ESTUDO DA SORÇÃO DE CATIONS DE AMÔNIO EM UM SORVENTE FIBROSO CARBOXÍLICO

STUDY OF THE SORPTION OF AMMONIUM CATIONS ON A FIBROUS CARBOXYLIC SORBENT

ИССЛЕДОВАНИЕ СОРБЦИИ КАТИОНОВ АММОНИЯ НА ВОЛОКНИСТОМ КАРБОКСИЛЬНОМ СОРБЕНТЕ

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RESUMO

Introdução: Águas residuais da produção de fertilizantes minerais, agroindústrias contendo íons amônio, causam prejuízos significativos à piscicultura; portanto, devem ser purificadas antes do descarte. A sorção por troca iônica é um método promissor para o isolamento de cátions amônio. O objeto do estudo foi uma fibra de quimissorção VION KN-1, que desenvolveu superfície e alta taxa de sorção. **Objetivo:** Estudar a cinética de sorção de cátions amônio de soluções aquosas no VION KN-1; treinar uma RNA para prever o grau de recuperação de íons de amônio de águas residuais usando o software *Statistica Neural Networks Versão 13*. **Métodos:** A concentração de íons de amônio na solução foi estabelecida por potenciometria direta. As isotermas de sorção foram construídas usando o método de concentrações variáveis. Para determinar o estágio limite, as dependências cinéticas obtidas foram representadas nas coordenadas das equações de Boyd-Adamson para difusão interna/externa. **Resultados e discussão:** Durante a sorção de soluções com diferentes teores de nitrogênio amoniacal, os valores dos coeficientes de distribuição (K_d) estão no nível de $2,3 \cdot 10^3 \text{ cm}^3/\text{g}$, o que excede significativamente este parâmetro para ionitas granulares. Os dados experimentais de sorção foram verificados usando as isotermas de Freundlich ($R^2 = 0,9224$) e Langmuir ($R^2 = 0,9996$). O grau máximo de recuperação (acima de 96%) foi alcançado passando uma solução com uma concentração de $11,3 \text{ mmol/dm}^3$. Usando uma série de dados experimentais, a rede neural MLP-3-5-1 foi treinada. O coeficiente de determinação $R^2 = 0,999420$ obtido para a amostra de treinamento caracteriza alto desempenho da rede. **Conclusões:** A equação de Langmuir descreve melhor o processo de sorção de NH_4^+ em um sorvente fibroso. É razoável usar VION KN-1 no estágio de tratamento fino. A dessorção do íon amônio da fibra foi realizada por meio de solução ácida. As soluções resultantes de sais de amônio podem ser usadas como fertilizantes líquidos. As redes neurais treinadas podem ser usadas para prever o grau de recuperação de íons de amônio pelo sorvente VION KN-1.

Palavras-chave: *tratamento de águas residuais, quimissorção, íons de amônio, fibra de troca iônica, redes neurais artificiais.*

ABSTRACT

Background: Wastewater from the mineral fertilizer production, agribusiness containing ammonium ions causes significant harm to fish farming; therefore, it must be purified before discharge. Ion-exchange sorption is a promising method for isolating ammonium cations. The object of the study was a chemisorption fiber VION KN-1, which has developed surface and high sorption rate. **Purpose:** To study the sorption kinetics of ammonium cations from aqueous solutions on VION KN-1; to train an ANN to predict the degree of recovery of ammonium ions from wastewater using Statistica Neural Networks Version 13. **Methods:** The ammonium ion concentration in the solution was established by direct potentiometry. Sorption isotherms were constructed using the method of variable concentrations. To determine the limiting stage, the obtained kinetic dependencies were represented in the coordinates of the Boyd-Adamson equations for internal/external diffusion. **Results and Discussion:** During sorption from solutions with different ammonium nitrogen contents, the values of distribution coefficients (K_d) are at the level of $2.3 \cdot 10^3 \text{ cm}^3/\text{g}$, which significantly exceeds this parameter for granular ionites. Experimental sorption data were verified using Freundlich ($R^2 = 0.9224$) and Langmuir ($R^2 = 0.9996$) isotherms. The maximum degree of recovery (over 96 %) was achieved by passing a solution with a

concentration of 11.3 mmol/dm³. Using an array of experimental data, the MLP-3-5-1 neural network was trained. The coefficient of determination $R^2 = 0.999420$ obtained for the training sample characterizes high network performance. **Conclusions:** The Langmuir equation better describes the process of NH_4^+ sorption on a fibrous sorbent. It is reasonable to use VION KN-1 at the fine treatment stage. Ammonium ion desorption from the fiber was performed by acid solution. The resulting solutions of ammonium salts can be used as liquid fertilizers. The trained neural networks can be used to predict the degree of recovery of ammonium ions by sorbent VION KN-1.

Keywords: wastewater treatment, chemisorption, ammonium ions, ion-exchange fiber, artificial neural networks.

