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AMMONIA REMOVAL FROM WATER USING CATIONIC MATERIALS KU-2-8 AND DOWEX MAC-3

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ABSTRACT

In this work, it is shown that theareighest efficiency of ammonium extraction from water using cationites KU-2-8 and DOWEX MAC-3 is achieved when they are used in their acidic form. It has been established that hardness ions, namely calcium and magnesium ions, complicate the process of ion-exchange ammonium extraction, since these cationites have a higher selectivity for hardness ions. The highest level of regeneration of cationite KU-2-8 was found by using acid solutions compared to sodium chloride solutions. The use of phosphoric and nitric acid solutions for regeneration will allow further use of regeneration solutions for fertiliser production.

Keywords: water purification, ion exchange, nutrients, ammonium, resin regeneration, waste disposal, low-waste technologies.

INTRODUCTION

Today, the main sources of centralised water supply in Ukraine are surface sources, especially in large cities. The vast majority of Ukrainian rivers from which water is abstracted contain organic and inorganic pollutants, often in excessive concentrations [1, 2]. The reason for this is the direct impact of the industrial sector, municipal wastewater and agriculture on water bodies [3, 4], as well as active hostilities on the territory of Ukraine [5, 6].

The largest environmental damage, to water bodies, from Ukraine's industrial sector is caused by the metallurgical and mining industries, which can release toxic compounds, heavy metals and radioactive pollutants into surface and groundwater [7]. Metallurgy can also cause pollution with nitrogen-containing compounds (N-compounds), as ammonium is formed in the production of iron and steel. Municipal wastewater is generated in huge quantities and contains various pollutants: organic residues, waste products, various xenobiotics, fats and oils [8]. The danger of inadequate treatment of municipal wastewater is the excessive flow of nutrients into water bodies. The problem of insufficiently effective wastewater treatment is associated with outdated water treatment technologies and the treatment facilities themselves, which have hardly been modernised over the past half century [9]. One of the largest negative impacts of agriculture on the environment is pesticide [10] and fertiliser pollution [11]. Fertilisers used in agriculture contain mainly such biogenic elements as nitrogen and phosphorus. The infiltration of compounds containing these elements into groundwater through soil infiltration poses a threat of contamination of surface waters, with which this groundwater is hydrologically connected [12].

The removal of nutrients from industrial and municipal wastewater is currently one of the most important issues, as it causes eutrophication of water bodies [13] due to the active reproduction of cyanobacteria [14]. Ukraine is one of the countries with low freshwater resources, so water treatment for both drinking water and wastewater treatment should be a priority for the implementation of a sustainable development strategy, which provides for the well-being of the population, efficient use of natural resources, and environmental protection [15].

Biogenic compounds can be extracted by a variety of methods [16], including physical, chemical, physicochemical and biological methods [17]. One of the most common methods used to remove nitrogen compounds from water is conversion to ammonia and blowing it out with air, oxidation with oxygen, active chlorine or ozone, followed by filtering the solution through activated carbon.

Chemical methods include oxidation with chlorine [18], ozone [19], peroxone, and electrochemical oxidation [20]. The chlorination method is intended only for the treatment of water with a low ammonium content, so its use is impractical for the treatment of municipal or industrial wastewater. Ozonation provides an effective oxidation of ammonia to nitrate, but it requires maintaining a pH value of > 8 and additional removal of nitrate ions from water, which is not rational on an industrial scale [21]. Electrochemical oxidation provides 99 % ammonium removal efficiency, but the disadvantages of the method include the negative effect of sodium chloride on oxidation, which significantly reduces the efficiency of the method, the need to maintain a neutral pH value, and the high cost of process maintenance [22].

