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Interaction of L-Ascorbic Acid with Activated Carbon: Kinetic Studies and the Effect of pH

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Abstract—This article investigates the interaction of vitamin C with commercial activated Granucol-type carbons and newly produced AC-C coal at 25 and 35°C. L-Ascorbic acid molecules have been shown to retain on the surface of coals due to physical interactions. The adsorption kinetics is perfectly described by the pseudo-second-order kinetic model. The adsorption rate is mainly determined by the intraparticle diffusion. The process of adsorption of vitamin C on activated carbon is accompanied by a change in the pH of the system. The absorption is highly dependent on the pH of the solution, decreasing as the pH of the solution increases prior to contact with the carbon. In the carbon phase, ascorbic acid oxidizes to dehydroascorbic acid.

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1. INTRODUCTION

L-Ascorbic acid, known as vitamin C, is a vital component for the proper functioning of the human body. L-ascorbic acid is important in the synthesis of neuropeptides, collagen, carnitine, dopamine, norepinephrine and adrenaline in the nervous system or adrenal glands [1, 2]. Vitamin C has an antioxidant function [3–5]. Vitamin C is a coenzyme in many metabolic processes. In addition, ascorbic acid is needed for wound healing and tissue growth, for the implementation of the function of the adrenal glands, the secretion of hormones and interferons, and the metabolism of folic acid, tyrosine and phenylalanine. Also it increases iron absorption and reduces blood cholesterol level and helps to reduce blood pressure [6]. Vitamin C serves as a reagent for the preparation of many materials in the pharmaceutical and food industry [7–10].

The human body does not have the ability to synthesize vitamin C, that is why it depends on exogenous sources of vitamin. According to [11], depending on the age and certain physiological conditions, recommended daily reference intakes are 60 mg for nonsmokers and 95 mg for smokers' women, 75 mg for nonsmokers and 110 mg for smokers' men. In another paper [12], it is recommended for children to use daily 45–65 mg of vitamin C, for women 75 mg and for men 90 mg. It is known that the main sources of Vitamin C are fruits, vegetables and berries. Many beverages—wines, juices, beer, etc. also contain vitamin C. These products are processed with activated carbon for clarification, stabilization and a good commercial appearance [10, 13–16]. But apart from the expected effects, during the active coal treatment, the sorbent interacts with other components including vitamins. In the article [17], the results of the investigation of the adsorption of vitamin C from aqueous solutions on activated carbon entrapped in alginate were discussed. It was shown that the Freundlich sorption model for ascorbic acid fits adsorption data better than the Langmuir model. In this work, it is shown that the sorption of vitamin C decreases monotonically with an increase in the pH of the solution. The content of the polyphenols and of vitamin C in wines treated with activated coals decreases [15]. In this case, the alcohol content practically does not change, and the pH increases slightly. The adsorption of vitamin C on silica gel, depending on pH, passes through a maximum at pH 6 [18].

The article [19] shows that the adsorption of vitamin C on mesoporous carbon is greatly increased with increasing pH, starting with pH 5, maximum adsorption capacity being up to 50 mg/g. The processes of vitamin C interaction with sorbents, the kinetics and the stages limiting the sorption rate, has not been sufficiently studied. Sorption data depending on the pH of the solution and their interpretation are contradictory. This article presents a study of the interaction of vitamin C with commercial and recently produced AC–C activated carbons. The results of the kinetics of vitamin C sorption on active carbon and the effect of pH are discussed.

2. MATERIALS AND METHODS

L-Ascorbic acid (Sigma-Aldrich) and commercial activated carbons Granucol BI, Granucol GE, and Granucol FA, which are widely used for purification

Table 1. Characteristic parameters of Granucol type coals [23, 24]

Activated carbon	S_{BET}, m^2/g	V_{mi}, cm^3/g	V_{me}, cm^3/g	V_t, cm^3/g	V_{me}, %	Humidity, %	Ash, %
Granucol GE	1384	0.364	0.721	1.085	66.4	1.93	16.76
Granucol BI	1221	0.310	0.693	1.003	69.1	4.19	19.21
Granucol FA	1360	0.347	0.846	1.193	71	1.94	19.95

S_{BET}—the surface of the pores, V_{mi}—volume of micropores, V_{me}—the volume of the mesopores, V_t—total sorption volume of pores.

and stabilization of beverages [13, 15, 16], were used. The Granucols are made from plant-derived activated carbon powder [20]. According to [20–22], Granucol BI is used to reduce the content of tannins and polyphenols, as well as to decolorize beer and colored wines. Granucol GE is used to adsorb unwanted flavors and odors in juice, beer, wine and spirits. Granucol FA is used to reduce tannins and polyphenols and to eliminate bright colors due to browning reactions in juice and wine. In the solution, the coal granules are dispersed in powder form, most of the particles being in the range of 45–90 μm. The main parameters of used coals according to literature data [23] and as specified by the manufacturer [24] are listed in Table 1. It should be noted that the Granukol coals used in the research contained up to 15% bentonite [24].

For the comparison, an activated carbon AC–C recently obtained from apricot kernels in the laboratory of the Institute of Chemistry of the Academy of Sciences of Moldova [25] has been used. Activated carbon AC–C is predominantly mesoporous (about 70% mesoporous), with a specific surface area of about 1385 m^2/g [25].

In all experiments, a sample of coal with an approximate mass of 0.05 g was placed in contact with 50 mL of vitamin C solution. At the end of the contact period, the solution was filtered, the pH was measured, and the vitamin C content was determined.

Analysis of the solutions for vitamin content was performed using UV-Vis spectroscopy (Specrometer DR 5000 Hach–Lange, Germany) at 264 nm. IR spectra were obtained using the Bruker ALPHA spectrometer with Platinum ATR single reflection module, at wavenumbers range of 400 to 4000 cm^{-1}. The initial concentration of vitamin C solution was 1 mmol/L. The exposure time of coal with the solution varied, the final one being about 200 min. The experiments were performed at 25.0 ± 0.2 and 35.0 ± 0.2°C using a water bath.