ABSTRACT

Сточные воды производства минеральных удобрений, а также агропромышленного комплекса, содержат ионы аммония. Загрязнённая азотными соединениями вода наносит существенный вред рыбохозяйству, поэтому перед сбросом ее необходимо очищать. Перспективным способом выделения катионов аммония является ионообменная сорбция. Объектом исследования являлось ионообменное волокно ВИОН КН-1, обладающее развитой поверхностью и высокой скоростью сорбции. Цель работы - изучение кинетики сорбции катионов аммония из водных растворов на ВИОН КН-1. Обучение искусственной нейронной сети для прогнозирования степени извлечения ионов аммония из сточных вод с использованием Statistica Neural Networks версия 13. Концентрацию ионов аммония в растворе определяли на иономере И-130 с ионоселективным электродом. Методом переменных концентраций построены изотермы сорбции. Для определения лимитирующей стадии, полученные кинетические зависимости, представляли в координатах уравнений Бойда-Адамсона для случая внутренней и внешней диффузии. При сорбции из растворов с разным содержанием аммонийного азота значения коэффициентов распределения (K_d) находятся на уровне $2.3 \cdot 10^3 \text{ см}^3/\text{г}$, что значительно превышает значения для зернистых ионитов. Экспериментальные данные по сорбции проверены с помощью изотерм Фрейндлиха ($R^2 = 0.9224$) и Ленгмюра ($R^2 = 0.9996$). Максимальная степень извлечения более 96 % достигалась при пропускании 200 см³ раствора с концентрацией 11.3 ммоль/дм³. Используя массив экспериментальных данных, обучили нейронную сеть MLP-3-5-1. Полученный для обучающей выборки $R^2 = 0.999420$ характеризует высокую производительность сети. Уравнение Ленгмюра лучше описывает процесс сорбции NH_4^+ на волокнистом сорбенте. ВИОН КН-1 целесообразно использовать на стадии доочистки. Десорбция ионов аммония из волокна осуществляли раствором кислоты. Образующиеся растворы аммонийных солей можно применить как жидкие удобрения. Обученные нейронные сети могут быть использованы для прогноза степени извлечения ионов аммония сорбентом ВИОН КН-1.

Keywords: очистка сточных вод, хемосорбция, ионы аммония, ионообменное волокно, искусственные нейронные сети

1. INTRODUCTION:

Ammonium ions are present in the gray and agricultural wastewater and in the wastewater from the production of mineral fertilizers. The concentration of ammonium nitrogen varies in the range of 50-200 mg/dm³. Water purification from ammonium is very important for the ecology and the environment. Such waters cannot be drained into natural water reservoirs, since with increased ammonium content, the ability of hemoglobin in fish to bind oxygen decreases, which leads to a reduction in their numbers and violation of ecological balance (Nageeb, 2013). Groundwater with excessive ammonium content is unsuitable for drinking. According to the standards of the main indicators

of water quality, following the requirements of the sanitary standards of the Russian Federation, the World Health Organization (WHO), and the European Community (EU), the concentration of ammonium nitrogen should not exceed 2 mg/dm³, 1.5 mg/dm³, and 0.5 mg/dm³, respectively.

Various methods are used to remove ammonium from wastewater, such as a reagent method, aeration in an alkaline environment, ion exchange, biochemical, and reverse osmosis. Ion exchange sorption is a promising method for the isolation of NH_4^+ (Al-Sheikh *et al.*, 2021; El-Shafeyet *et al.*, 2014; B. Clark *et al.*, 2020).

The study of the kinetics of ammonium ion sorption makes it possible to establish the rate at

which the equilibrium is reached. An important issue in sorption technology is the determination of the limiting stage of mass transfer. Information about this will help to choose the optimal sorption conditions. It is well known that the exchange process on sorbents consists of five stages:

- diffusion of sorbed ions from an external solution to the surface of the grain ;
- diffusion of sorbed ions through the thickness of the grain to the active ion-exchange groups;
- chemical ion-exchange reaction;
- diffusion of displaced ions towards the surface of the grain;
- diffusion of counterions from the surface of the grain into the volume of the solution.

Stages 1 and 5 are external or film diffusion; stages 2 and 4 are internal or gel diffusion (Kokotov *et al.*, 1970, Riemann *et al.*, 1973).

There is extensive literature on the issue of ammonium sorption on natural and treated with various compounds aluminosilicates (Sakalova *et al.*, 2018, Han *et al.*, 2020) wast. The sorption of NH_4^+ ions on natural mineral sorbents — zeolite, glauconite, and palygorskite — was studied. It was found that in the range of temperatures studied (293–308 K), zeolite exhibits the best sorption properties; the temperature has a slight effect on the sorption process. It is shown that the experimental isotherms for zeolite and glauconite are straight lines. The sorption isotherm of ammonium ions on palygorskite deviates from linearity, which necessitates its analysis using the well-known theoretical models. It is established that the Langmuir equation best describes the process of sorption of ammonium cations on palygorskite (Malyovannyi *et al.*, 2013, Gumnitsky *et al.*, 2013).

The adsorption equilibrium of NH_4^+ ions from aqueous solutions on natural clinoptilolite was considered (Ivanova *et al.*, 2010). The possibility of changing the adsorption capacity by chemical modification of natural clinoptilolite was also tested. The zeolite forms enriched with Ca^{2+} , Ba^{2+} , K^+ , Cu^{2+} , and Co^{2+} ions were used. The experimental data were verified using the Freundlich and Langmuir isotherms. The kinetics of adsorption were studied and the effect of adsorbent mass, particle size, and contact time of solid and liquid phases on the adsorption capacity. The diffusion coefficients in macropores and in zeolite crystals were calculated using the assumption of a two-porous model.

Sorption of ammonium ions on natural clinoptilolite and artificial zeolite has been studied (Guida *et al.*, 2020; Adam *et al.*, 2020). To effectively remove ammonium from water, a hollow fiber ceramic membrane obtained from clinoptilolite has been proposed. The effect of different sintering temperatures in the manufacture of membranes (900–1150 °C) and the pH of the initial ammonium solution was investigated. The maximum adsorption activity was reached at pH 7.

To increase the efficiency of adsorption of ammonium nitrogen from wastewater, the surface properties of activated carbon were improved by modifying it with iron (III) salt solution (Ren *et al.*, 2021). The total pore volume and specific surface area of the activated carbon increased after modification. The best adsorption effect was achieved under neutral conditions.

It has been shown that it is possible to use the solution to the problem of mixed diffusion dynamics of sorption with a linear isotherm to describe the initial sections of the output curves of ammonium ions on the Na-form of clinoptilolite-containing tuff of the Chuguevskoye deposit (Nikashina *et al.*, 2008).