Physical methods used for ammonium removal include steaming [23], vacuum ammonium distillation [24], and precipitation in the form of struvite [25]. The steaming method results in high ammonium removal efficiencies, but the main disadvantage is the formation of large amounts of scale, which makes it impossible to use the method for large-scale applications. Vacuum distillation provides ultra-high ammonium recovery, but due to the time required, it is not suitable for treating large volumes of water. The method of precipitation in the form of struvite provides an extraction efficiency of 91 - 96 %, but the disadvantage is the problem of sediment disposal [26].

Biological methods of nutrient removal [27] are one of the most appropriate for use in the treatment of large volumes of municipal wastewater, but the denitrification efficiency usually does not reach more than 90 % [28].

Physicochemical [29] methods include adsorption [30] and ion exchange [31, 32]. The adsorption method does not always provide effective ammonium removal [33], as HCO_3^-, Cl^-, SO_4^{2-} ions which are often presented in water reduce the adsorption capacity of nitrogen-containing compounds [34]. To effectively remove ammonium from water, it is necessary to use fine-porous materials [35], which requires a long filtering period, which is why this method is not advisable. Currently, research is underway into the possibility of using the ion exchange method to remove ammonium ions from water as an additional treatment process. The use of the ion exchange method [36] is effective and due to its simplicity and relatively low cost, is promising [37]. Ion exchange resins are polymers with electronically charged sites where one ion can replace another, thereby removing undesirable ions from the water to be treated [38]. Regeneration solutions and flushing water, if nitrogen-containing components are removed, can be used to prepare fertilisers. This method can be used for highly efficient water purification from biogenic compounds both in water treatment and for the removal of N-compounds from wastewater before discharge into a water body. Ion exchange technologies for ammonium removal can be an alternative to biological processes, but greater efficiency will be achieved by using the processes in parallel, which will allow for comprehensive treatment.

The purpose of this work is to investigate the efficiency of ammonium extraction using KU-2-8 and DOWEX cationites.

EXPERIMENTAL

The strongly acidic cationite KU-2-8 in Na^+ and H^+ forms and DOWEX MAC-3 in H^+ form were used to determine the efficiency of ion-exchange ammonium removal from water.

Cationite KU-2-8 was converted to the acidic form with a solution of C (HCl) = 1 %, and to the salt form with C (NaCl) = 1 %. In the process of sorption, model solutions containing 6.0, 30.0, 75.0, and 100.0 mg-eq dm^{-3} (Na^+ form), 30.0, 75.0, and 100.0 mg-eq dm^{-3} (H^+ form) were filtered through the cationite. The volume of the KU-2-8 was 20 cm^3, and the consumption of regeneration solution of solutions during sorption was maintained at ~ 13 cm^3 min^{-1}. Samples of 100 - 300 cm^3 were taken and the residual ammonium concentration, alkalinity or acidity, and pH were measured.

A weak acid cationite was used to determine the effect of calcium ions in water on the efficiency of ammonium sorption. Model solutions containing 30.0 mg-eq dm^{-3} of ammonium and calcium for DOWEX MAC-3 in the H^+ form were used for sorption. The solutions were passed through a 20 cm^3 cationite layer. The residual ammonium, calcium, pH, and alkalinity concentrations were determined in 100 cm^3 samples.

The regeneration of KU-2-8 cationite into the H^+ form was carried out with 5 - 10 % HCl solutions, 5 % HNO_3, H_3PO_4 and H_2SO_4 solutions, and into Na^+ with a 10 % NaCl solution at a regeneration solution flow rate of ~ 2 cm^3 min^{-1}. Samples of the regeneration solution were taken in 20 cm^3 and the ammonium concentration, alkalinity, acidity, and pH were determined.