The pH of solutions was corrected with accuracies of ± 0.1 pH units (CE pH meter, ISOLAB Laborgerate GmbH, Germany) by use of HCl or NaOH (Sigma-Aldrich) solutions. The experiments were performed without shaking the system. After the adsorption period expired, the solution was filtered using paper filters. The first portions of the filtrate after soaking the filter with the solution were discarded, and the necessary measurements were made with the rest of the filtrate.

3. RESUITS AND DISCUSSION

3.1. Adsorption Kinetics

For the kinetic studies, the amount of adsorbed vitamin C (St) as a function of the contact time of the activated carbon with the solution was calculated with Equation (1):

$$S_t = \frac{(C_0 - C_t)V}{m}, \quad (1)$$

where S_t (mmol/g) is the vitamin C sorption; C_0 and C_t (mmol/L) are the vitamin C concentration in solution before and after contact with the carbon, respectively; V (L) is the volume of the solution in contact with carbon; and m (g) is the mass of the air dried activated carbon.

The kinetic mechanism of adsorption was investigated using pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models [26, 27]. The integral PFO kinetic model (nonlinear form) is described with Eq. (2):

$$S_t = S_e(1 - e^{-k_1 t}). \quad (2)$$

where S_t and S_e (mmol/g) are the amount of vitamin C adsorbed at a time t and at equilibrium, respectively; k_1 is the rate constant in the PFO kinetic model (min^{-1}).

The linear form of the PFO kinetic model, which is expressed by the Eq. (3), allows us to determine the value of k_1:

$$\ln(S_e - S_t) = \ln(S_e - k_1 t). \quad (3)$$

The integral form of the PSO kinetic model is expressed by Eq. (4):

$$S_t = \frac{k_2 S_e^2 t}{1 + k_2 S_e t}, \quad (4)$$

where S_t and S_e have the same meaning as in Eq. (2), and k_2 (g $mmol^{-1}$ min^{-1}) is the rate constant of PSO kinetic model.

The k_2 and S_e values were determined using the linear form of the PSO kinetic model, which is expressed by the Eq. (5):

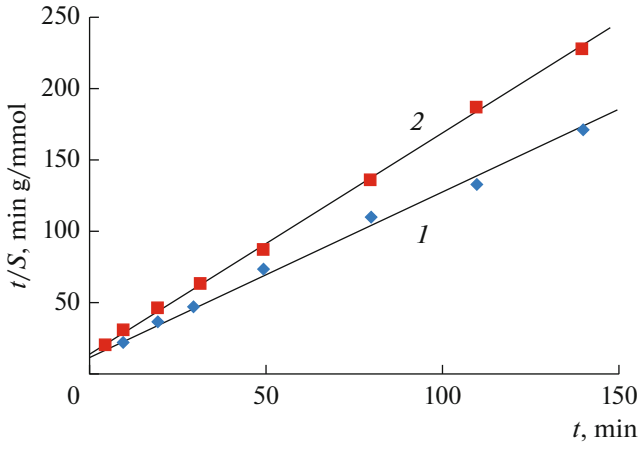


Fig. 1. Graph $t/St = f(t)$ for the adsorption of vitamin C on Granucol BI coal at 25 (*1*) and 35°C (*2*).

$$\frac{t}{S_t} = \frac{1}{k_2 S_e^2} + \frac{1}{S_e} t. \quad (5)$$

The research has shown that experimentally obtained kinetic curves do not match the PFO kinetic model, but they are very well described with the PSO kinetic model both at 25 and 35°C. If the graph obtained according to Eq. (5) is linear, then it is considered that the kinetic curve is described with the PSO kinetic model. The graph in Fig. 1 can be used as an example.

The kinetic curves of vitamin C adsorption, obtained experimentally at 25°C on the activated carbons Granucol BI, Granucol GE, Granucol FA and AC–C are shown in Fig. 2. The kinetic curves obtained at 35°C are shown in Fig. 3.

The characteristics of the kinetics of adsorption of vitamin C on activated carbon at 25 and 35°C calculated using the PSO kinetic model are presented in the Table 2. The larger is the k_2 value, the slower is the adsorption rate. As shown in Table 2, the adsorption rate of vitamin C at both 25 and 35°C is lowest for AC–C activated carbon and highest for Granucol FA. During adsorption on Granucol BI, Granucol GE and Granucol FA, pH of solutions at the time of contact with carbon, pH_t (Table 2), slightly changes from the initial pH_0 (pH of the solution before contact with the coal). It grows slightly. In the case of adsorption on AC-C carbon, the pH of the solution increases significantly. As the temperature rises up to 35°C, the pH of the vitamin C solution that comes in contact with the coals decreases slightly.

