Romanian Journal of Ecology & Environmental Chemistry, 7(2), 2025
https://doi.org/10.21698/rjeec.2025.207

Article

Removal of nitrite ions from water by adsorption on chemically activated carbon

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Received:	Reviewed:	Accepted:
16.11.2025	16.12.2025	17.12.2025

Abstract

In recent years, increasing emphasis has been placed on understanding the surface chemistry of activated carbons used for ion adsorption from aqueous media. The aim of the present study was to investigate the removal of nitrite ions using CAN-7 activated carbon, a mesoporous material with an acidic surface resulting from its preparation by chemical activation with phosphoric acid. To characterize the adsorption behavior, batch experiments were carried out while varying key operational parameters, such as solution pH, initial nitrite concentration and contact time. The experiments were performed at a liquid-to-solid ratio of 1:500, using activated carbon particles with sizes between 0.8 and 2.0 mm. The equilibrium data were analyzed using the Langmuir and Freundlich isotherm models, as well as several kinetic approaches, including pseudo-first-order, pseudo-second-order, and intra-particle diffusion models. The Langmuir isotherm provided the best fit to the experimental results, indicating that nitrite adsorption on CAN-7 occurs predominantly through chemisorption on the energetic homogeneous surface.

Keywords: *activated carbon, adsorption, nitrite ions, kinetics, isotherms*

INTRODUCTION

Water quality, particularly its chemical composition, exerts a direct impact on public health. A number of noncommunicable diseases are considered to be determined by the chemical composition of water [1÷3]. In the Republic of Moldova, approximately 20% of reported illnesses are linked to the consumption of water that fails to comply with drinking standards, compared to about 6% in Western European countries. Monitoring data for groundwater indicate that numerous artesian wells contain elevated levels of ammonium, nitrate, and nitrite ions, often surpassing the maximum allowable concentrations (MAC) [4].

Excessive ammonium concentrations have been observed in several centralized water supply systems, including those in Orhei and Criuleni districts. In Hincesti district, about 60% of samples contain up to 12.2 mg/L ammonium ions; in Leova, 40% of samples range between 2.2 and 8.2 mg/L; in Causeni, 19% of wells contain 2.1÷5.8 mg/L nitrites, while in Ialoveni, 32% of wells contain 69÷280 mg/L nitrates [5,6]. Seasonal intensification of nitrification processes during summer months contributes to the accumulation of nitrite ions in groundwater, with occasional peaks reaching 7÷18 mg/L in wells across the country [7÷9]. Typically, unpolluted groundwater contains only trace levels of nitrite and nitrate; however, contamination with nitrogenous compounds necessitates appropriate treatment to meet national and international drinking-water standards [4,10].

Research on water treatment processes has focused more extensively on nitrate removal, while nitrite remediation has been comparatively less investigated. Nevertheless, several nitrite elimination strategies have been documented, including biological nitrification–denitrification [11÷17], chemical denitrification [11,17÷24], and adsorption-based approaches [24÷32]. Various classes of adsorbents—such as polymers, mineral materials, and activated carbons (AC)—have been tested for this purpose [17,24,26÷33].

The objective of the present study was to evaluate the efficiency of a locally produced activated carbon, CAN-7, derived from walnut shells, for nitrite removal from water, with the aim of assessing its suitability for use in household filtration systems.

EXPERIMENTAL PART

CAN-7 activated carbon was used as an adsorbent, which is a domestic activated carbon, obtained from walnut shells by the chemical activation method with phosphoric acid [34]. The activated carbon CAN-7 characteristics has been evaluated by a series of indices, like as elemental analysis, ash content, pH of suspension, SEM-EDX.

The adsorption experiments were carried out in 100 mL Erlenmeyer flasks using a shaker with thermostat, and the mixing intensity was maintained constant throughout the experiments. The batch experiments were conducted at 1: 500 solid: liquid ratio, at 3÷10 pH range, contact time between 10 and 1800 minutes and temperature between 6 and 28ºC. Solutions of 0.1 mol/L NaOH and 0.1 mol/L HCl were used to adjust the solution pH to the desired value. After contact time, the phases were separated by filtration and in the solution the nitrite and phosphate ions were determined by UV-Vis spectrometric methods [35] using HACH DR/2500 spectrophotometer.