The following formulas were used to determine the exchangeable dynamic capacity of the ionite before slippage, mg-eq dm^{-3} (EDC) and the total exchangeable dynamic capacity, mg-eq dm^{-3} (TEDC) [39]:

$$EDC = \frac{V_{\mathrm{p}}(C_{\mathrm{p}} - C_{\mathrm{i}})}{V_i}\ , \qquad (1)$$

$$TEDC = \frac{\sum_{\mathrm{i}=1}^{n}(C_{p.} - \mathrm{C_i}) \cdot V_s}{V_{\mathrm{i}}}, \qquad (2)$$

where $C_{p.}$ is the initial concentration of ions in the solution, mg-eq dm^{-3}; C_i is the concentration of ions in the i-th sample after sorption, mg-eq dm^{-3}; V_p is the volume of sample before ion leakage, dm^3; V_i is the volume of ionite, cm^3; V_s is the volume of solution, cm^3; n is the number of samples taken before the ionite capacity is exhausted.

The Eq. (3) was used to determine the degree of desorption (A, %) of the ionite:

$$\mathrm{A}_n = \frac{\sum_{i=1}^{n} \mathrm{M}_{\mathrm{i.}}^{d.}}{\mathrm{M}_{s.}} \cdot 100\% \qquad (3)$$

where $M_{i.}^{d}$ is the mass of desorbed ions in the i-th sample of the regeneration solution, mg-eq; $M_{s.}$ is the mass of sorbed ions, mg-eq.

RESULTS AND DISCUSSION

The efficiency of ion-exchange ammonium ion removal using KU-2-8 and DOWEX MAC-3 cationites was investigated. When studying the effectiveness of using DOWEX MAC-3 cationite in the H^+ form for NH_4^+ extraction, it was found that effective ion extraction occurs in the first 300 cm^3 of solution at an initial concentration of 30.0 mg-eq dm^{-3}. The possibility of using this cationite for NH_4^+ extraction in the presence of hardness ions was also investigated. To evaluate the effect of hardness ions on the process of ammonium ion sorption, a study was conducted on the extraction of ammonium on cationite in the H^+ form in the presence of Ca^{2+} ions (Fig. 1).

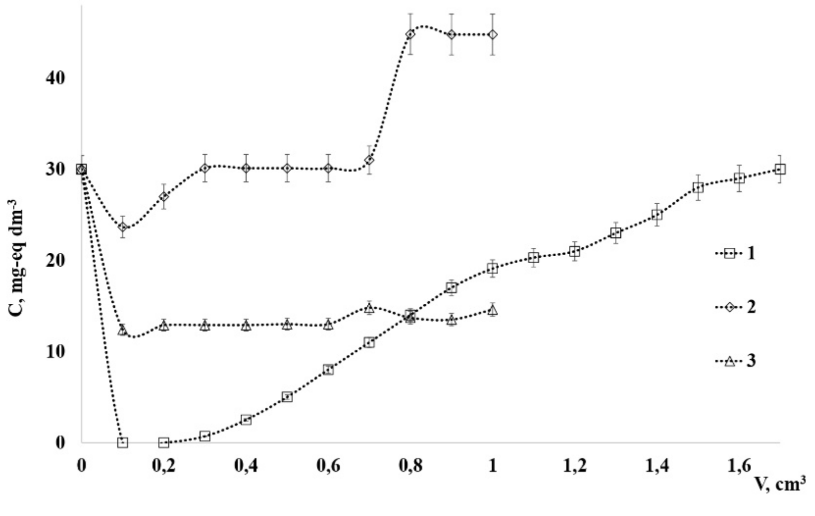

Fig. 1. Dependence of the concentration of ammonium ions (1; 2) and calcium ions (3) on the volume of NH_4Cl solution in distilled water (1) and a model solution of NH_4Cl and $CaCl_2$ in a 1 : 1 ratio (2; 3) through DOWEX MAC-3 cationite in the H^+ form at an initial ammonium concentration of 30.0 mg-eq dm^{-3} (V_i = 20 cm^3, V_p = 100 cm^3).