The dynamics and kinetics of the process of adsorption of NH_4^+ cations on dispersed silicon earth (Opoka) were studied (Kuzmina *et al.*, 2008). The dependence of the ammonium ion sorption and dynamic capacity on the linear flow rate was considered. It was shown that an increase in the flow rate leads to an increase in the mass transfer coefficient and a significant decrease in the dynamic sorption of NH_4^+ . Kinetic studies have shown that the process of ammonium ion sorption on Opoka is a two-stage process.

In Belchinskaya *et al.* (2013), the kinetics and dynamics of sorption of NH_4^+ on natural and activated by sulfuric acid and alkali aluminosilicate sorbent $M_{45}K_{20}$, consisting of montmorillonite and clinoptilolite, were studied. The diffusion coefficients and the adsorption equilibrium constant were calculated. It was shown that an increase in the sorption capacity of the sorbent after acid and alkaline activation is observed.

To recover ammonium ions from wastewater, a zeolite with improved sorption properties was used. Zeolite N was hydrothermally synthesized from kaolin clay with the addition of potassium hydroxide and potassium chloride solutions. The resulting zeolite had increased cation-exchange capacity

and improved ion exchange kinetics (Probst *et al.*, 2021). NaY zeolite obtained from rice husk ash wastes showed a high adsorption capacity of the monolayer (42.37 mg/g) (Alias *et al.*, 2010). Composite adsorbents based on porous hydrogel and zeolite were obtained and recommended for wastewater treatment from ammonium ions (Putra *et al.*, 2020).

The bentonite treated with NaCl solution with a particle size of 0-0.05 mm showed the maximum increase in recovery efficiency (55.7 %) for ammonium ions. The Langmuir adsorption isotherm matched the equilibrium adsorption data well (R^2 from 0.97 to 0.98), while the Freundlich model proved to be mismatched ($R^2 = 0.77$) (Sakalova *et al.*, 2018).

The ability to remove ammonium ions from an aqueous solution using lightweight expanded clay aggregate (LECA) was investigated. The maximum adsorption capacity of the monolayer, estimated using the Langmuir isotherm, was obtained in the range of 0.229 to 0.254 (mg/g). The contact time required to reach the equilibrium state was 150 min (Sharifnia *et al.*, 2016).

The removal of nitrate and ammonium ions from the water was performed through zeolite (clinoptilolite) filtering material of 400 mm height and particle size 0.315-0.63 mm, at a filtration rate of 5 mg/l. The removal efficiency of NH_4^+ was 95-99.9%. It was noted that zeolite particles of this size are not suitable for the sorption of nitrate ions from aqueous solutions (Mažeikiene *et al.*, 2008).

The study (Kuzmina *et al.*, 2008) devoted to substantiating the theoretical fundamentals of competing for adsorption of multicomponent systems on mineral sorbents is of interest. The sorption capacity of zeolite with respect to ammonium ions in static and dynamic conditions was studied. The authors showed that the presence of foreign ions in the solution significantly reduces the sorption capacity of zeolite with respect to the analyte.

The ion-exchange fiber VION KN-1 is widely used to study the kinetics of the sorption of ammonium ions from solutions. The small diameter of the elementary fiber of 30-50 microns provides a large specific surface area of the fibers (3-10 m^2/g) compared to granular gel ionites (0.1 m^2/g). The fiber has better swelling properties, a high sorption rate, and a high resistance to abrasion by a moving flow. A less regenerating solution is required for the regeneration of fibrous ion exchangers compared

to granular ion exchangers (Kuzmina *et al.*, 2008). Besides, the fiber can be used in filters and other equipment of any design used in water treatment processes.

Artificial neural networks (ANNs) are a non-linear and nonparametric data processing method used in various fields of chemical technology for predicting the quality of the resulting product, as well as for automation, optimization, and planning of the technological process (Han *et al.*, 2011; Heddam *et al.*, 2011; Heddam *et al.*, 2018, Alver *et al.*, 2020).

The increasing scarcity of good quality water resources and the growing use of fertilizers imply the use of efficient water and wastewater treatment methods. To predict the performance of biological systems in removing pollutants from water, artificial neural networks were used (Khataee *et al.*, 2011). In (Giwa *et al.*, 2016), a new bioreactor configuration for wastewater treatment was considered. ANNs were also used to simulate the operation of the bioreactor during water treatment from phosphate ions and ammonium cations.

The removal efficiency of ammonia nitrogen was studied from simulated wastewater with spent foundry sand based on 120 serial experiments, which were modeled by a three-layer artificial neural network. The sorption process was well described using the ANN model, and the maximum sorption capacity was 0.9 mg/g (Faisal *et al.*, 2019).

In this study, hybrid prediction models were used to estimate ammonium adsorption from wastewater on zeolite. Feed-forward Neural Network (FF-NN) and Elman-Recurrent Neural Network (ER-NN) containing two different activation functions were used to determine nonlinear dependencies. As a result, the ammonium adsorption rate can be estimated with a 95% probability using the best hybrid model (H-PM4) (Cagcag Yolcu *et al.*, 2021).

This work aims to study the kinetics of sorption of ammonium cations from aqueous solutions on a fibrous carboxyl sorbent VION KN-1, determine the limiting stage of the process, study the effect of the fiber layer height on the degree of recovery of ammonium ions, as well as train an artificial neural network to predict the degree of ammonium ion recovery from wastewater using the software package *Statistica Neural Networks13*

2. MATERIALS AND METHODS:

2.1. Materials (VION KN-1)

The object of the study was VION KN-1. The criteria for choosing VION KN-1 fiber were its characteristics, such as a developed surface, which facilitates the access of reagents to the exchange centers of the fiber; better swelling ability; high sorption rate, and greater resistance to abrasion by a moving flow. For the regeneration of fibrous ion exchangers, a less regenerating solution is required in comparison with granular ion exchangers (Kopylova *et al.*, 2006). In this regard, fibers are widely used both for wastewater treatment from various ions (Xu *et al.*, 2021; Grachek *et al.*, 2020; Wang *et al.*, 2020) and for the recovery of rare earth elements (Li *et al.*, 2020).