The activated carbon adsorption capacity for nitrite ions was calculated according to the Eq. 1:

$$a_{t,e}=\frac{C_0-C_{t,e}}{m}\cdot V \qquad (1)$$

where: $a_{t,e}$ (mg/g) amount of nitrite ions adsorbed in adsorption time (t) and equilibrium time (e); C_0 (mg/L) initial nitrite ions concentration; $C_{t,e}$ (mg/L) the concentration of the nitrite ions in the solution during the adsorption time (t) and equilibrium time (e); m (g) adsorbent mass; V (L) total volume of solution for adsorption.

In order to describe the adsorption behaviour of nitrite ions on activated carbon CAN-7, the pseudo-first order (Eq. 2), pseudo-second order (Eq. 3) and intra-particular diffusion (Eq. 4) kinetic models, and two commonly used isotherm models, the Langmuir (Eq. 5) and Freundlich (Eq. 6) isotherms [36÷40].

$$q_t = q_e - q_e \cdot e^{K_1 t} \qquad (2)$$

where q_e (mg/g) and q_t (mg/g) parameters represent adsorption capacities at equilibrium and time t respectively, and K_1 represents the rate parameter (1/min) [36].

$$q_t = \frac{K_2 \cdot q_e^2 \cdot t}{1 + K_2 \cdot q_e \cdot t} \qquad (3)$$

where q_e (mg/g) and q_t (mg/g) parameters represent adsorption capacities at equilibrium and time t respectively, and K_2 represents the rate parameter (g/mg/min) [37].

$$q_t = K_{id} \cdot t^{1/2} + C \qquad (4)$$

where C is the intercept and K_{id} (mg/gh$^{1/2}$) is the intra-particle diffusion rate constant, which can be determined from the slope of the linear plot of q_t versus $t^{1/2}$ [38].

$$q_e = \frac{q_{max} \cdot b \cdot C_e}{1 + b \cdot C_e} \qquad (5)$$

where q_e is the amount adsorbed at equilibrium per unit weight of adsorbent (mg/g), C_e is the equilibrium concentration of adsorbate in the liquid phase (mg/L), q_{max} and b are the Langmuir constants and are referred to as the maximum adsorption capacity (mg/g) and affinity parameter of the adsorption system [39].

$$q_e = K_f \cdot C^{1/n} \qquad (6)$$

where K_f is the Freundlich parameter that expresses the amount adsorbed (mg/g)(L/mg)$^{1/n}$ and n represents the adsorption strength. The value of $1/n$ ranges between 0 and 1; values below 1 imply chemisorption and values above 1 indicate a cooperative adsorption [40].

RESULTS AND DISCUSSION

Characteristics of the CAN-7 activated carbon

The sample CAN-7 is a mesoporous activated carbon (V_{total} = 0.81 cm^3/g, V_{meso} = 0.32 cm^3/g determined from benzene adsorption isotherms [41] with acidic surface due to method of obtaining (chemical activation with phosphoric acid) the estimated value of pH$_{pzc}$ is about 4.2 [34, 41]; with low ash content mainly of sodium, potassium, calcium, magnesium and iron oxides [41]. Along with C (78.4%), H (2.8%) and O (18.2%) the phosphorus in content of about 0.6 %.

Figure 1 illustrates the surface morphology of the activated carbon, while the EDX spectra confirm the presence of carbon, oxygen, and phosphorus, indicating the occurrence of surface organic groups that may act as active sites for adsorption. The acidic character of the CAN-7 surface was further verified through FTIR spectroscopy and Boehm titration method, both demonstrating the presence of specific oxygen-containing functional groups [42]. In the IR spectrum of the CAN-7 sample, a series of absorption bands specific to activated carbons were identified [42], which represent both the skeleton of activated carbons and the functional groups. Additionally, the absorptions specific to groups containing phosphorus are found CP-O (550-900 cm^{-1}), P=C and P-O (1000-1250 cm^{-1}) [42]. The concentration of acidic groups on the surface of CAN-7 activated carbon, determined by Boehm titration method, follows the series: carboxylic strong acidic (0.74 meq/g)=phenolic (0.74 meq/g)>>> carboxylic weak acidic (0.31 meq/g) [42].