The process of sorption of ammonium ions from a solution with a concentration of 6 mg-eq/dm^3 on cationite KU-2-8 in the Na$^+$ form is rather slow (Fig. 2). With an increase in the initial concentration of ammonium ions to 30.0 and 100.0 mg-eq dm^{-3} during sorption on KU-2-8 in H$^+$ and Na$^+$ forms, it was found that the ionite acquired faster saturation at higher concentrations (Fig. 3, Fig. 4). Therefore, during 20 mL on KU-2-8 in H$^+$ form sorption 2400 and 1200 mL of solution (Fig. 3). When using KU-2-8 in Na$^+$ form, similar results were obtained (Fig. 4).

Since the actual calcium content in natural waters is much higher than the ammonium content, a concentration ratio of 1 : 3 was chosen. At the chosen ratio of NH_4^+ to Ca^{2+} concentrations, the efficiency of ion-exchange ammonium removal on cationite was quite low. On cationites KU-2-8 and DOWEX MAC-3 in the presence of calcium ions in the ratios of 1 : 1 and 1 : 3, sorption of ammonium ions almost does not occur, and then even sorbed ammonium ions are displaced by calcium. The low values of the TEDC for ammonium ions also confirm

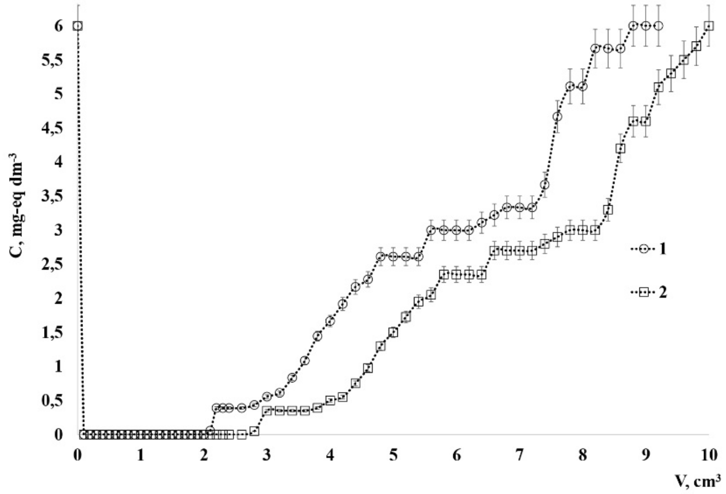

Fig. 2. The initial curve of ammonium ion sorption on cationite KU-2-8 in Na$^+$ (1) and H$^+$ (2) form from a solution in distilled water ($[NH_4^+]$ = 6.0 mg-eq dm^{-3}), (V_i = 20 cm^3).