Ion exchange fiber VION KN-1 ion exchanger is obtained based on polyacrylonitrile (PAN) fiber (Perepelkin *et al.*, 1985). The radius of the chemisorption fiber PAN = $11 \cdot 10^{-6}$ m. The sorption of water vapor by the nitron fiber at a relative air humidity of 65% is 0.8-1.5 wt. %, and at 95% - 1.5-3.5 wt. %. The swelling of the fiber in water is 3-6 wt.% (Perepelkin *et al.*, 1985).

The fibrous sorbent VION KN-1 is synthesized by the sequential processing of nitron fiber with a 20-25% aqueous hydrazine solution for 1.5-2.0 hours, followed by hydrolysis with caustic soda.

The functional groups of the cation exchange fiber are $\sim\text{COO}^-$ (H^+ carboxyl groups (H^+ - or Na^+ - forms) (Figure 1).

Figure 1. The structure of the ion-exchange fiber VION KN-1 in the hydrogen form

This ion exchanger does not dissolve in any known solvent, including when heated. It is capable of multiple regenerations, which are carried out with solutions of acid and alkali. With the help of the fibrous sorbent VION KN-1, wastewater with a concentration of harmful or valuable substances less than 40 mg/l undergoes treatment and additional treatment. At such concentrations, the use of other capture methods is not economically efficient. The sorbent VION KN-1 is produced in the form of chemisorption fiber and nonwoven fabric (Figure 2), the characteristics of which are shown in Table 1.

Table 1. Characteristics of a chemisorptive fiber and nonwoven fabric VION KN-1

Characteristics	Value
Static ion exchange capacity (IEC), mg-eq/g	3.5-5.0
Linear density, tex	0.6-0.8
Breaking load, CN / tex	3.5-10.0
Breaking elongation, %	25-40
Fabric width, cm	156-164
Surface density, g/m ²	250, 800

2.2. Experimental procedures of VION KN-1 obtention or synthesis

The ammonium ion concentration in the solution was determined using an I-130 ion meter with an ion-selective electrode according to a calibration graph (voltage dependence on the logarithm of molar concentration).

The study of the equilibrium and kinetics of sorption of ammonium ions was carried out under static conditions from aqueous solutions of ammonium chloride with stirring at a temperature of 298 K.

Figure 2. Image of the sorbent VION KN-1 on Altami BIO microscope in the form of a chemisorption fiber (a) and nonwoven fabric (b)

In the work, the limited volume method was used (Selemenev *et al.*, 2004), the essence of which was as follows: a sample of an air-dry ion exchanger weighing 0.3 ± 0.0002 g was placed in 100 ml of ammonium chloride solution. The working ammonium chloride solutions were

prepared from a standard solution by dilution. The ammonium ion concentration in the prepared solutions was in the range of 32 to 204 mg/dm³ (1.78÷11.3 mmol/ dm³). The experiment was carried out at a temperature of 298±2 K. Constant mixing led to an instantaneous equalization of the concentration of NH₄⁺ ions in the solution volume. The change in the ammonium ion concentration was determined by direct potentiometry using an NH₄⁺- selective electrode. The amount of the sorbed substance (mol/g) was calculated by the formula (Eq. 1).

$$CE = (C_o - C_p) \cdot \frac{V}{m} \quad (\text{Eq. 1})$$

where m is the mass of the fiber sample, g; V is the solution volume, dm³; C_o , C_p is the initial and equilibrium concentration of ammonium ions in the solution, mol/ dm³.

To calculate the coefficient of internal diffusion (D , m²/ sec), the following equation was used (Eq. 2):

$$D = Br^2 / \pi^2 \quad (\text{Eq. 2})$$

Where r is the radius of the chemisorption fiber, m ($r_{PAN} = 11 \cdot 10^{-6} m$); B is the slope angle tangent of the direct dependence $Bt = f(t)$, where $Bt = (F/1.08)^2$.

The half-sorption time $T^{1/2}$ (min), indirectly characterizing the sorption rate, was calculated by the equation (Eq. 3):

$$T^{(1/2)} = 0.03r^2(D \cdot 60), \quad (\text{Eq. 3})$$

The distribution coefficient of ammonium ions (K_d , sm³/g) was calculated by the formula (Eq. 4):

$$K_d = (R/100 - R) \cdot V/m, \quad (\text{Eq. 4})$$

where R is the degree of recovery, %.

To calculate the empirical constants a , b , and k , the Langmuir and Freundlich equations were used. The Langmuir equation (Eq. 5):

$$1/CE = 1/kb \cdot 1/C_p + 1/k, \quad (\text{Eq. 5})$$

where CE is the sorption value, mol/g, b , k are empirical constants, and C_p is the equilibrium concentration of the reagent in the solution, mol/dm³.

The Freundlich's equation (Eq. 6):

$$\lg CE = \lg a + 1/n \cdot \lg C_p, \quad (\text{Eq. 6})$$

Where CE is the sorption value, mol/g, a is the empirical constant, and C_p is the equilibrium concentration of the reagent in the solution, mol/dm³.