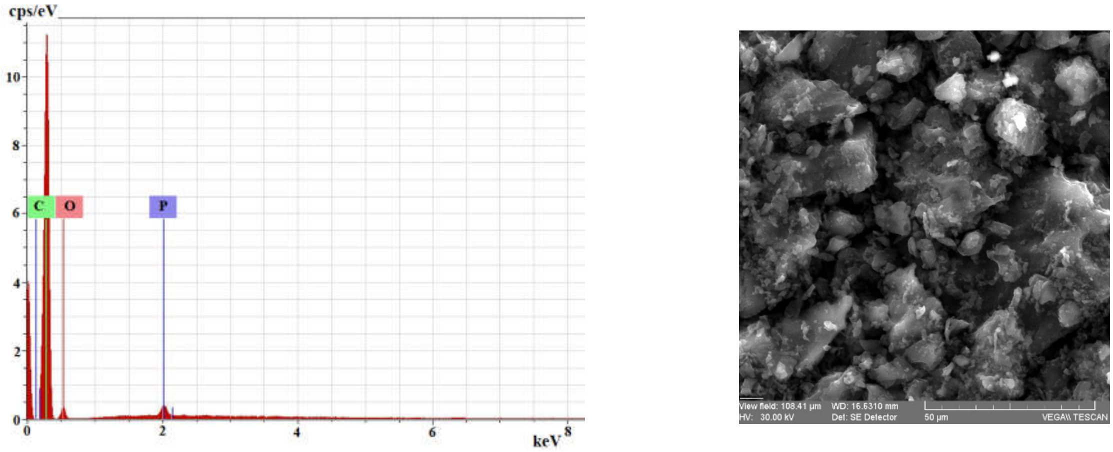


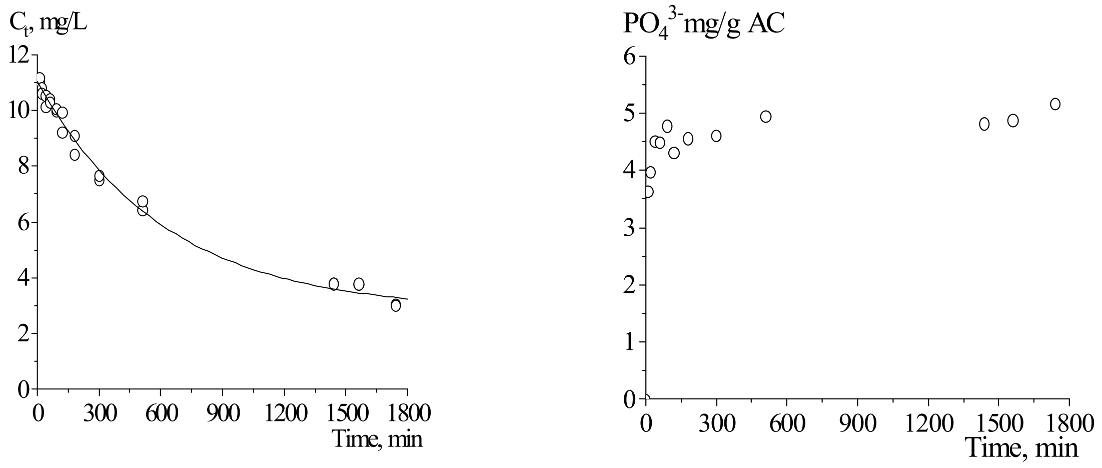
Fig. 1. SEM-EDX analysis of the activated carbon CAN-7

Nitrite ions adsorption on activated carbon CAN-7

Effect of contact time and kinetic models

The kinetics of the nitrite ions adsorption on activated carbon CAN-7 was studied at a solid/liquid ratio of 1:500, with the activated carbon fraction between 0.8 and 2.0 mm. At pre-set time intervals, the phases were separated by filtration using blue band paper, and the concentration of nitrite ions and phosphate ions were determined in the solution. Additionally, the pH and conductivity values of solutions were evaluated (data not presented here). According to obtained data, about 50% of nitrite adsorption/removal occurs in 600 minutes (Fig 2 (a)). Phosphates are washed off from the activated carbon surface in the amount of 4.5-5.0 mgPO$_4^{3-}$/gAC, which corresponds to approximately 0.16% of phosphorus (Fig 2 (b)). According to research results (elemental analysis and EDX), it was found that CAN-7 activated carbon contains approximately 0.6% phosphorus, which is bound to the activated carbon skeleton and originates from the activating agent (phosphoric acid) [41].