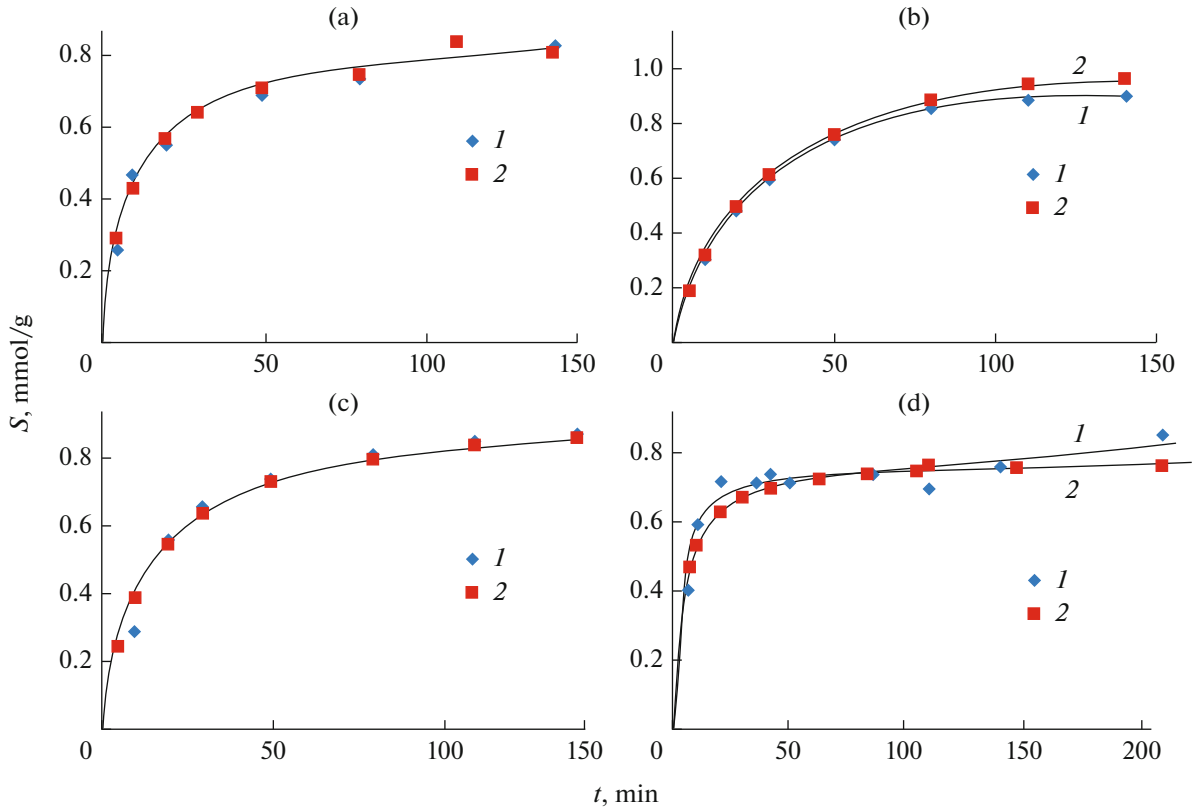


Fig. 2. Kinetic curves of vitamin C adsorption at 25°C on the activated carbons Granucol BI (a), Granucol GE (b), Granucol FA (c) and AC–C (d) obtained experimentally (1) and calculated (2) with the pseudo-second order kinetic model.

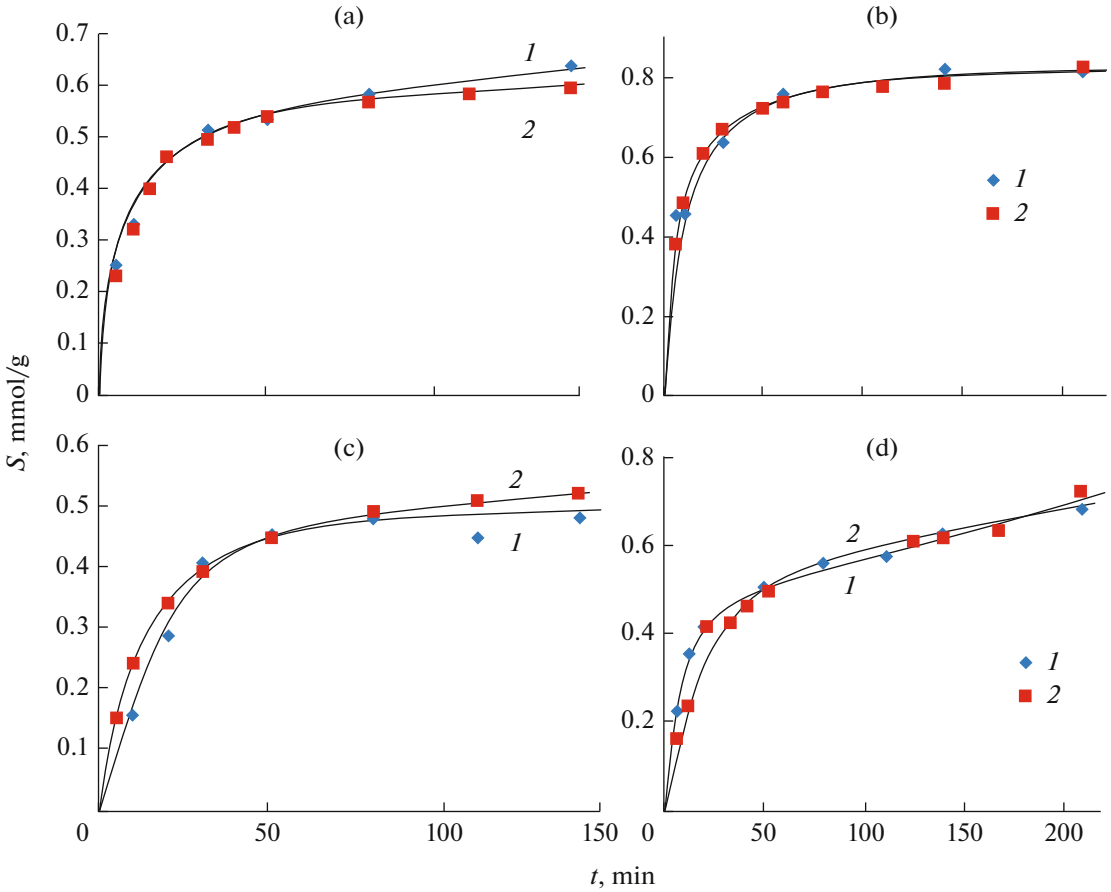


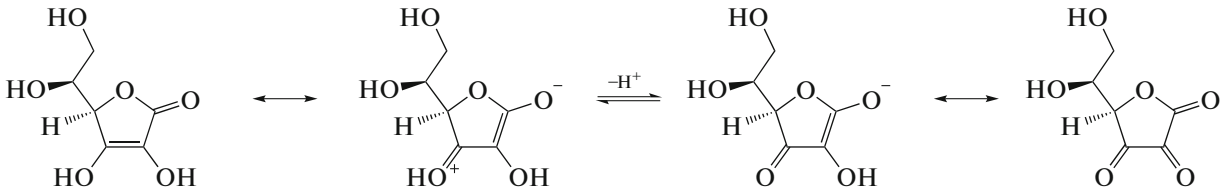
Fig. 3. Kinetic curves of vitamin C adsorption at 35°C on the activated carbons Granucol BI (a), Granucol GE (b), Granucol FA (c) and AC–C (d) obtained experimentally (*1*) and calculated (*2*) with the pseudo- second order kinetic model.