The procedure for the sorption of ammonium ions under dynamic conditions was as follows. One of the most commonly used and effective options for the sorption process is passing wastewater through a fixed ionite bed (column or filter). The filter was filled with samples of ion-exchange fiber weighing from 0.31 to 0.93 g. A solution containing ammonium ions of a known concentration was passed through the filter at a certain rate. The content of ammonium nitrogen in the portions of the filtrate (50 cm³) at the outlet from the filter was determined by the potentiometric method. The passage of the solution was stopped when the concentration of ammonium ions in the filtrate became equal to the initial concentration. The data obtained were used to plot the output curves in the coordinates $C/C_o = f(V)$.

The experimental data were processed using the Statistica Neural Networks software package to predict the degree of recovery of ammonium ions from wastewater. Given the results of the preliminary experiments, the main parameters (input parameters), which have a significant effect on ion sorption, were selected: pollutant concentration, mol/dm³; the volume of a flowing solution, dm³; fiber mass, g. The response function (output parameter) was the degree of ammonium recovery, %.

2.3 Neural network models

Based on the data obtained, neural network models were constructed; an automated neural network with the following settings was chosen as the construction strategy: network type - multilayer perceptron, the minimum and maximum number of hidden neurons - 3 and 10, respectively. Five networks with the smallest error with respect to the control sample were saved. The neural network MLP-3-5-1 with high determination coefficients was 0.999420; 0.998767; 0.998572 was selected from them for training testing and sampling control. Such coefficients indicate a high performance of the trained network. The training error was 0.279647; a second-order accuracy training algorithm, BFGS, was chosen. The activation function of

hidden neurons is identical, and of the output neurons are logistic.

3. RESULTS AND DISCUSSION:

The obtained experimental data on the sorption of ammonium ions by the fibrous sorbent VION KN-1 from solutions with different contents of ammonium nitrogen are shown in Table 2. The maximum value of the sorption capacity of the VION KN-1 sorbents at a given concentration of ammonium ions is 2.47 mmol/g. During sorption from dilute solutions, the distribution coefficients (K_d) are at the level of $2.3 \cdot 10^3 \text{ cm}^3/\text{g}$, which is significantly higher than the values for granular ionites.

The degree of recovery (R) of ammonium ions increases with decreasing concentration, which indicates the efficiency of the fiber for the additional treatment of diluted effluents to the maximum permissible concentration (MPC). Such indicators of the degree of recovery (less than 90%) can be explained by the fact that the selected ratio of the volume of the solution and the fiber sample ($330 \text{ cm}^3/\text{g}$) is not the optimal ratio of the parameters: solution volume – ionite sample – pollutant concentration.

Sorption isotherms were constructed using the method of variable concentrations to assess the affinity of the investigated fibrous ionites to ammonium ions. For this, equilibrium concentrations were used, which are the residual content of ammonium ions in the solution after saturation of the fibrous sorbent.

The isotherms of sorption of ammonium ions by ion-exchange fiber in the coordinates $CE=f(C_p)$ have the form of a convex curve (Figure 3). The initial regions for both ionites are close to linear (Henry's region)). The sorption value is practically proportional to the ammonium ion concentration in these regions. At low concentrations ($C_p < 1 \text{ mmol/dm}^3$), almost quantitative sorption occurs in the solution.

Figure 3. Isotherms of sorption of ammonium ions by the fibers VION KN-1

The Langmuir and Freundlich models were applied to quantitatively describe the equilibrium sorption process of ammonium ions. Figure 4 shows that the sorption isotherms by the VION KN-1 sorbent are linearized in the coordinates of the Langmuir equation, as indicated by the values of the reliability R^2 . This indicates that sorption has a monolayer character, and its maximum is achieved when the monolayer is completely filled and there is no interaction between the sorbed ions. The values of the parameters in the equations of the Langmuir and Freundlich models and the correlation coefficients are presented in Table 3.

b

Figure 4. Isotherms of ammonium ion sorption by fibrous sorbents in the coordinates of the Langmuir (a) and Freundlich (b) equations

The dependence of the sorption capacity of ionites on the initial ammonium ion concentration in the inverse coordinates $1/CE=f(C_H)$ (Figure 5) was constructed. This dependence can be used to obtain an analytical equation that makes it possible to predict the sorption capacity of fibrous sorbents.

Based on the obtained straight-line correlations, the values of the CE_{00} and K_L constants were reliably calculated. By introducing constants into the Langmuir equation, we obtain analytical equations for predicting the sorption capacity of VION KN-1 (Eq. 7):

$$CE = 10.32 \cdot \frac{0.02975C_H}{1 + 0.02975C_H} \quad (\text{Eq. 7})$$

Figure 5. The dependence of the sorption capacity of fibrous sorbents on the content of ammonium nitrogen in the solution in the inverse

coordinates

The equation obtained makes it possible to determine the sorption capacity of the test fiber concerning ammonium ions at any initial concentration. The value of the sorption capacity calculated according to the above equation with varying the initial content of ammonium nitrogen agrees well with the experimentally obtained data (Table 4).

Table 4. The calculated and experimental data on the sorption capacities of the fibrous sorbent VION KN-1 for ammonium ions

Ionite brand	C_H , mmol/dm ³	Value CE, mmol/g	
		Calculated	Experimental
VION KN-1	1.79	0.52	0.52
	4.79	1.29	1.31
	7.56	1.90	1.93
	11.3	2.59	2.46

To determine the optimal ratio between the solution volume and the mass of fibrous sorbents, the dependence of the degree of recovery of ammonium ions on the hydromodule was built (Figure 6). The dependences show that with a decrease in the ratio between the volume of solution and the mass of fibers (hydromodule), the degree of recovery increases. In this case, complete recovery of ammonium ions is not achieved (less than 90%). This is due to the fact that the sorption process was carried out at a higher initial concentration of ammonium ions in the solution ($C_H = 10$ mmol/dm³).

