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## USE OF NATURAL SORBENTS TO REDUCE NITRATE CONTENT IN NATURAL JUICE

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### Abstract

Today, the search for the most optimal modes of storage and processing of fresh plant products, which reduce the content of nitrates in food, remains relevant. In this work, the possibility of using natural sorbents of different nature to reduce the content of nitrates in fresh vegetable juices was studied. The juice was separated from the sorbent by filtration and the filtrate was analyzed for nitrate content by the ionometric method on a pH meter-ionometer "Expert-001". The sorption activity of sorbents was calculated according to the indicators of the total binding index (R, %) and distribution coefficient (D), sorption isotherms were constructed and their thermodynamic parameters were determined. The objects of the study were fresh vegetable juices; dispersed mineral bentonite, gelatin and synthetic flocculant polyoxyethylene were used as sorbents. Determination of the total binding index (R, %) and the sorbents' distribution coefficient (D) of nitrates from juices showed that the use of sorbents reduces the nitrate content by an average of 1.2–1.9 times. The maximum sorption activity at 3 hours of contact with the juice occurred with bentonite (total nitrate binding 16.1%; distribution coefficient 19.0), the minimum – with the synthetic sorbent polyoxyethylene (total binding 5.5%; distribution coefficient 5.8). It is established that the experimental production of Langmuir adsorption isotherms and the calculation of thermodynamic properties of sorption allow to optimize the choice of sorbents to reduce nitrate contamination of fresh vegetable juices. The most effective sorbents for reducing the content of nitrates in natural juices are bentonite and gelatin. The given conditions of processing natural juices by sorbents can be recommended for prevention of nitrate pollution of natural juices at their production.

**Keywords:** vegetable juices; nitrate pollution; sorbent; thermodynamic parameters; sorption.

## ВИКОРИСТАННЯ ПРИРОДНИХ СОРБЕНТІВ ДЛЯ ЗНИЖЕННЯ ВМІСТУ НІТРАТІВ У НАТУРАЛЬНИХ СОКАХ

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### Анотація

На сьогоднішній день актуальним залишається пошук оптимальних режимів зберігання і переробки свіжої рослинної продукції, які забезпечують зниження вмісту нітратів у продуктах харчування. В даній роботі вивчалась можливість використання природних сорбентів різної природи для зниження вмісту нітратів в свіжих овочевих соках. Шляхом фільтрування відділяли сік від сорбенту та аналізували фільтрат на вміст нітратів іонометричним методом на рН-метрі-іонометрі «Експерт-001». Сорбційну активність сорбентів розраховували за показниками загального зв'язування (R, %) та коефіцієнта розподілення (D), будували ізотерми сорбції та визначали їх термодинамічні параметри. Об'єктами дослідження були: свіжі овочеві соки; в якості сорбентів використовували дисперсний мінерал бентоніт (БЕН), желатину (ЖЕЛ) та синтетичний флокулянт поліоксиетилен (ПОЕ). Визначення показника загального зв'язування (R, %) та коефіцієнта розподілення (D) нітратів із соків сорбентами показали, що використання сорбентів призводить до зменшення вмісту нітратів у середньому в 1.2–1.9 разу. Максимальну сорбційну активність при 3 годинному контакті з соком мав бентоніт (загальне зв'язування нітратів 16.1 %; коефіцієнт розподілення 19.0), мінімальну – синтетичний сорбент поліоксиетилен (загальне зв'язування 5.5 %; коефіцієнт розподілення 5.8). Встановлено, що експериментальне одержання ізотерм сорбції Лангмюра та розрахунок термодинамічних показників сорбції дозволяє оптимізувати вибір сорбентів для зменшення нітратного забруднення свіжих овочевих соків. Найбільш ефективними сорбентами для зниження вмісту нітратів у натуральних соках є бентоніт та желатина. Наведені умови обробки натуральних соків сорбентами можна рекомендувати для профілактики нітратного забруднення натуральних соків при їх виробництві.