To describe the kinetics of nitrite ions adsorption on CAN-7 activated carbon, the pseudo-first-order (Eq. 2), the pseudo-second-order (Eq. 3), and intraparticle diffusion kinetic (Eq. 4) models were applied [36÷38]. The calculated kinetic parameters of the nitrite ions adsorption on activated carbon are presented in Table 1. The applicability of the kinetic models is compared by judging the determination coefficients (R^2) and the agreement between calculated and the experimental equilibrium adsorption capacity (q$_e$). The determination coefficient for the pseudo-second-order kinetic model is close to 1 (R^2 = 0.979), and the calculated adsorption capacity (0.807 mg/g) is very close to the experimentally found value (0.803 mg/g), which justifies the adsorption mechanism (Table 1). This suggests that the rate-determining step in the adsorption processes may be chemisorption, which involves valence forces through electron exchange between the adsorbent and the nitrite ions in the solution [43,44]. These results are in agreement with those reported in the scientific literature dealing with adsorption of nitrite or nitrate ions on activated carbons [28, 45÷47].



a ***b***

Fig. 2. The effect of contact time on nitrite ions adsorption on CAN-7 activated carbon, solid: liquid ratio= 1:500, 25 ºC: a) variation of nitrite ions concentration in solution, b) the amount of phosphate ions determined in solutions after contact with activated carbon

Intra-particle diffusion model (based on the theory proposed by Weber and Morris) was used to identify the diffusion mechanism. The intra-particle diffusion is valid when the adsorbate uptake (q_t) versus the square root of the contact time ($t^{1/2}$) is linear and the plot passes through origin [38]. The plot of nitrite ions uptake against square root of contact time is linear over the whole-time range ($R^2 = 0.992$), but it does not pass through the origin (the intercept is negative, about -0.23) (Figure 3).

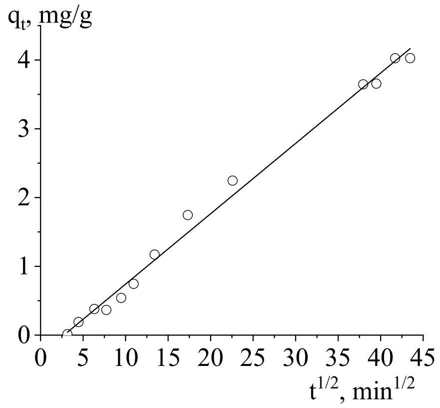


Fig. 3. The plot of nitrite ions uptake against square root of contact time (Weber-Morris model). (The experimental conditions are same as in Fig. 2)

Table 1. Kinetic models parameters

Kinetic models	q_e (exp), mg/g	Parameters	Values
pseudo-first-order	0.803	q_e (cal), mg/g	2.861
		k_1, min^{-1}	0.0031
		R^2	-0.926
pseudo-second-order		q_e (cal), mg/g	0.807
		k_2, g/mg min	0.143
		R^2	0.979
intra-particle diffusion		K_{id}, g/mg $min^{½}$	6.4×10^{-4}
		R^2	0.948
		$\overline{D}$, m^2/s	9.6×10^{-16}

This means that the intra-particle diffusion was not the only rate-controlling step, it could be a combining effect of the surface reactions. The diffusion speed ($\overline{D}$) of nitrite ions in the pores of activated carbon CAN-7 is quite low 9.6×10^{-16}, this can be explained by the size of activated carbon granules, which is between 0.8 and 2.0 mm (Table 1). Other researchers found higher values (10 times) of the diffusion coefficients of nitrite ions in the pores of a granulated activated carbon [28, 33].

Equilibrium study

In order to provide an adequate description of the adsorption data, Langmuir (Eq.5) and Freundlich (Eq.6) isotherm models have been applied to describe the adsorption behaviour of nitrite ions onto CAN-7 in aqueous solutions [39, 40]. The Langmuir isotherm is defined by several key parameters, including the equilibrium adsorption capacity q_e, the equilibrium concentration C_e, the maximum monolayer adsorption capacity q_{max}, and the Langmuir constant K_L, which reflects the affinity or free energy of adsorption [39]. In addition, the adsorption strength and sorbent capacity are influenced by the Freundlich constants K_f and n [40].