Figure 6. Effect of the hydromodule on the degree of recovery of ammonium ions

Thus, when treating wastewater with a high content of ammonium ions (10 mmol/dm³), a

two-step process is required. At lower concentrations of ammonium ions, the test fiber can be used to purify water up to the MPC value.

The kinetic dependences of sorption make it possible to determine the time of the establishment of the equilibrium, which characterizes the rate of this process. Figure 7 shows the kinetic curves of ammonium ion sorption by the fiber. It is clear that the type of kinetic sorption curves is similar and is described by the equation $CE = CE_m(1 - \exp(-\beta\tau))$, where CE is the sorption value, τ is the sorption time, CE_m and β are constant values. The constant CE_m characterizes the sorbent capacity at saturation; β characterizes the slope of the curve.

Figure 7. Kinetics of ammonium ion sorption by VION KN-1 fiber. The concentration of the external solution is 1-11.3; 2-7.55; 3-4.72; 4-1.78 mmol/dm³, respectively.

The sorption rate of ammonium cations on a fibrous sorbent is maximal during the first 10 minutes. The high sorption rate is explained not only by the structural characteristics of the fiber but also by the mixing of the external solution - sorbent system. This probably leads to a decrease in film thickness and an increase in mass transfer. With an increase in the concentration of the external solution, the time of the establishment of the equilibrium state and the sorption capacity increase.

To assess the contribution of the diffusion of ammonium ions through the “film” and inside the VION KN-1 fiber matrix, the kinetic dependences obtained in this work were presented in the coordinates of the Boyd – Adamson equations for the case of internal and external diffusion. To determine the limiting stage, the following dependences of the degree of conversion (F) on the sorption time τ were obtained and analyzed: $F - \sqrt{\tau}$ and $-\ln(1-F) = f(\tau)$, where $F = CE\tau/CE_m$,

$CE\tau$ is the sorption capacity of the fiber for a certain sorption time, CE_m is the maximum sorption capacity of the fiber.

Figure 8 shows the kinetic curves. The correlation coefficients are close to unity; the linearity of the dependencies in these coordinates indicates the contribution of internal diffusion. For a cylinder-shaped ion exchanger, linearity must be observed with practical exactness at $F \sim 0.5$.

Figure 8. Kinetic curves of ammonium ion sorption by VION KN-1 fiber in the coordinates of the Boyd-Adamson equations for the case of internal diffusion. The concentrations of external solutions: 1 - 11.3; 2-7.55; 3-4.72; 4 - 1.78 mmol/dm³, respectively.

To assess the effect of external diffusion, the kinetic sorption curves were considered in the coordinates $-\ln(1-F) = f(t)$ at different degrees of sorbent filling (Figure 9).

Figure 9. Kinetic curves of ammonium ion sorption by VION KN-1 fiber in the coordinates of the Boyd-Adamson equations for the case of external diffusion. The concentrations of the external solution: 1 - 11.3; 2-7.55; 3-4.72; 4 - 1.78 mmol/dm³, respectively.

It is clear that linearity is observed in the low concentration region. However, with increasing F , the curves cease to be linear, which emphasizes the effect of internal diffusion on

sorption at a certain degree of fiber filling.

The value of the kinetic coefficients B for the ion exchange fiber was determined from the linear dependences ($Bt - f(t)$) (Figure 10). The obtained correlation coefficients (R^2) confirm the limiting contribution of internal diffusion to ammonium ion sorption.

Figure 10. Dependence of the parameter Bt on the duration of ammonium ion sorption by VION KN-1 carboxyl fiber. The concentrations of the external solution: 1 - 11.3; 2-7.55; 3-4.72; 4 - 1.78 mmol/dm³, respectively.

In order to make sure that the sorption by VION KN-1 fiber is limited by intradiffusion restrictions, the method with interruption of the ionite-solution contact, described by Kressman and Kitchener (Nesterov *et al.*, 2007), was applied.

Figure 11 shows that after a certain time ("storing" of the ionite, 10 min) and the resumption of contact of the solution with the fiber, the rate of the ion-exchange process at the initial moment increases. This also indicates the gel kinetics of the sorption of ammonium ions by the VION KN-1 fiber.

Figure 11. Kinetic dependences of the ammonium ion sorption taking into account the time of interruption of the ionite-solution contact (1); without interruption (2)

The calculated values of the kinetic characteristics are shown in Table 5.

With a decrease in the external solution concentration, the degree of recovery of ammonium ions by VION KN-1 fiber increases.

The Langmuir and Freundlich models were used to quantitatively describe the equilibrium process of ammonium ion sorption by a fibrous sorbent. The calculation results are presented in Table 6.

Table 6. Calculation results for Langmuir and Freundlich models

Model	Parameters	Confidence value
Langmuir	$k = 3.233$ $b = 773.27$	$R^2 = 0.9996$
Freundlich	a	$R^2 = 0.9224$

The obtained confidence values of R^2 show that the isotherms are linearized only in the inverse coordinates of the Langmuir equation and are not described in the coordinates of the Freundlich equation. Empirical constants k and b are found for the studied isotherms. The possibility of using the Langmuir equation to describe the ammonium ion sorption indicates the equivalence of all active centers of the ion exchange fibrous sorbent, which contributes to the formation of a monomolecular sorption layer on the surface of the ion exchanger.

In this work, the study of sorption under dynamic conditions was carried out. To find the optimal fiber height in the dynamic mode, the output curves of the ammonium ion sorption were obtained (Figure 12).

Figure 12. Output curves of the ammonium ion sorption by VION KN-1 fiber. The solution concentration is 11.3 mmol/dm³; the solution flow

rate is 3 cm³/min.

The total dynamic exchange capacity (TDEC) with respect to ammonium ions for VION KN-1 fiber is ≈ 4.3 mmol/g.