**Ключові слова:** овочеві соки; нітратне забруднення; сорбент; термодинамічні показники; сорбція.

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## ИСПОЛЬЗОВАНИЕ ПРИРОДНЫХ СОРБЕНТОВ ДЛЯ СНИЖЕНИЯ СОДЕРЖАНИЯ НИТРАТОВ В НАТУРАЛЬНЫХ СОКАХ

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### Аннотация

На сегодняшний день остается актуальным поиск оптимальных режимов хранения и переработки свежей растительной продукции, которые обеспечивают снижение содержания нитратов в продуктах питания. В данной работе изучалась возможность использования природных сорбентов разной природы для снижения содержания нитратов в свежих овощных соках. Фильтрованием отделяли сок от сорбента и анализировали фильтрат на содержание нитратов ионометрическим методом на рН-метре-иономере «Эксперт-001». Сорбционную активность сорбентов рассчитывали по показателям общего связывания (R, %) и коэффициента распределения (D), строили изотермы сорбции и определяли их термодинамические параметры. Объектами исследования являлись: свежие овощные соки; в качестве сорбентов использовали дисперсный минерал бентонит (БЭН), желатину (ЖЕЛ) и синтетический флокулянт полиоксизтилен (ПОЭ). Определение показателя общего связывания (R, %) и коэффициента распределения (D) нитратов из соков сорбентами показали, что использование сорбентов приводит к уменьшению содержания нитратов в среднем в 1.2–1.9 раза. Максимальную сорбционную активность при 3 часовом контакте с соком имел бентонит (общее связывание нитратов 16.1%; коэффициент распределения 19.0), минимальный – синтетический сорбент полиоксизтилен (общее связывание 5.5%; коэффициент распределения 5.8). Установлено, что экспериментальное построение изотерм сорбции Лангмюра и расчет термодинамических показателей сорбции позволяет оптимизировать выбор сорбентов для уменьшения нитратного загрязнения свежих овощных соков. Наиболее эффективные сорбенты для снижения содержания нитратов в натуральных соках – бентонит и желатина. Условия обработки натуральных соков сорбентами, установленные в работе, могут быть рекомендованы для профилактики нитратного загрязнения натуральных соков при их производстве.

*Ключевые слова:* овощные соки; нитратное загрязнение; сорбент; термодинамические показатели; сорбция.

### Introduction

The problem of nitrate accumulation in plant raw materials and products made from them is associated with the daily use of mineral fertilizers, chemicalization of agriculture and environmental degradation. According to the Ministry of Health of Ukraine, the content of nitrates in 10 % of plant products always exceeds the maximum permissible levels, in this regard, the rationing of the amount of nitrates in vegetable products was introduced.

Per 1 kg of body weight, the daily permissible dose of nitrates does not exceed 3.7 mg, and nitrite – 0.2 milligrams per kg of body weight. The main part of nitrates (70 %) is consumed with vegetables, about 20 % – with drinking water [1; 2].

Agriculture in the modern world cannot provide environmentally friendly products. Therefore, the search for and development of measures to reduce the intake of nitrates in the human body is one of the most pressing problems today. One of the measures to reduce nitrates is to grow different varieties of crops with low ability to accumulate nitrogen compounds [3–5]. To date, the topical issue is the search for the most optimal modes of storage and processing of fresh plant products, which reduce the content of nitrates in food [6]. The accumulation of high levels of nitrates in plants occurs when they are

absorbed in excess. It is proved that with an increase in the amount of protein in plants and with a decrease in the amount of sugars – the level of nitrates increases. During the cultivation of vegetable crops, the optimal dose of nitrates is considered to be 100 kg/g [7–9].

The soil type and its composition significantly affect the quantitative composition of nitrates in vegetables. The amount of fertilizers is reduced for soils with low content of phosphorus, potassium and trace elements; for ultra-acidic soils (pH < 4) and those with a high content of mineral nitrogen fertilizers are generally prohibited for use. The accumulation of nitrates in vegetables is greatly influenced by the relative humidity, especially when growing crops on irrigated soils [10–12].

Recently, the facts of high nitrate content in water, vegetables, fruits and juices have been noted. Thus, the load of nitrates on a person increases, which is potentially dangerous for the integrated indicators of their health [13–15].

One of the promising areas for improving the technology of juice purification is the use of natural dispersed minerals of Ukrainian deposits, which have sufficient adsorption capacity, can be modified, regenerated and disposed of [16; 17]. They are cheaper than synthetic ones, and do not require long preparation. It is known that natural adsorbents, including palygorskite, are used to

clarify and stabilize blends in winemaking, bleaching and refining oils, wastewater treatment, as fillers for insectofungicides, as well as medicines [18; 19].