The data show that the adsorption of vitamin C at 25°C is greater than at 35°C. It shows that the adsorption of vitamin C on the investigated activated carbons is a physical process. It is important to note that the adsorption capacity of the newly produced AC–C coal has the same order of magnitude as that of the commercial coals of the Granucol-type. As can be seen from Table 2, the sorption capacity of the studied coals ranges from 130 to 207 mg/g. The absorption capacity of porous materials is much lower and varies from 0 to 5 mg/g in acidic medium (pH 5) and up to 50 mg/g in basic medium (pH 11.5) [19]. But, as it is known in the basic medium, the ascorbic acid is converted into oxyanions and, as an intermediate in the oxidation process, to dehydroascorbic acid or even to 2,3-diketogulonic acid according to Scheme 1 [28, 29].

Table 2. Kinetic parameters of adsorption at equilibrium (S_e) of vitamin C on activated carbons at 25 and at 35°C (data in brackets)

Activated carbon	S_e, mmol/g	S_e, mg/g	k_2, g/mmol min	pH_0	pH_t
Granucol BI	0.83 (0.631)	146.16 (111.12)	0.1258 (0.18)	3.84	4.17–4.31 (4.0–4.17)
Granucol GE	1.176 (0.822)	207.09 (144.75)	0.0301 (0.1725)	3.84	4.10–4.37 (4.02–4.18)
Granucol FA	0.94 (0.568)	165.53 (100.02)	0.072 (0.129)	3.87	4.16–4.38 (4.10–4.19)
AC–C	0.784 (0.723)	130.08 (127.32)	0.271 (0.0566)	3.84	5.66–6.52 (5.50–6.23)

pH_t is the pH of the solution that changes during adsorption.



Scheme 1. Scheme of the conversion of vitamin C to dehydroascorbic acid.

A decrease in the concentration of vitamin C in a solution due to its partial conversion can lead to an erroneous conclusion about an increase in adsorption in an alkaline medium.

3.2. Determination of the Step Limiting the Rate of Adsorption

The adsorption rate can be determined by: (1) external diffusion (diffusion through the liquid film around the sorbent granule), (2) internal diffusion (diffusion inside the sorbent granule), and (3) process of interaction of the sorbate molecule with the sorbent.

In the case of physical sorption, the sorbate-sorbent interactions occur very quickly; therefore, they cannot be the limiting stage of the adsorption rate [30]. The experiments were performed using a diluted solution (1 mmol/L), and the sorption rate can be limited by external diffusion. On the other hand, the vitamin C molecules are relatively large, and it can be expected that the rate of adsorption on mesoporous carbons will be determined by internal diffusion.

The diffusion mechanisms cannot be identified through PFO or PSO kinetic models. To determine the diffusion mechanism of the adsorption process, the internal diffusion model [31], expressed by Eq. (6), was used:

$$S_t = k_{id} t^{0.5} + C_i, \quad (6)$$

where k_{id} (g mmol^{-1} min^{-1}) is the intraparticle diffusion rate constant, C_i is a constant that gives an idea about the effect of boundary layer thickness: the larger C_i, the greater the contribution of the film diffusion in the rate limiting step.

If the dependence $S_t = f(t^{0.5})$ is linear, then the adsorption rate is determined by the internal diffusion. The S_t dependences on $t^{0.5}$ for the adsorption of vitamin C at 25 and 35°C on investigated activated carbons Granucol BI, Granucol GE, Granucol FA and AC–C are presented graphically in Fig. 4.

As can be seen from Fig. 4, the dependence of S_t on $t^{0.5}$ is well fitted by two linear sections. The adsorption of vitamin C on activated carbons probably occurs in two stages.

It was shown in [32] that the S_t dependence on t in the case of adsorption of methylene blue and chromium(III) from aqueous solution on the pomelo peel consists of even three lines. In most cases, the S_t graph does not go through the origin of the coordinate system and the presence of the boundary layer effect is characterized by the nonzero value of C_i. It follows from the graphs in Fig. 4 that the internal diffusion is not the only diffusion mechanism, but we cannot draw a very precise conclusion about the diffusion mechanism. Therefore, we decided to determine the diffusion mechanism by another method.

Internal diffusion, as the main factor influencing the sorption rate, is also determined from the graph of Eq. (7) [33]:

$$-\ln(1 - F) = f(t), \quad (7)$$

where $F = S_t/S_e$.

The results are shown in Figs. 5 and 6. If the $-\ln(1-F)$ dependence on t is a straight line, then the adsorption rate is determined by the external diffusion. As can be concluded from Figs. 5 and 6, the internal diffusion is the dominant limiting step in the rate of vitamin C adsorption on activated carbons.

3.3. Adsorption as a Function of Solution pH

As mentioned earlier, the solution pH changes during the adsorption of vitamin C by the active carbons (Table 2). Changes in pH can affect the quality of liquids when processed with activated carbon. Therefore, it is necessary to know how the pH will change at equilibrium (pH_e) depending on the initial pH (pH_0) of the solution that is in contact with the carbons.

For the investigation, approximately 0.05 g coal samples contacted with 50 mL of a 1 mmol/L solution of vitamin C at 25°C for 5 hours. The results are shown graphically in the Fig. 7. Figure 7 shows that up to about pH_0 6, the pH_e increases. After pH_0 6, pH_e remains almost constant and approximately equal to 6 in the case of Granucol coals.