To determine the efficiency of the degree of recovery, the curves of the dependence of the degree of recovery on the mass of the fiber sample and the volume of the solution were constructed and analyzed (Figure 13). As can be seen from Figure 13, the increase in the weight of the sample results in an increase in the degree of recovery of ammonium ions. Thus, by passing 200 cm³ of a solution with a concentration of 11.3 mmol/dm³ through a layer of VION KN-1 fiber weighing 0.93 g, a recovery rate of more than 96% can be achieved.

Figure 13. Curves of the dependence of the degree of recovery on the height of the layer (weight of the sample) and the volume of the passed solution (1 – $m=0.31$ g, $h = 0.5$ cm; 2 – $m=0.62$ g, $h = 1.0$ cm; 3 – $m=0.93$ g, $h = 1.5$ cm)

To assess the effect of the initial solution concentration on the form of the output curves, the dependences of the relative concentration of C/C_0 on the volume of the outflowing solution and on the concentration were obtained and analyzed (Figure 14).

Figure 14. Output curves of ammonium ion

sorption by the fiber VION KN-1 with different solution concentrations. The solution flow rate is 3 cm³/min (1 – 3.34 mmol/dm³; 2 – 7.10 mmol/dm³; 3 – 10.00 mmol/dm³)

Figure 14 shows that at the lowest content of ammonium ions in the initial solution (3.34 mmol/dm³), the relative concentrations for VION KN-1 are less than 0.065. This means that with a decrease in concentration in a dynamic mode, the sorption characteristics of the fibers do not decrease, which makes their use relevant for the purification of dilute solutions containing ammonium ions.

The proof of these statements can be found in Figure 15.

Figure 15. The curves of the dependence of the degree of recovery on concentration and on the volume of the passed solution (1 – 3.34 mmol/dm³; 2 – 7.10 mmol/dm³; 3 – 10.00 mmol/dm³)

As can be seen from Figure 15, when a 700 cm³ solution is passed through a VION KN-1 layer ($m_{\text{fiber}} = 0.93$ g) at low concentrations of ammonium ions in the initial solution, the recovery is greater than 93.

The ammonium ion desorption from the fiber is carried out using acid solutions. As a result, solutions of ammonium salts are formed. The solutions obtained contain ammonium nitrogen, a biogenic element, and can be used to produce liquid mineral fertilizers.

The prediction of the degree of recovery of ammonium ions from wastewater was carried out using the STATISTICA neural network. To establish the dependence of the degree of recovery (R) on the factors listed, the values of the levels and variation intervals of which are shown in Table 7, an experiment has been planned.

A dataset was obtained to train the neural network. This is a set of experiments for which

the values of the input factors and the output parameter (the degree of recovery of ammonium ions) are indicated. An element of the dataset is presented in Table 8.

The adequacy of the trained neural network was checked graphically. For this, a histogram of residues was constructed, i.e., the difference between the input and the output by the network values of the degree of recovery (R) of ammonium ions by the fibrous sorbent (Figure 16).

Figure 16. Histogram of the dependence of residues on the number of observations

As can be seen, there are outlying elements, i.e., deviations in some input values of the degree of recovery from the values of the trained network. These are residues equal to -3.5; -2; -1.5; 1; 1.5. The bulk is concentrated in a small range of residues from -1 to 0.5.

The scattering dependence of the target and output values of the neural network is shown in Figure 17. It is clear that most of the input (target) values are on a straight line at an angle of 45° , i.e., they match the output values.

Figure 17. The dependence of the target values of the degree of recovery of ammonium ions on the output values.

When sorption is carried out under industrial conditions, to maximize the recovery of ammonium ions from wastewater, it is advisable to take into account the boundary conditions of the action of the influencing factors. For this, the pairwise influence of the parameters on the course of the sorption process was studied.

Figure 18 shows the response surface of the output parameter (degree of recovery) depending on the pollutant concentration and the volume of the flowing solution.

Figure 18. The response surface of the output parameter depending on the pollutant concentration and the volume of the flowing solution.

The analysis of the graphical dependencies shows that an increase in the ammonium ion concentration and the volume of the flowing solution reduces the degree of recovery. A value of R greater than 90% can be achieved by passing a solution of volume up to 0.2 dm^3 and a concentration of 0.002 mol/dm^3 .

Figure 19 shows the response surface of the output parameter (degree of recovery)

depending on the fiber mass and the volume of the flowing solution.

Figure 19. The response surface of the output parameter depending on the fiber mass and the volume of the flowing solution.

With an increase in the volume of the flowing solution and a decrease in the fiber mass, the degree of recovery decreases. When a solution with a volume of 0.3 dm³ is passed through a fiber layer with a mass of more than 0.6 g, then the ammonium ion concentration decreases by 84%.

Figure 20 shows the response surface of the output parameter (degree of recovery) depending on the fiber mass and the pollutant concentration.

With a decrease in concentration and an increase in fiber mass, the degree of recovery of the pollutant increases. At a concentration of less than 0.009 mol/dm³ and a fiber mass of more than 0.6 g, the degree of recovery is more than 90 %.

To predict the degree of recovery as a result of ammonium ion sorption under industrial conditions, the test data were analyzed (11 values) (Table 9).

After the analysis, the model was launched using the new input data. Similar sampling was generated, and the saved model with the network identifier, which was previously selected as the best one, was uploaded. A small value of the mean square error indicates a good quality of the constructed model based on the new input data (Table 10).

Figure 20. The response surface of the output parameter depending on the fiber mass and the pollutant concentration.

In Table 10, the column Target presents the data obtained during the experiment, in the column Output - the predicted values of the output parameter for predicting the degree of recovery of ammonium ions. These values were produced by a trained neural network MLP 3-5-1. By comparing the first and second columns, the values of the absolute deviation are calculated. The absolute deviation for the entire data selection does not exceed 1.384330 (the column Absolute residues). The parameters of the relative deviation (Squared residues) are also obtained. The residual sum of squares is minimal and ranges from 0 to 1.916369.