As the problem of removing nitrates from food is still not fully solved, we studied the possibility of using natural sorbents of different kinds to reduce the content of nitrates in natural juices.

### Materials and Methods

The objects of the study were: freshly prepared carrot and tomato juices; as sorbents we used dispersed mineral bentonite, gelatin and synthetic flocculant polyoxyethylene.

Fresh vegetable juice was prepared using an electromechanical juice squeezer. The resulting juice was collected in one container and mixed. 1 g of sorbent was added to the conical flask and 100 cm<sup>3</sup> of vegetable juice was added. The flasks were periodically shaken for 3 hours. After that, the juice was separated from the sorbent by filtration and the filtrate was analyzed for nitrate content by the ionometric method on a pH meter-ionometer "Expert-001" according to the methodology [20].

A membrane-based nitrate ion-selective electrode and a silver chloride reference electrode were prepared for operation according to the passports to these electrodes. Next, the ionometer was calibrated according to the calibration solutions of potassium nitrate in pC<sub>NO<sub>3</sub><sup>-</sup></sub> units according to the operating instructions of this device.

Ionometric method for determining the content of nitrates in vegetable crop products is based on measuring the activity of nitrate ion by an ion-selective electrode in a salt suspension of 1% solution of potassium alum at a ratio of sample and alum solution 1:5. To do this, a 10 cm<sup>3</sup> aliquot was pipetted from the obtained juice filtrates, it was placed in a 100 cm<sup>3</sup> beaker and 50 cm<sup>3</sup> of a 1% solution of potassium alum was added when stirring on a magnetic stirrer for 1-2 minutes. The concentration of nitrate ion in the resulting suspension was measured with the prepared electrodes.

The sorption activity of the sorbents was calculated from the indicators the total binding index (R, %) and the distribution coefficient D.

The total binding index was calculated by the formula (1):

$$R = \frac{C_0 - C}{C_0} \cdot 100, \quad (1)$$

where:

C<sub>0</sub> – initial concentration of nitrates in vegetable juice (mg / dm<sup>3</sup>);

C – equilibrium concentration of nitrates in vegetable juice (mg / dm<sup>3</sup>);

V – the volume of the analyzed juice (cm<sup>3</sup>);

100 – percentage conversion factor (%).

The distribution coefficient was calculated by the formula (2):

$$D = \frac{R}{100 - R} \cdot \frac{V}{m}, \quad (2)$$

where:

R – extraction degree (%);

V – volume of the analyzed juice solution (cm<sup>3</sup>);

m – mass of the sorbent (g).

To construct the sorption isotherms, a concentration series with a nitrate content in vegetable juice of 1.61 g / dm<sup>3</sup>, 0.896 g / dm<sup>3</sup>, 0.47 g / dm<sup>3</sup>, 0.252 g / dm<sup>3</sup>, 0.146 g / dm<sup>3</sup> was created. After 3 (or 24) hours of sorption, the concentration of nitrates was determined as described above, in the coordinates of the concentration of sorbed substance (S) ÷ equilibrium concentration (C). Indicator (S) was calculated by the formula (3):

$$S = \frac{C_0 - C}{m} \cdot V, \quad (3)$$

where:

C<sub>0</sub> – initial concentration of nitrates in vegetable juice (mg / dm<sup>3</sup>);

C – equilibrium concentration of nitrates in vegetable juice (mg / dm<sup>3</sup>);

V – volume of the analyzed juice (cm<sup>3</sup>);

m – mass of the sorbent (g).

To calculate the thermodynamic sorption constants (limit value of sorption S<sub>∞</sub>, sorption equilibrium constant K), the obtained isotherms were graphically represented in the coordinates 1/S ÷ 1/C. The graph in these coordinates is a straight line (Fig. 1).

For this purpose, we obtained the experimental isotherms (Figs. 3, 4) in coordinates 1/S ÷ 1/C. The graph in these coordinates is a straight line. The limit value of sorption (S<sub>∞</sub>) is calculated as a line segment 1/S<sub>∞</sub>, which is cut off when extrapolating the straight line to zero concentration.