Figures 4 and 5 show the adsorption isotherms of nitrite ions on CAN-7 activated carbon determined at different operating temperatures (6°C, 18°C and 28°C) and pH values (3, 5, 7 and 10). Tables 2 and 3 show the constants of the theoretical Langmuir and Freundlich isotherms applied to approximate the experimental data.

According to the obtained data, with the increase in temperature from 6 to 28°C, the adsorption value increases with about 30% (from 5 to 6.6 mg/g) (Figure 4 and Table 2). And with the increase of the pH value from 3 to 7, the adsorption value decreases by about 5.5 times (Figure 5 and Table 3). Higher adsorption values at lower pH values are due to the excess of protons at a lower pH, which leads to an increase in the number of positively charged positions on the activated carbon surface, which favors the adsorption of nitrite ions due to electrostatic attraction. It should be noted that at low pH values, nitrite ions are in equilibrium with the HNO_2 form, because HNO_2 is a weak acid with K_a $5.1\cdot10^{-4}$ [48]:

$$HNO_2 + H_2O \leftrightarrow H_3O^+ + NO_2^- \qquad (7)$$

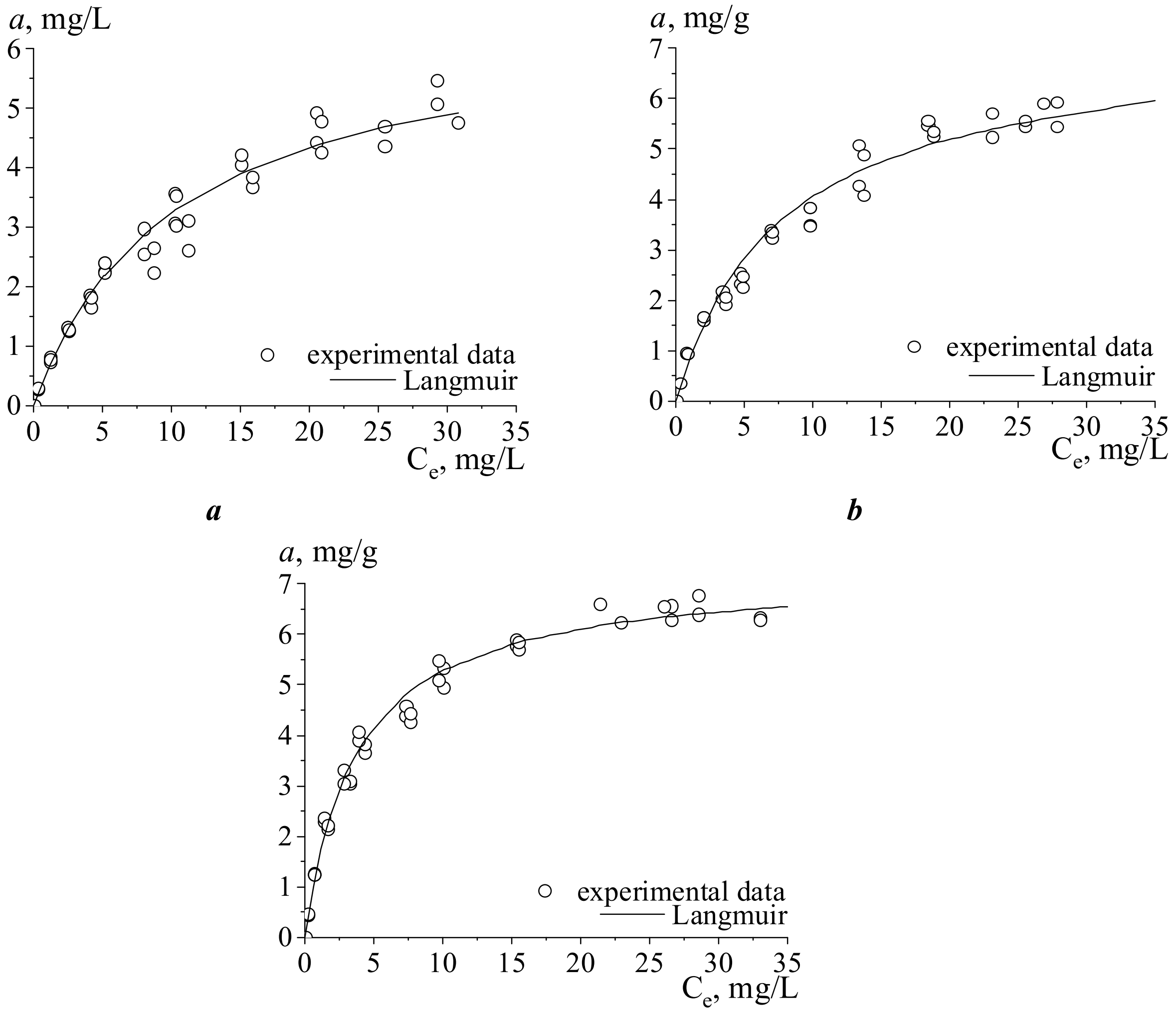
Both the Langmuir and Freundlich isotherms provide accurate descriptions of nitrite adsorption onto CAN-7 activated carbon. The coefficient of determination (R^2) was used to assess which model best fits the experimental data. The results show that the Langmuir isotherm yields the highest agreement, with R^2 values reaching or exceeding 0.99, which reflects highly favourable adsorption behaviour. Based on the Langmuir model, nitrite uptake proceeds through monolayer formation on a surface with uniform energy sites, with minimal interactions occurring between adsorbed nitrite ions [39].