The mean square error of the test sampling is 0.596459226. The low value of the mean square error of the introduced new data indicates a sufficient amount of data for training and the existence of a consistent connection between the ammonium ion concentration in the solution, the volume of the passed solution, the ionite mass of the ion exchanger, and the degree of recovery.

4. CONCLUSIONS:

The assessment of the kinetic characteristics of the ammonium ion sorption by VION KN-1 fiber showed that a high rate characterizes the absorption of the analyte by the fibrous sorbent. A decrease in the concentration of the external solution leads to a decrease in the time for the establishment of the equilibrium state and in the value of the sorption capacity. The experimental data obtained as a result of studying the kinetics of ammonium ion sorption by a carboxyl fiber sorbent under static conditions and the calculations performed made it possible to establish the approximation of the kinetic

equations, which are valid for the case of internal diffusion. The experimental data on the sorption were verified using the Freundlich and Langmuir isotherms. The Langmuir equation best describes the process of ammonium cation sorption on VION KN-1 fibrous sorbent. A trained neural network can predict the parameters of the technological process for the treatment of ammonium ions wastewater with high accuracy.

The parameters affecting the ammonium ion sorption from solutions have been established. The maximum recovery of more than 96% was achieved when passing through a 200 cm³ solution with a concentration of 11.3 mmol/dm³. It is shown that it is reasonable to use ion-exchange fibers at the stage of additional treatment. The solutions obtained after desorption VION KN-1 contain ammonium ions, which allows their use as liquid nitrogen-containing fertilizers.

The trained neural networks can be used to predict the degree of recovery of ammonium ions by the fibrous sorbent VION KN-1, which is proved by the data obtained to verify the adequacy of neural network models.

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7. OPEN ACCESS

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Table 2. Sorption properties of the fibrous sorbent VION KN-1 during the recovery of ammonium ions

Ionite	C_{initial} , mmol/dm ³	C_p , mmol/dm ³	CE , mmol/dm ³	R , %	K_d , cm ³ /g
VION KN-1	1.79	0.23	0.52	87.25	2281.07
	4.79	0.85	1.31	82.30	1549.53
	7.56	1.77	1.93	76.59	1090.40
	11.3	3.9	2.47	65.49	632.48

Table 3. Models and constants of sorption isotherms

Ionite	Langmuir model			Freundlich model		
	CE_{00} , mmol/g	K_L , dm ³ /mmol	R^2	K_F , mmol/g	1/n	R^2
VION KN-1	3.1898	0.8526	0.9999	1.2596	0.5214	0.9665

Table 5. Kinetic parameters of the process of ammonium ion sorption by VION KN-1 fiber

$C_{\text{NH}_4^+}$, mol/dm ³	CE , mmol/ g	R , %	K_d , sm ³ /g	$B \cdot 10^4$	$D \cdot 10^{15}$, m ² /sec	$\tau^{1/2}$, min
0.0113	2.468	65.52	633.41	19	23.31	2.59
0.00755	1.921	76.57	1089.34	10	12.27	4.93
0.00472	1.3135	82.29	1548.84	8	9.81	6.17
0.00178	0.5214	87.25	2281.04	7	8.59	7.3

Table 7. Values of the levels and variation intervals of the factors

Factor		Factor variation levels			Variation interval
Name	Notation				
Ammonium ion concentration	C , mol/dm ³	0.002	0.007	0.012	0.005
Solution volume	V , dm ³	0.05	0.35	0.65	0.3
Ionite mass	m , g	0.31	0.62	0.93	0.31

Table 8. Data element of the sorption process by the fiber VION KN-1

Test No.	Ammonium ion concentration, mol/dm ³	Solution volume, dm ³	Mass, g	Purification degree, %
1	0.002	0.05	0.31	99.40
2	0.002	0.35	0.31	98.70
3	0.002	0.65	0.31	96.40
4	0.007	0.05	0.93	99.50
5	0.007	0.35	0.93	97.35
6	0.007	0.65	0.93	83.80
7	0.012	0.05	0.62	99.05
8	0.012	0.35	0.62	58.70
9	0.012	0.65	0.62	32.00

Table 9. Test values

Concentration, mol/dm ³	Volume, dm ³	Mass, g	R, %
0.0065253940	0.35	0.3128	53.37069
0.0100000000	0.20	0.3136	57.20000
0.0115954000	0.15	0.6272	91.37589
0.0099900000	0.30	0.9378	93.42342
0.0060673890	0.45	0.6272	77.66356
0.0040765630	0.55	0.6282	84.48161
0.0019952620	0.70	0.6290	95.71635
0.0033431070	0.60	0.9347	98.04329
0.0070961730	0.65	0.9411	83.81542
0.0019319010	0.35	0.3113	89.10855
0.0078406740	0.50	0.4545	60.49453

Table 10. Predicted values. Neural network: MLP 3-5-1 (test sampling)

Observation No.	R Target	R- Output MLP 3-5-1	R- Absolute residues MLP 3-5-1	R- Squared residues MLP 3-5-1
1	53.37069	54.55938	1.188691	1.412987
2	57.20000	57.20866	0.008663	0.000075
3	91.37589	91.39071	0.014817	0.000220
4	93.42342	92.44196	0.981461	0.963265
5	77.66356	77.34208	0.321479	0.103348
6	84.48161	85.86594	1.384330	1.916369
7	95.71635	95.62773	0.088623	0.007854
8	98.04329	99.27424	1.230950	1.515239
9	83.81542	83.89240	0.076987	0.005927
10	89.10855	89.79660	0.688049	0.473411
11	60.49453	60.89747	0.402934	0.162356