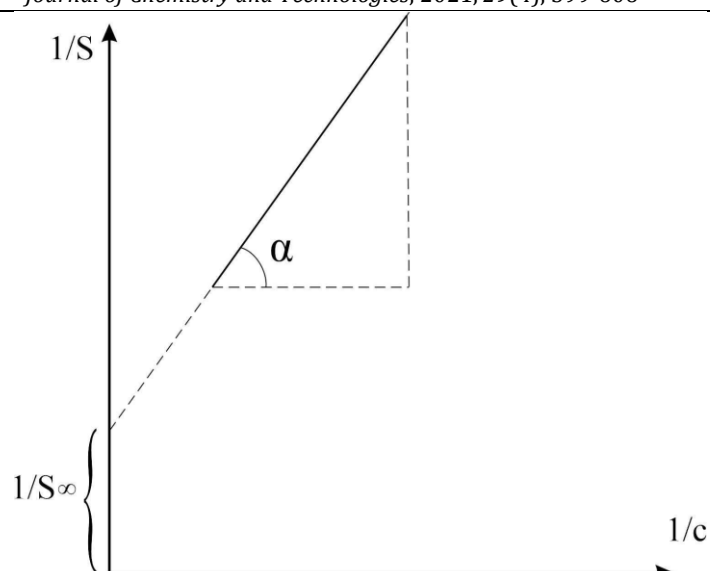


Fig. 1. Graphical method of calculating the sorption constant

Sorption equilibrium constant ( $K_{eq}$ ) is calculated by the tangent of the angle of inclination ( $\text{tg}\alpha$ ) of the straight line by the formula (4, 5):

$$\text{tg}\alpha = 1/S_{\infty} * K_{eq}; \quad (4)$$

$$K_{eq} = 1/S_{\infty} * \text{tg}\alpha. \quad (5)$$

The isobaric-isothermal potential, or Gibbs energy, was calculated by the formula (6):

$$\Delta G = - R T \ln K_{eq}, \quad (6)$$

where:

$\Delta G$  – isobaric-isothermal potential, or Gibbs energy, J / mol;

$R$  – universal gas constant 8.34 J / mol \* K;

$T$  – absolute temperature, K;

$K_{eq}$  – sorption equilibrium constant.

### Results and discussion

According to the obtained experimental data, the original carrot and tomato juices had a high content of nitrates. In carrot juice, the concentration of nitrates was 310 mg / dm<sup>3</sup> (at a rate of 250 mg/dm<sup>3</sup>), in tomato juice – 215 mg/dm<sup>3</sup> (at a rate of 150 mg / dm<sup>3</sup>). The use of sorbents led to a significant reduction in the content of nitrates in the studied juices by an average of 1.2–1.9 times (Table 1).

Table 1

Nitrate content (for nitrate ion) after 3 and 24 hours of sorption in carrot juice (mg / dm<sup>3</sup>)

Juice	Sorbent					
	Bentonite (BEN)		Gelatin (GEL)		Polyoxyethylene (POE)	
	3 hours	24 hours	3 hours	24 hours	3 hours	24 hours
Carrot	260.0±5.1	162.0±7.3	279.0±5.1	219.0±9.1	293.0±2.5	293.0±2.5
Tomato	187.0±5.1	109.0±9.1	193.0±2.5	160.0±5.1	191.0±7.6	191.0±7.6

Increasing the sorption time from 3 to 24 hours had a positive effect on reducing the amount of nitrates in juices. For instance, the study found that with prolonged sorption of bentonite sorbent in tomato juice, the nitrate content decreased from 187 mg/dm<sup>3</sup> to 109 mg/dm<sup>3</sup>, in carrot juice – from 260 mg/dm<sup>3</sup> to 162 mg/dm<sup>3</sup> respectively.

The experimental results of determining the total binding ( $R$ ,%) and the distribution coefficient ( $D$ ) of nitrates from juices by the

investigated sorbents are given in Fig. 2, 3 and Table 2.

According to research results of carrot juice, bentonite had the maximum sorption activity, in which the total binding of nitrates was 16.1 % at 3 hours of contact with the juice. This is also confirmed by the largest value of the distribution coefficient (19.0) for this sorbent. The minimum activity was shown by the synthetic sorbent polyoxyethylene, whose total binding at 3 hours of contact with the juice was only 5.5 %, and the distribution coefficient was 5.8.