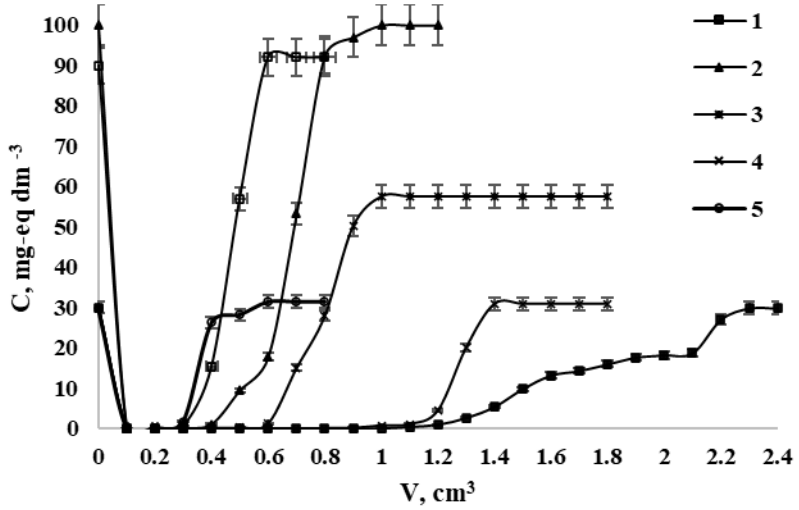

Fig. 3. Dependence of the concentration of ammonium ions (1; 2; 3; 5) and calcium ions (4; 6) on the volume of NH_4Cl solution in distilled water (1; 2) and model solutions of NH_4Cl and $CaCl_2$ in the ratios 1 : 1 (3; 4) and 1 : 3 (5; 6) through the cationite KU-2-8 in the H$^+$ form at initial concentrations of $[NH_4^+]$, mg-eq dm^{-3}: 30.0 (1; 3; 4; 5; 6), 100.0 (2) (V_i = 20 cm^3, V_p = 100 cm^3).

this (Figs. 5 - 7).

The value of TEDC of cationite DOWEX MAC-3 in the H^+ form, at an initial concentration of 30 mg-eq dm^{-3}, is 1282.0 mg-eq dm^{-3} in distilled water, in water containing calcium ions, the DOWEX decreases to 46.7 mg-eq dm^{-3} for ammonium and 831.5 for calcium (Fig. 5).

The value of the TEDC of cationite KU-2-8 in the H^+ form is 2581.1, 3144.5 mg-eq dm^{-3} at initial concentrations of 30.0 and 100.0 mg-eq dm^{-3}, respectively. For cationite in the Na^+ form, the TEDC is 2620.8 mg-eq dm^{-3} at an initial concentration of 30.0 mg-eq dm^{-3}.

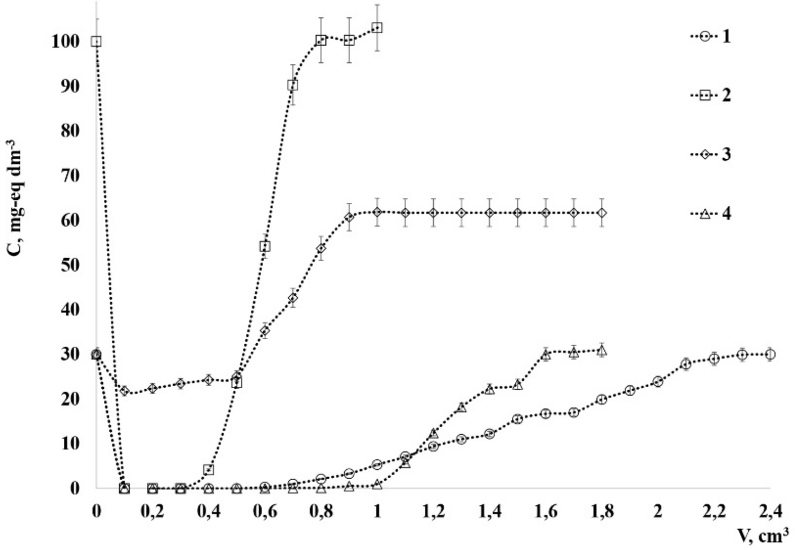

Fig. 4. Dependence of the concentration of ammonium ions (1; 2; 3) and calcium ions (4) on the volume of NH_4Cl solution in distilled water (1; 2) and a model solution of NH_4Cl and $CaCl_2$ in the ratios 1 : 1 (3; 4) passed through the strongly acidic cationite KU-2-8 in the Na^+ form at the initial concentrations of $[NH_4^+]$, mg-eq dm^{-3}: 30.0 (1; 3), 100.0 (2) (V_i = 20 cm^3, V_p = 100 cm^3).