Table 2. Langmuir and Freundlich isotherm constants for the adsorption of nitrite ions on CAN-7 activated carbon at different temperatures

Temperature, °C	Langmuir				Freundlich		
	a_{exp}, mg/g	q_{max},mg/g	K_L,L/mg	R^2	K_f	1/n	R^2
6	5.05	4.84	0.201	0.994	3.473	0.541	0.952
18	5.91	5.50	0.233	0.995	3.651	0.562	0.985
28	6.60	6.51	0.355	0.999	3.587	0.476	0.963

Table 3. Langmuir and Freundlich isotherm constants for the adsorption of nitrite ions on CAN-7 activated carbon at different pH values

pH	Langmuir				Freundlich		
	a_{exp}, mg/g	q_{max},mg/g	K_L,L/mg	R^2	K_f	1/n	R^2
3	8.10	9.15	1.193	0.982	4.525	0.589	0.972
5	6.60	7.23	0.273	0.998	1.587	0.476	0.964
7	1.40	1.69	0.026	0.982	-	-	-
10	0.35	0.97	0.034	0.739	-	-	-

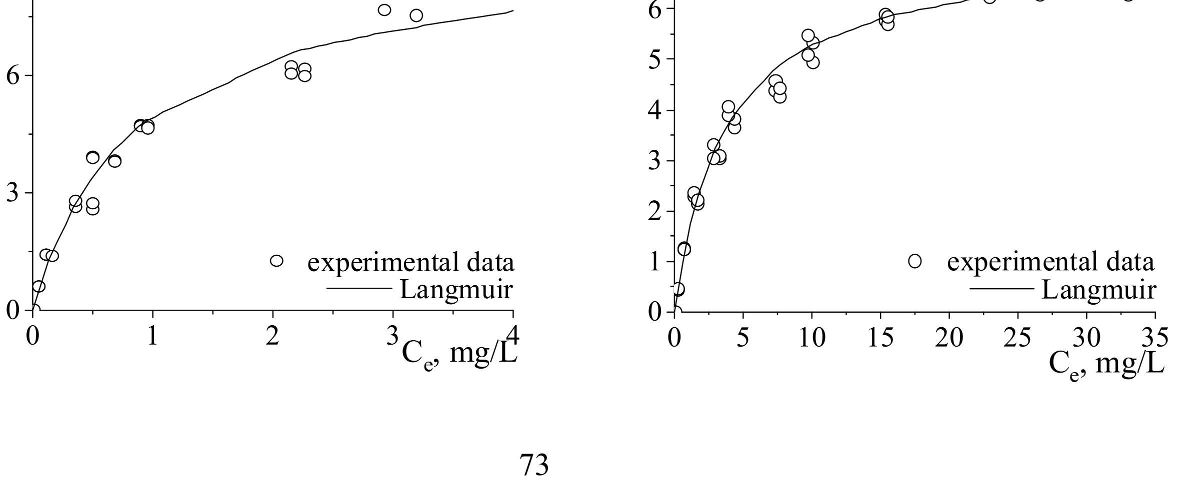


a ***b***

c

Fig. 4. Isotherms of nitrite ions adsorption on activated carbon CAN-7 for different temperatures: (a) 6°C, (b) 18°C, (c) 28°C. Solid: liquid ratio= 1:500, pH=5

Taking into account the results obtained in this study, as well as the production cost of the activated carbon CAN-7 (approximately 190 EUR/kg [34]), the findings support the continuation of research aimed at evaluating the applicability of CAN-7 as an adsorbent and/or catalyst in domestic water filtration systems.