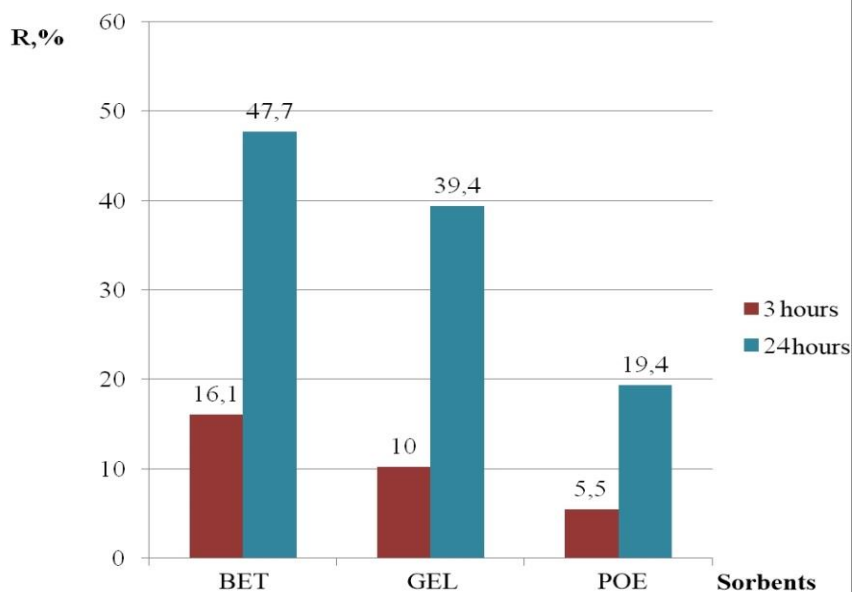


Fig. 2. Total binding (R, %) of nitrates by sorbents from carrot juice after 3 and 24 hours of sorption ( $\text{mg} / \text{dm}^3$ )

Distribution coefficients (D) of nitrates on sorbents in vegetable juices (according to average data) Table 2

№	Juice	Sorbent					
		Bentonite (BET)		Gelatin (GEL)		Polyoxyethylene (POE)	
		3 hours	24 hours	3 hours	24 hours	3 hours	24 hours
1	Carrot	19.0	91.2	11.1	41.6	5.8	24.1
2	Tomato	14.9	97.2	11.3	34.4	12.6	23.5

In the case of tomato juice at 3 hours of contact, the sorption activity of bentonite remained the highest, but decreased compared to its activity in carrots by an average of 1.25 times (total binding 13 %, partition coefficient 14.9).

Gelatin in both studied juices was similar in activity on two indicators, and the sorption capacity of polyoxyethylene increased in tomato on average by 2.1 times (total binding 11.2 %, partition coefficient 12.6).

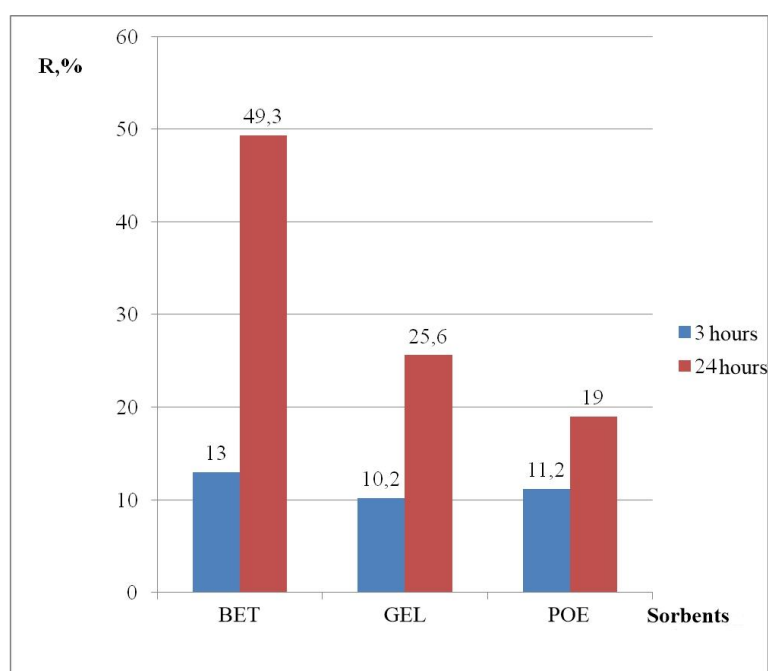


Fig. 3. Total binding (R, %) of nitrates by sorbents from tomato juice after 3 and 24 hours of sorption ( $\text{mg} / \text{dm}^3$ )

According to the results, bentonite had the maximum sorption activity, in which the total binding of nitrates was 16.1% at 3 hours of contact with the juice. This is also confirmed by the largest value of the distribution coefficient (19.0) for this sorbent.