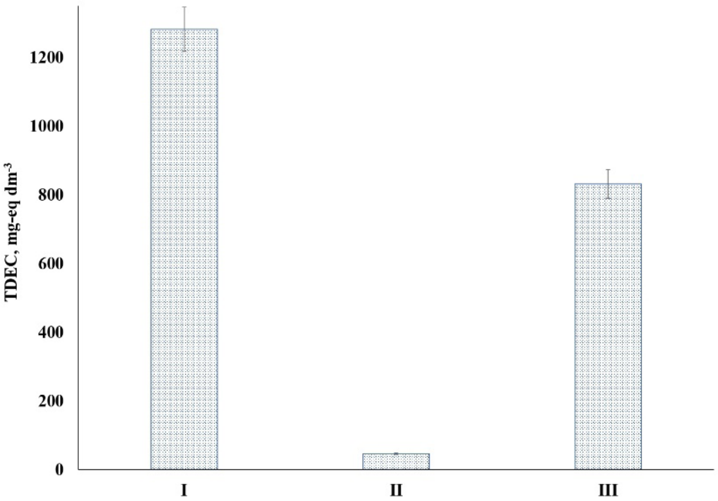

Fig. 5. Values of the TEDC of DOWEX MAC-3 cationite in the H^+ form for ammonium (I, II) and calcium (III) ions during the sorption of NH_4^+ ions from distilled water (I) and in the presence of Ca^{2+} ions (II, III) at an initial ammonium concentration of 30.0 mg-eq dm^{-3}.

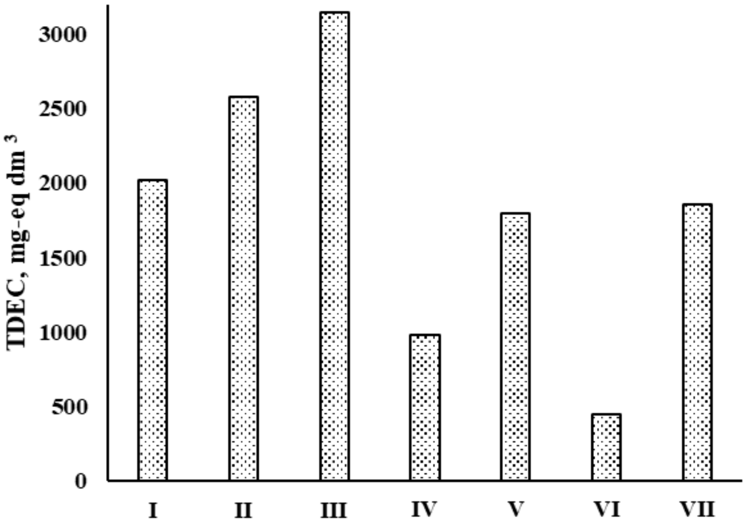

Fig. 6. Values of the EDC of cationite KU-2-8 in the H^+ form at the initial concentrations of $[NH_4^+]$, mg-eq dm^{-3}: 6.0 (I), 30.0 (II; IV; V; VI; VII), 100.0 (III) for ammonium (I; II; III; IV; VI) and calcium (V; VII) ions from the skipped volume of NH_4Cl solution in distilled water (I; II; III) and model solutions of NH_4Cl and $CaCl_2$ in the ratios of 1 : 1 (IV; V) and 1 : 3 (VI; VII) (V_i = 20 cm^3).