During the contact of AC–C activated carbon with the solution of vitamin C, pH_e as a function of pH_0 changes in the same way, but the dependence is more pronounced (Fig. 7b, line *2*).

It is interesting to know which factor changes the pH of the solution: activated carbon, vitamin C, or the process of sorbent–sorbate interaction. It was found that when keeping the solution of 1 mmol/L vitamin C at 25°C for 6 hours, the pH practically did not change, being 3.87. Upon contacting of 0.055 g of Granucol BI

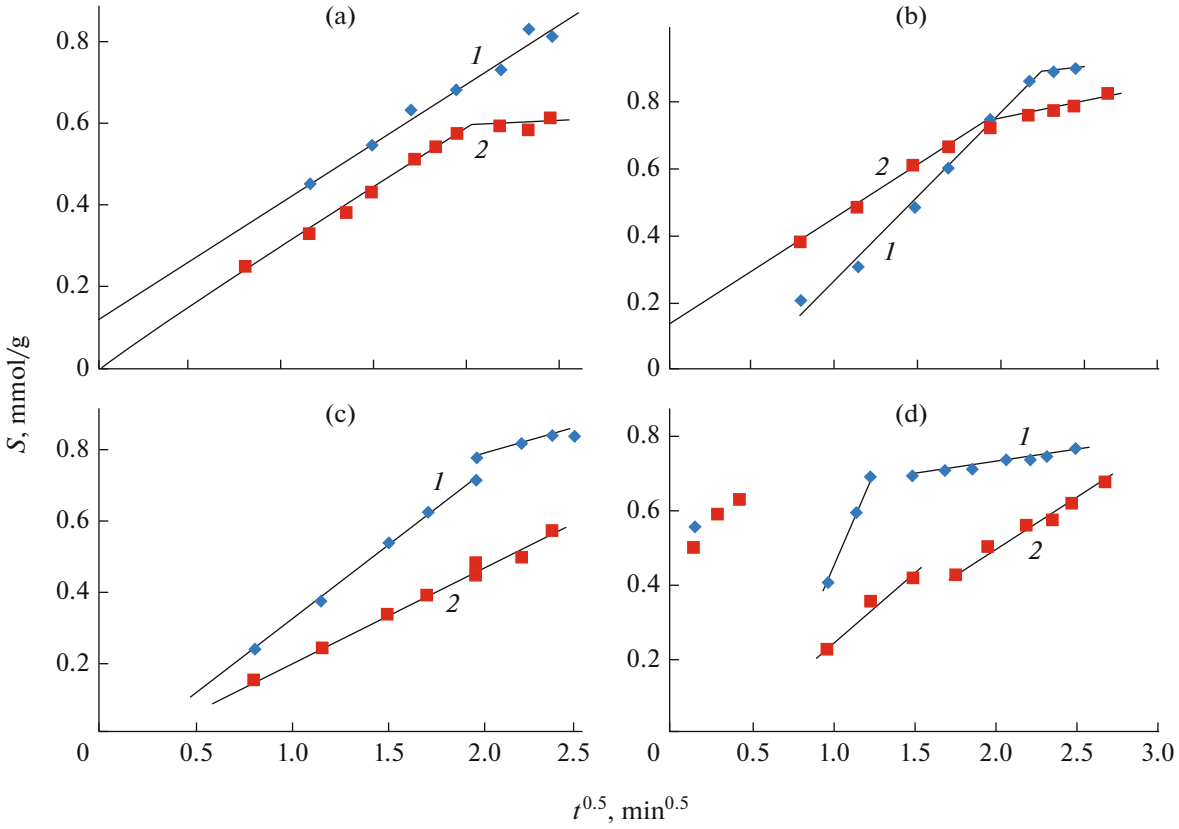


Fig. 4. Graph $S_t = f(t^{0.5})$ for the adsorption of vitamin C on Granucol BI (a), Granucol GE (b), Granucol FA (c) and AC–C (d) at 25 (*1*) and 35°C (*2*).

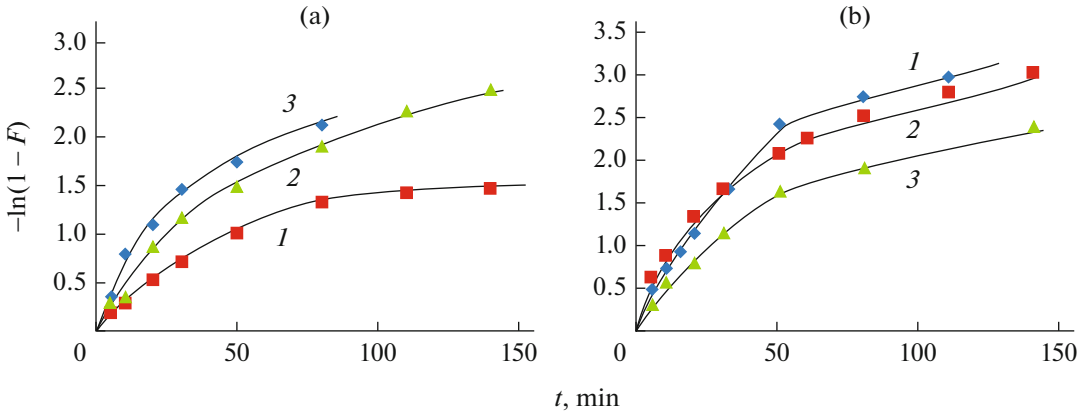


Fig. 5. Graph $-\ln(1 - F) = f(t)$ for the adsorption of vitamin C on activated carbon Granucol BI (1), Granucol GE (2) and Granucol FA (3) at 25 (a) and 35°C (b).

with 50 mL of distilled water at 25 and 35°C for 6 h, the pH changed from 5.75 to 4.5 and 4.53, respectively.