The minimum activity was shown by the synthetic sorbent polyoxyethylene, the total binding of which at 3 hours of contact with the juice was only 5.5 %, and the distribution coefficient was 5.8.

Thus, at 24 hours of contact, sorption on bentonite increased significantly in both carrot and tomato juices and was, respectively: total

binding 47.7 % and 49.3 %; distribution coefficients – 91.2 and 97.2. At the same time, the activity of polyoxyethylene increases in carrot and tomato juices, respectively, by 19.4 % and 19.0 % in terms of total binding, and the distribution coefficient increases by 24.1 and 23.5, respectively.

The activity of gelatin was close to bentonite, in the case of carrot juice, and was close to polyoxyethylene, in the case of tomato juice.

The obtained data of isotherms of nitrates sorption from vegetable juices by the studied sorbents (Figs. 4, 5) allows to obtain information about the mechanism of sorption.

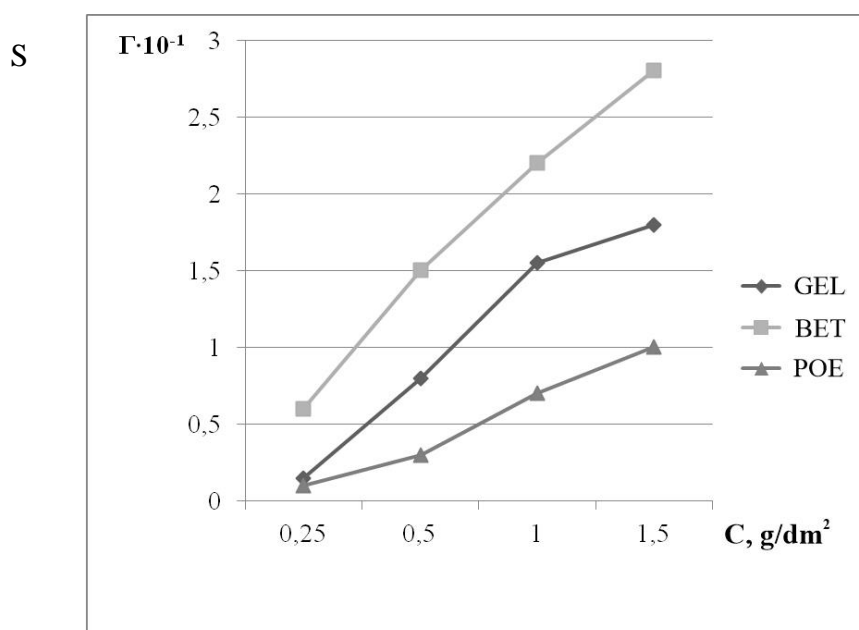


Fig. 4. Isotherms of nitrates sorption from carrot juice

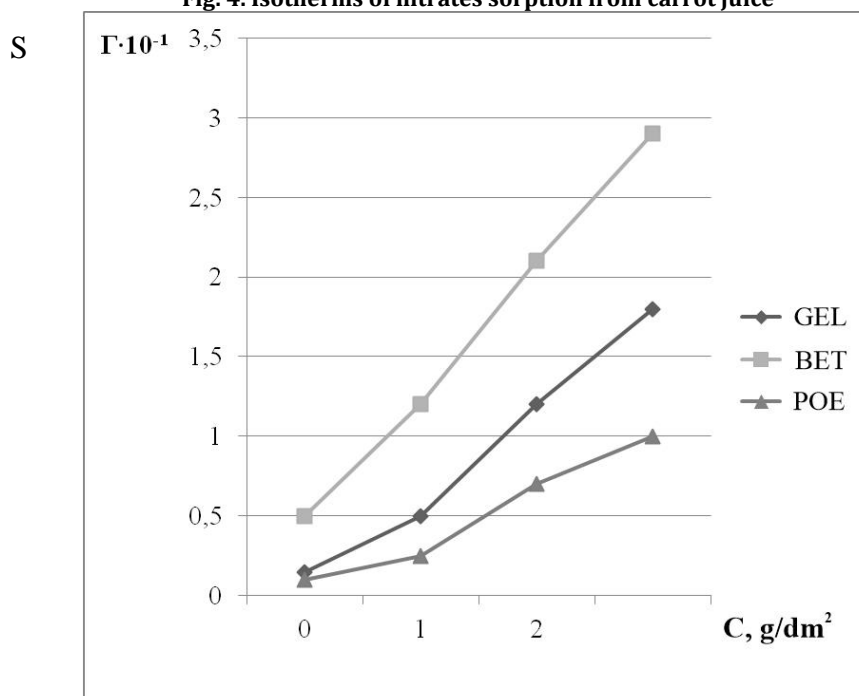


Fig. 5. Isotherms of nitrates sorption from tomato juice

All the obtained isotherms are curved in the initial section relative to the concentration axis. According to the Brunauer classification, this type of isotherm belongs to type 1, the Langmuir isotherm, which is associated with monomolecular adsorption taking place with a parallel orientation on the sorbent of the solute – the adsorbent. This suggests that the sorption of nitrates from vegetable juices by the studied sorbents takes place according to the monomolecular adsorption type. Comparing

isotherms, we see a decrease in the steepness of the isotherms, a decrease in the efficiency of sorption in the transition from bentonite to polyoxyethylene. So, for example, in case of carrot juice indicators of sorption of nitrates decrease in a row of bentonite - gelatin - polyoxyethylene:  $S = 2.8 \cdot 10^{-1}$ ;  $1.7 \cdot 10^{-1}$ ;  $1.0 \cdot 10^{-1}$  respectively.

The results of the graphical calculation of thermodynamic sorption constants – limit value of adsorption ( $S_{\infty}$ ), Gibbs energy ( $\Delta G$ ) and sorption constants ( $K$ ) – are presented in Table 3.

Table 3

Thermodynamic constants of nitrates sorption from vegetable juices

Constants	Carrot juice			Tomato juice		
	Bentonite	Gelatin	POE	Bentonite	Gelatin	POE
$\Gamma_{\infty}$	0.909	0.400	0.200	1.000	0.454	0.196
$\text{tg } \lambda$	3.750	7.179	8.816	3.780	8.261	8.287
$K$	0.293	0.348	0.567	0,265	0.268	0,616
$\Delta G$ , J / mol	-2990.000	-2569.900	-1334.400	-3237.832	-3210.500	-1181.450