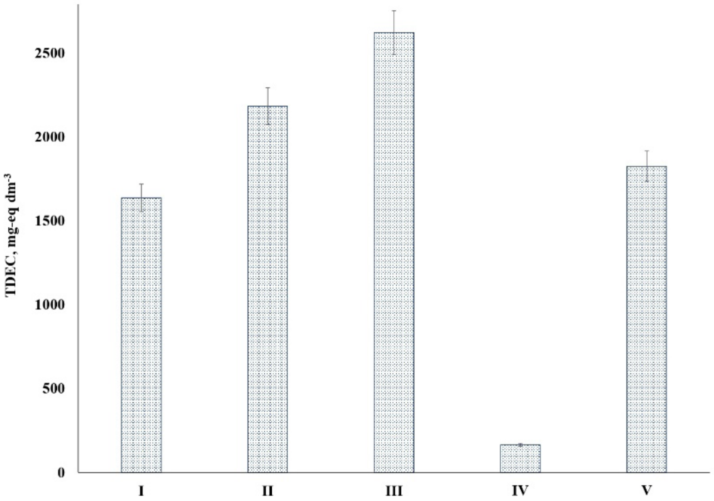

Fig. 7. Values of EDC of cationite KU-2-8 in Na^+ form at initial concentrations of $[NH_4^+]$, mg-eq dm^{-3}: 6.0 (I), 30.0 (II; IV; V), 100.0 (II) for ammonium ions (I; II; III; IV) and calcium (V) from the skipped volume of NH_4Cl solution in distilled water (I; II; III) and model solution of NH_4Cl and $CaCl_2$ in the ratio of 1:1 (IV; V).

For cationite KU-2-8 in the H^+ form, the value of the EDC before slippage increases with the initial concentration, and is quite low in the Na^+ form, which indicates the low selectivity of cationite for ammonium ions in this form.

In the presence of calcium ions in the solutions, the TEDC is 164.7 and 975.0 mg-eq dm^{-3} for ammonium ions and 1824.3 and 1797.3 for calcium ions at an initial concentration of ammonium ions of 30.0 mg-eq dm^{-3}. These results confirm the rather low selectivity for ammonium ions of cationite KU-2-8 in the Na^+ form.

Therefore, considering the data obtained, it can be stated that the use of DOWEX MAC-3 cationite is inappropriate for the removal of ammonium in the presence of calcium ions.

To regenerate the strongly acidic cationite KU-2-8 in the H^+ and Na^+ forms, we used 5 % solutions of HCl, H_2SO_4, H_3PO_4, HNO_3, as well as 10 % solutions of HCl and NaCl (Fig. 8, Fig. 9). When using a 5 % HCl solution, the regeneration efficiency was 84.0 % at a consumption of regeneration solution of 3 cm^3 cm^{-3} and increased to 99 % at a consumption of regeneration solution of 7 cm^3 cm^{-3}. Increasing the concentration of hydrochloric acid to 10 % makes it possible to increase the regeneration efficiency to 92.6 % at a consumption of regeneration solution of 3 cm^3 cm^{-3}.

When using 5 % NaCl, the regeneration efficiency was low. Therefore, a higher concentration solution NaCl was chosen for research. At a consumption of 10 % NaCl as a regeneration solution qp = 3 cm^3 cm^{-3}, the regeneration efficiency is 71.6 % and increases to 86.3 and 91.9 % at a consumption of 4 and 5 cm^3 cm^{-3}. Thus,

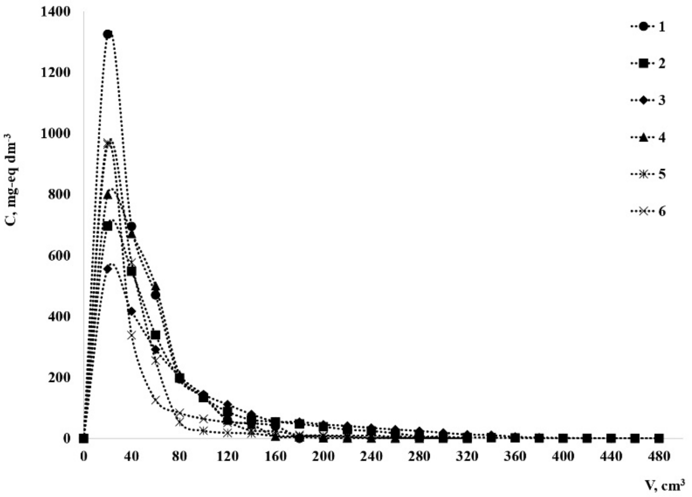

Fig. 8. Dependence of the initial ammonium concentration on the volume of skipped regeneration solutions of 5 %: HCl (1), H_2SO_4 (2), H_3PO_4 (3), HNO_3 (4) and 10 %: HCl (5), NaCl (6) through the strongly acidic cationite KU-2-8 in the NH_4^+ form (V_i = 20 cm^3).