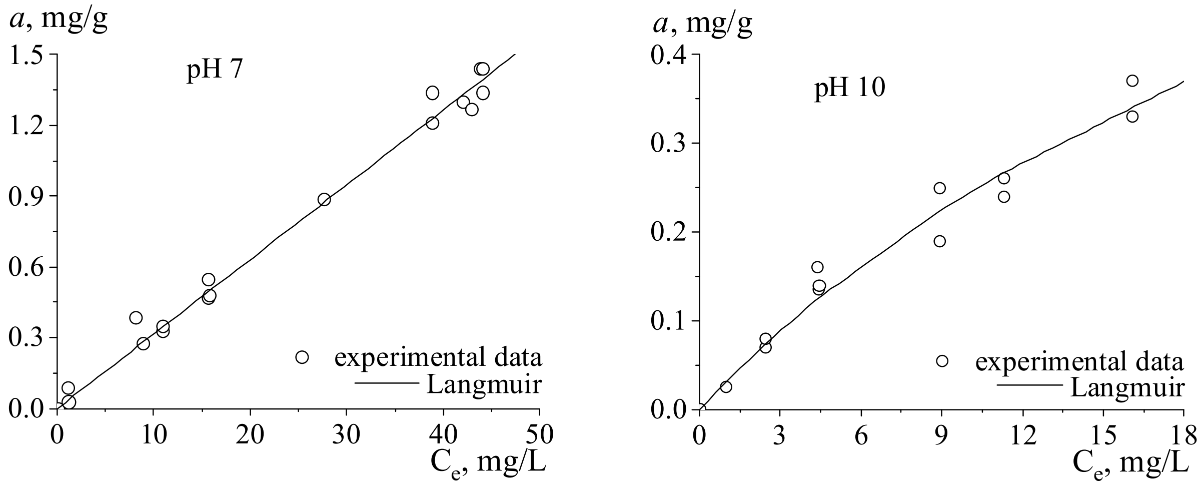


Fig. 5. Isotherms of nitrite ions adsorption on activated carbon CAN-7 at different pH values. Solid: liquid ratio= 1:500

CONCLUSIONS

The adsorption of nitrite ions onto the autochthonous activated carbon CAN-7 was investigated as a function of initial adsorbate concentration, solution pH, and temperature. Adsorption equilibrium was reached after approximately 600 min.

Kinetic modelling using the pseudo–first-order, pseudo–second-order, and intra-particle diffusion models showed that the pseudo–second-order model provides the best fit to the experimental data. The model yielded a determination coefficient of 0.98, and the calculated adsorption capacity (0.807 mg/g) closely matched the experimental value (0.803 mg/g), indicating that the rate-limiting step is likely chemisorption involving valence interactions through electron exchange between nitrite ions and surface sites. Equilibrium adsorption increased by ~30% (from 5.0 to 6.6 mg/g) as the temperature rose from 6 to 28°C, suggesting an endothermic process. In contrast, increasing the pH from 3 to 7 decreased adsorption efficiency by ~5.5, demonstrating the strong pH dependence of nitrite uptake.

Isotherm analysis showed that the Langmuir model best describes the adsorption behaviour, with coefficients of determination ≥0.99. This result implies monolayer adsorption on a homogeneous surface and negligible adsorbate–adsorbate interactions, confirming favourable adsorption conditions for nitrite removal using activated carbon CAN-7.

ACKNOWLEDGEMENTS

This work was supported by Republic of Moldova subprogram *Advanced research in computational and ecological chemistry, identification of technological procedures for treatment, formation of water quality and quantity* (ECOAQUA) 2024-2027 (code 010603) of Moldova State University (Institute of Chemistry).

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Citation: Goreacioc, T., Nastas, R., Lupascu, T., Removal of nitrite ions from water by adsorption on chemically activated carbon, *Rom. J. Ecol. Environ. Chem.*, **2025**, 7, no.2, pp. 68÷76.