at its small (milligram) concentrations and a large salt background, modeling the composition of natural and man-made mineral raw materials.

Under dynamic conditions more closely approximated to real technological conditions, the CM also does not concede by its effectiveness to ANB [3], purifying the water from the boron below the MRL (0.5 mg / l) [3, 21, 22].

The equation (II) shows that the mechanism of boron absorption is analogous to polycondensation reactions with the splitting of water and the formation of heteropolymers in inorganic, colloidal chemistry and biochemistry, as well as technologies for processing hydromineral raw materials. The thermodynamic evaluation of this reaction, taking into account the salting out effect according to the formula given above for free energy, showed that ΔF^{o}_{298} is in the range from -10.5 to -20.0 kJ / mol. The free energy of Gibbs calculated by us in the reaction: 2 OH → H_2O + O equals -19.0 kJ / mol which means it is in the same interval. It is interesting that the energy of hydrogen bond widely distributed in nature is also in the range of 12.6÷33.6 kJ / mol [23].

Other things being equal, the CM is significantly cheaper than synthetic ANB type sorbents, because it is obtained from cellulose waste, and when modified, non-defective materials are used. The CM after use is easily regenerated by washing with a 3% solution of hydrochloric acid. The recovery of boron-containing waters after the regeneration of sorbents is sufficiently effective in the form of components of fertilizers [18, 19, 20], while simultaneously obtaining purified water, which will help solve the problem of complex use of mineral raw materials and ecology [24, 25].

Study of the interphase distribution of boric acid between aqueous solutions and the solid phase of chemically modified cellulose, the development of non-waste technologies for processing and utilization of natural and man-made boron-containing hydromineral raw materials.

Conclusion. Based on the work done, the following conclusions can be drawn:

– a method for obtaining an efficient borselective sorbent by chemical "sewing" to mercerized natural cellulose (CM) of chlorinated derivatives of polyhydric alcohols has been developed;

– CM is a more affordable and inexpensive sorbent and, other things being equal, does not concede to the best boron elective ion exchangers of the ANB type (boron-selective anionite) by its sorption characteristics;

– in heterogeneous systems it is proposed to depict data on the interphase distribution of matter in analytical (mathematical) and graphical interpretation (in the form of diagrams), taking into account three variables (time, concentration and recovery percentage), which allows obtaining results without conducting additional experiments.

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МОДИФИКАЦИЯЛАНҒАН ЦЕЛЛЮЛОЗА МЕН СУ ЕРІТІНДЕРІНІҢ АРАСЫНДАҒЫ БОР ҚЫШҚЫЛЫНЫҢ ФАЗААРАЛЫҚ БӨЛІНУІ

Аннотация. Гидроминералды шикізатты өңдеу қажеттілігіне байланысты көптеген зерттеушілер табиғи материалдар негізінде органикалық сорбенттерді алудың іргелі және қолданбалы мәселеріне, атап айтқанда, мерсеризация әдістерімен модификацияланған әртүрлі целлюлозаға (сілтілі ерітінділерімен өңдеу) және функционалдық топтардың химиялық модификациясына қызығушылық танытады.

Бор қышқылының фазааралық бөлуді нақтылы, Prunus armeniaca (абрикос) мерсеризденген, содан кейін химиялық түрде модификацияланған ұсақталған өрік сүйегіне зерттеу жүргізілді.

Сорбент материалында бор қышқылына арналған селективті қасиеттері бар, оның су фазасынан алынып, H_3BO_3-ні сорбент (өзгертілген целлюлоза) қатты фазасында шоғырландыратыны анықталды. Элементтік химиялық және масс-спектрометриялық талдау жүргізілді, сорбенттердің ИК-спектрлері түсірілді, олардың негізінде құрылымдар берілді және бор қышқылын модификацияланған целлюлоза (МЦ) фазасы арқылы жұту механизмі ұсынылды.

Түйін сөздер: бор қышқылы; өзгерту; фазааралық таралуы.

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МЕЖФАЗОВОЕ РАСПРЕДЕЛЕНИЕ БОРНОЙ КИСЛОТЫ МЕЖДУ ВОДНЫМИ РАСТВОРАМИ И МОДИФИЦИРОВАННОЙ ЦЕЛЛЮЛОЗЫ

Аннотация. В связи с необходимостью переработки гидроминерального сырья многие исследователи проявляют интерес к фундаментальным и прикладным проблемам получения органических сорбентов на основе природных материалов, в частности к различным видам целлюлозы, модифицированной методами мерсеризации (обработкой растворами щелочей), а также химическим изменением функциональных групп.

В работе изучено межфазное распределение борной кислоты на мерсеризованной, затем химически модифицированной дробленой косточке абрикоса обыкновенного, Prunus armeniaca (урюка).

Установлено, что вещество сорбента обладает селективными свойствами к борной кислоте, извлекая ее из водной фазы и концентрируя H_3BO_3 в твердой фазе сорбента (модифицированной целлюлозе). Проведены элементный химический и масс-спектрометрический анализы, сняты ИК-спектры сорбентов, на основании которых дана их структура, а также предложен механизм поглощения борной кислоты фазой модифицированной целлюлозы (ЦМ).

Ключевые слова: борная кислота; модификация; межфазовое распределение.

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