But when 0.054 g of AC–C carbon was in contact with 50 ml of distilled water for 6 hours, the pH changed from 5.75 to 8.47 at 25°C and to 9.6 at 35°C. Thus, Granucol carbons lower the pH of the vitamin C solution, while AC–C leads to a significant increase in pH. But does the adsorption process influence the pH of the vitamin C solution? The data in Fig. 7 clearly show that the sorption process affects solution pH. Therefore, it can be expected that the adsorption

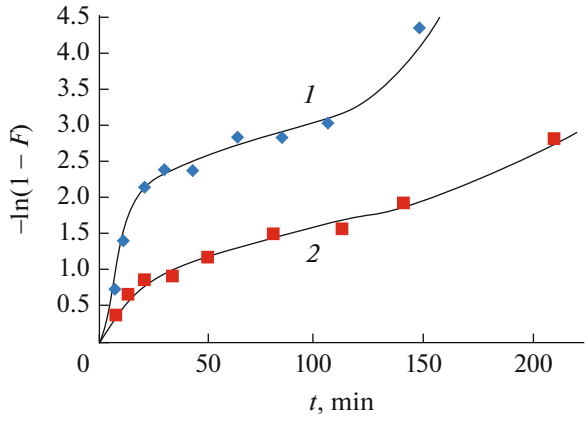


Fig. 6. Graph $-\ln(1-F) = f(t)$ f for the adsorption of vitamin C on activated carbon AC–C at 25 (*1*) and 35°C (*2*).

of vitamin C on activated carbon will depend on the pH of the solution.

The adsorption of vitamin C on Granucol and AC–C carbons depending on the initial pH (pH_0) and the equilibrium pH (pH_e) is shown in Fig. 8. Figure 8 shows that as the pH_0 increases, the adsorption of vitamin C decreases. The adsorption decreases with an increase in pH_e as well. In almost all cases, the adsorption of vitamin C on the activated carbons as a function of pH_e is higher than at the same value. At pH_0 close to 6, the adsorption suddenly decreases, and then remains almost constant at further increase in pH_0. This is explained by the fact that the pH_e remains practically constant regardless of the pH_0 value higher than 6 (Fig. 7).

The change in pH_e and adsorption as a function of pH_0 is conditioned by 2 processes: the change of the pH of the vitamin C solution by coal and the dissociation of the Vitamin C molecules as an acid. The pK_a for L-ascorbic acid is 4.2. In the basic medium, vitamin C molecules turn into oxyanions [28]. Depending on the pH in the system, a dynamic equilibrium is established between electrically neutral and ionized particles. On the other hand, it is known that activated carbons may contain some acidic groups [34]. In a basic medium, these groups are deprotonated, converting carbons into polyanions. The existence of bentonite in Granucol coals also influences the pH of the system. As a result of these processes, the pH in the "coal-vitamin C solution" system decreases, and as a result of the interaction of particles with the same charges, the sorption of the vitamin decreases. A decrease in the adsorption of vitamin C on activated carbon as a function of pH was also observed in [17], but the authors explained it differently.

According to the IR spectrum (Fig. 9), the coal does not contain obvious amounts of acidic groups. So their influence cannot be significant.

But, as mentioned earlier, Granucol coals contain up to 15% bentonite, a complex mineral whose composition is determined by the content of montmorillonite in clay, which has the formula $Si_8Al_4O_{20}(OH)_4 \cdot nH_2O$ [35]. In the used coals, traces of Fe(III) were also detected.

Spectrum 1 in Fig. 9 confirms the existence of bentonite in coals: the bands at 1034, 520, and 465 cm^{-1} are attributed to Si–O stretching for Si–O–Al bending [36]. Absorption in the range 2500–3500 cm^{-1} refers to the OH groups of both bentonite and water compounds. The bands in spectrum 3 (Fig. 9) at 2972 and 2900 cm^{-1} are attributed to the OH groups of vitamin C which was retained by coal. The intensive band at 1658 cm^{-1} (spectrum 3, Fig. 9) is attributed to the stretching vibrations of C=C in lactone ring. And the bands at 1139, 1111, 1075, and 1043 cm^{-1} are attributed to C–O–C stretch [37]. If we compare spectra 2 and 3 in Fig. 9, the band at 1658 cm^{-1} disappears

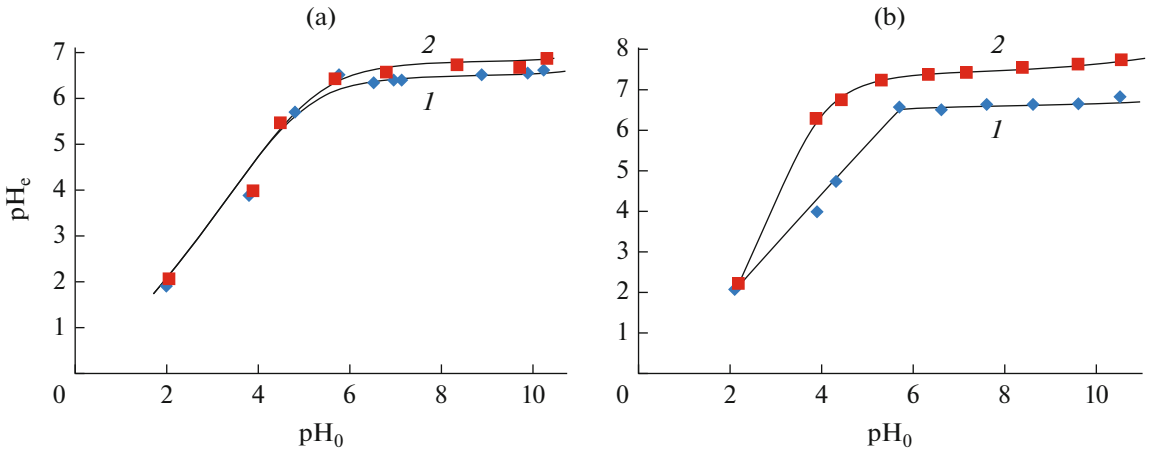


Fig. 7. Variation of pH_e with pH_0 of the solution during the sorption of vitamin C at 25°C on the activated carbon (a): Granucol BI (*1*), Granucol GE (*2*); (b): Granucol FA (*1*) and AC–C (*2*).