The Gibbs energy is a thermodynamic characteristic that shows the degree of affinity of nitrate ions to the surface of the sorbent and can serve as a criterion for the efficiency of the sorption process.

The increase in the efficiency of nitrate sorbent binding in the polyoxyethylene – gelatin – bentonite row is accompanied by the increase in the chemical affinity between nitrates and sorbents, which is manifested in a decrease in the Gibbs energy. For example, the Gibbs energy decreases from -1334.4 J / mol to -2990.0 J / mol in the transition from polyoxyethylene to bentonite in the case of carrot juice, which is reflected in the sorption isotherms. In this case, bentonite is characterized by higher absolute values of the Gibbs energy compared to other sorbents in the cases of both juices, which indicates its greater sorption capacity with nitrates. This is confirmed by the experimental values of the distribution coefficient ( $D$ ) of nitrates on the sorbents (Table 2).

Thus, we can assume that the higher sorption of nitrates on the dispersed mineral bentonite is due to the presence on its surface of a much larger number of positively charged centers than on the surfaces of gelatin and polyoxyethylene. The lower activity of gelatin can be explained by the effect of pH, especially in the case of tomato juice, as the pH of tomato juice is close to the isoelectric point of gelatin. If it is necessary to reduce the concentration of nitrates, it is possible to recommend treatment of vegetable juices with dispersed mineral bentonite, gelatin,

polyoxyethylene (10 g of sorbent per 1 liter of juice) with a contact duration of 3 to 24 hours.

## Conclusions

The appropriateness of using natural sorbents to reduce the content of nitrates in natural juices has been experimentally confirmed. The research results indicate the possibility of using experimentally obtained Langmuir isotherms and the results of calculation of thermodynamic sorption parameters to optimize the choice of sorbents in order to reduce nitrate contamination of fresh vegetable juices.

It has been experimentally proven that the most effective sorbents to reduce the content of nitrates in natural juices are bentonite and gelatin, which are widely used in the food industry. The given conditions of processing of natural juices by sorbents can be recommended for prevention of nitrate contamination in natural juices during their production that has the important sanitary-and-hygienic as well as valeological value.

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