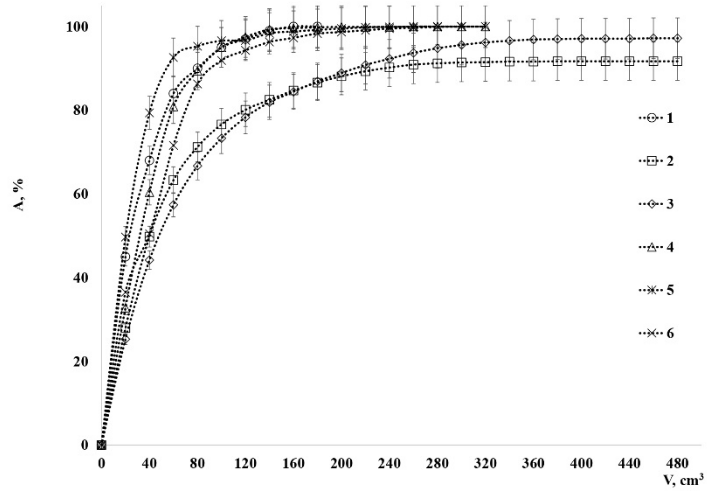

Fig. 9. Dependence of the degree of ammonium desorption on the volume of regeneration solutions. of 5 %: HCl (1), H_2SO_4 (2), H_3PO_4 (3), HNO_3 (4) and 10 %: HCl (5), NaCl (6) through the cationite KU-2-8 in the NH_4^+ form (V_i = 20 cm^3).

when using hydrochloric acid solutions, regeneration is faster and better compared to sodium chloride solutions.

The choice of nitric and phosphoric acids as regeneration solutions allows for the subsequent processing of spent regeneration solutions to produce liquid fertilisers. The consumption of regeneration solution (qp = 4 cm^3 cm^{-3}), i.e. 80 cm^3 of solution per 20 cm^3 of ionite, allows achieving 89.4 % nitric acid regeneration efficiency, and increasing it to 95.0 % and 97.5 % at q_p of 5 and 6 cm^3 cm^{-3}, respectively.

When using phosphoric and sulfuric acids, slightly worse results were obtained. At a consumption of regeneration solution of q_p = 5 and 6 cm^3 cm^{-3}, the regeneration efficiency with sulfuric acid is 76.5 and 80.1 %, and with phosphoric acid - 73.3 and 78.3 %. Only when the consumption of regeneration solution of regeneration solutions was increased to 11-12 cm^3 cm^{-3} did the regeneration rate increase to above 90 %.

Thus, it can be argued that the efficiency of acid regeneration is quite effective and rises with an increase in the concentration of the acid solution from 5 % to 10 %. The most effective and, accordingly, the lowest costs are observed when using nitric and hydrochloric acids.

Therefore, taking into account the above results, despite the rather positive results of sorption and desorption of ammonium ions on cationite KU-2-8, the ion exchange method should be recommended for water treatment that does not contain hardness ions or for water that has been previously softened, for example, by reagent [40 - 42], ion exchange [43, 44] or membrane methods [45, 46].

CONCLUSIONS

The efficiency of ammonium extraction on cationites KU-2-8 and DOWEX MAC-3 was investigated depending on the form of the cationite. It was shown that cationite in the Na^+ form has a lower selectivity for ammonium ions compared to the H^+ form. The regeneration of cationite KU-2-8 was more efficient when using acid solutions. It has been established that the use of cationics is inappropriate for ammonium removal in the presence of hardness ions, which indicates the need for preliminary water softening.

Authors' contributions: *M.G.: Conceptualization, Supervision, Editing; I.T.: Investigation, Data praocessing, Writing original draft, visualization; I. F.: Literature review, Methology.*

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