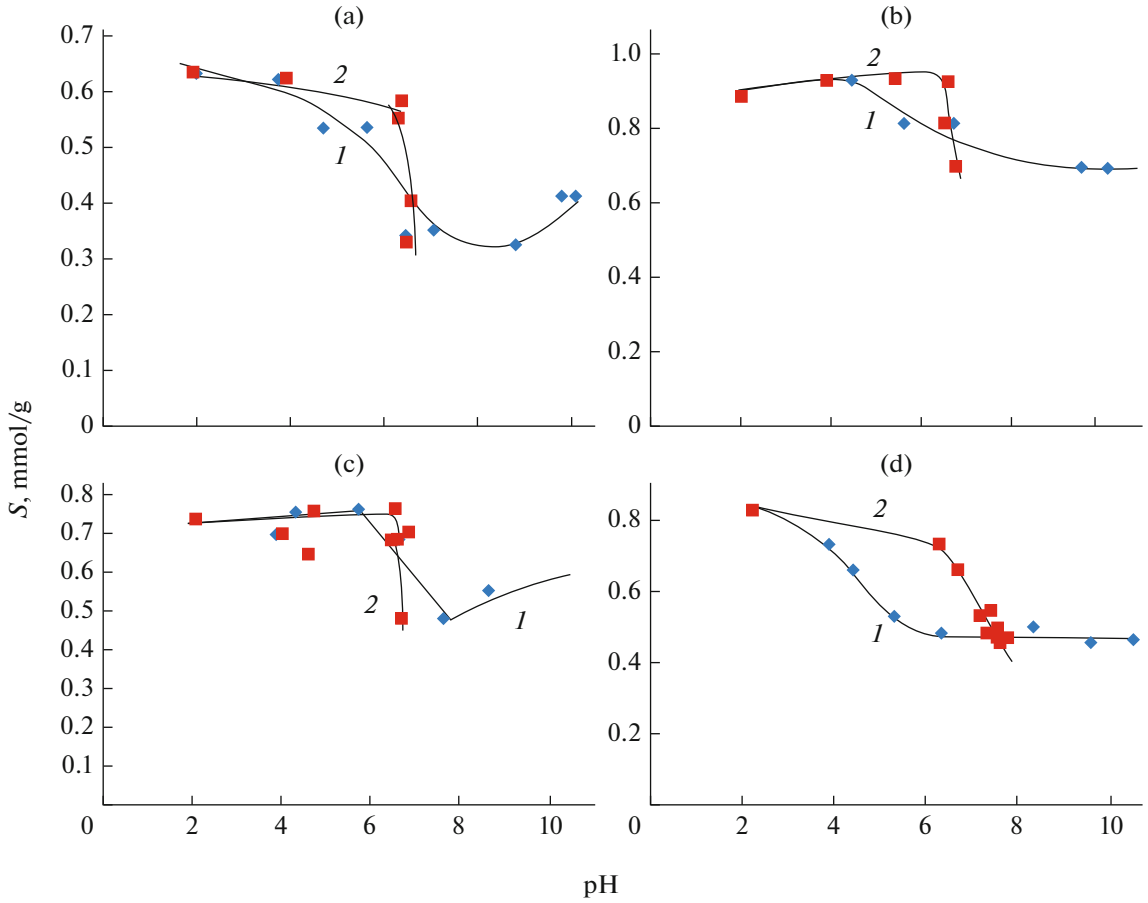


Fig. 8. Adsorption of vitamin C at 25°C on activated carbon Granucol BI (a), Granucol GE (b), Granucol FA (c) and AC–C (d), depending on the initial pH (*1*) and the equilibrium pH (*2*) of the solution.

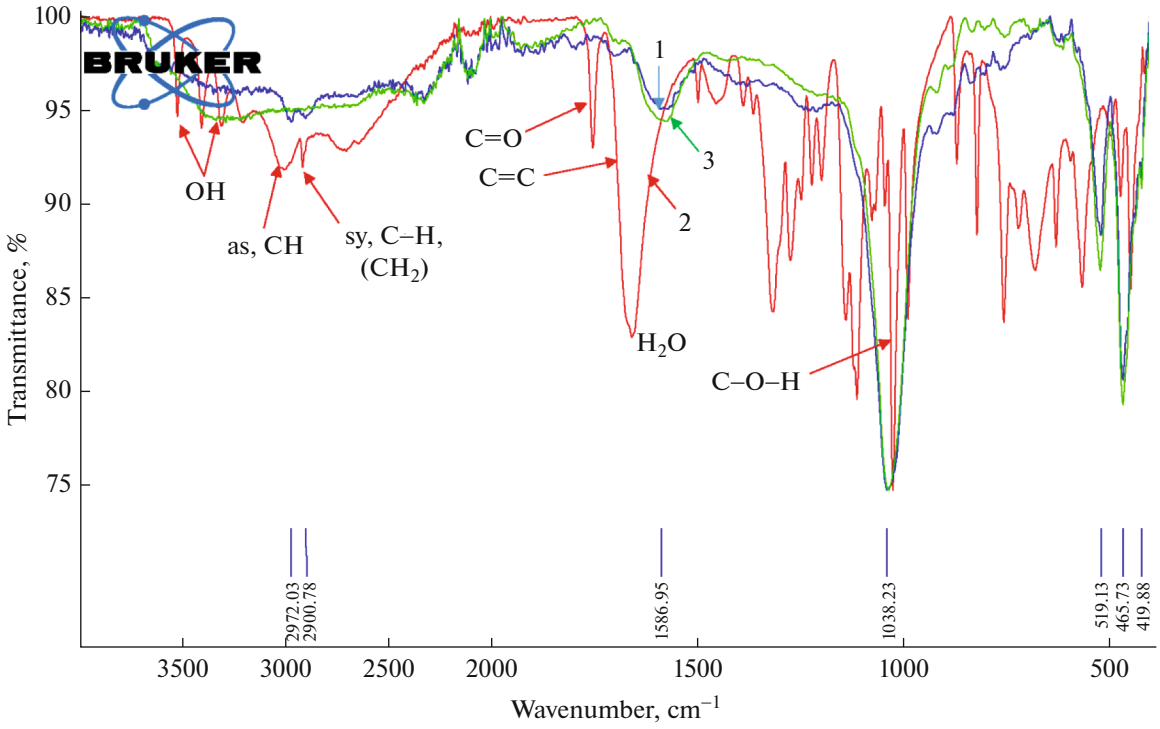


Fig. 9. IR spectra of Granucol BI coal samples before (1) and after (2) adsorption and IR spectrum of vitamin C (3).

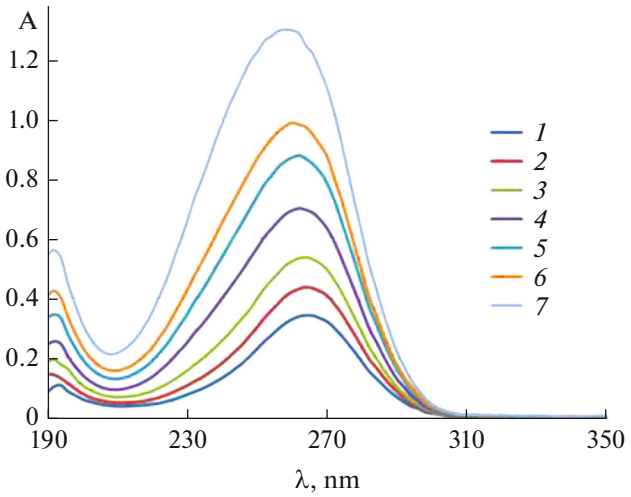


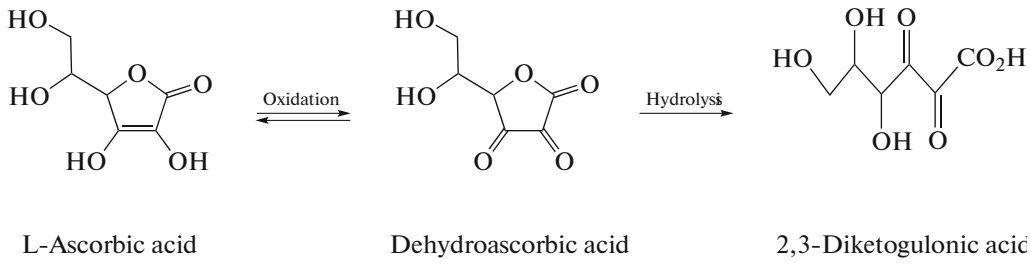
Fig. 10. UV spectra of vitamin C solution after contact with Granucol BI coal at 35°C for 5 (*1*), 10 (*2*), 20 (*3*), 30 (*4*) 50 (*5*), 80 (*6*), 110 (*7*), 140 (*8*) and 210 min (*9*).

(spectrum 2). This indicates that one of the chemical bonds in C=C breaks. So, in the phase of activated carbon, the ascorbic acid oxidizes turning into dehydroascorbic acid according to Scheme 1. At the same time, the state of ascorbic acid does not change in the liquid phase (Fig. 10).

From the data in Figs. 2–8, it can be concluded that vitamin C also oxidizes in the phase of coal AC–C, although it does not contain bentonite.

Finally, it can be said that in the complex system containing solution of vitamin C and activated carbon, the dynamic balance in the conversion of vitamin C, the ion-exchange property of bentonite and air oxygen which can be in the pores, changes the pH of the system and its effect on sorption.

The obtained data suggest that when using activated carbons to purify liquid systems or detoxify organisms, it must be taken into account that not only the adsorption capacity changes, but also some chemical parameters of the system do. It is also necessary to take into account the presence or absence of oxidants in the system. L-Ascorbic acid is relatively easily oxidized (including with dissolved oxygen in the liquid) to form dehydroascorbic acid, which can be hydrolyzed to form 2,3-diketogulonic acid (Scheme 2). The change in color of some liquids during treatment with activated carbon can be caused not only by the adsorption of polyphenol substances, but also by a change in pH.



L-Ascorbic acid Dehydroascorbic acid 2,3-Diketogulonic acid

Scheme 2. Scheme of the conversion of vitamin C to 2,3-diketogulonic acid.

4. CONCLUSIONS

The commercial activated carbons Granucol BI, Granucol GE, Granucol FA and the coal AC-C obtained in the laboratory of the Institute of Chemistry of the Academy of Sciences of Moldova possess a rather high capacity for adsorption of L-ascorbic acid. The adsorption value reaches up to 207.09 mg/g at 25°C and 144.75 mg/g at 35°C, which is much higher than values reported so far in the literature. The fact that the adsorption of vitamin C on activated carbons is higher at 25°C than at 35°C demonstrates that sorbent-sorbate interactions are physical (van der Waals forces), predominantly the dispersion forces. The kinetics of adsorption is perfectly described by the kinetic model of pseudo-second order. The process of adsorption of vitamin C on activated charcoal is accompanied by a change in the pH of the system. As the initial pH of the solution increases to about 6, the equilibrium pH increases slightly. With the further increase in the initial pH from 6 to 10 and more, the pH of the solution at equilibrium remains practically constant at about 6. The adsorption of vitamin C on activated carbons depends on the pH of the solution. Starting from an equilibrium solution pH of about 6, adsorption suddenly drops. The change in sorption depending on pH can be explained by the fact that in the basic medium, a part of vitamin C molecules are converted into another substance in the form of oxyanions. In a basic medium, acidic groups of coals also convert them into polyanions. In the activated carbon phase, vitamin C is oxidized to form dehydroascorbic acid, and the anionic form is an intermediate.

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The authors declare no conflict of interest.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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