

INVESTIGATION OF THE REMOVAL OF CATIONS FROM AQUEOUS SOLUTIONS USING SAPROPEL

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Abstract. Sapropel is a sediment found at the bottom of lakes, consisting of both organic and inorganic materials. Water contains various cations, such as ammonium, calcium, and magnesium, which are generally harmless to human health; however, excessive concentrations of these ions can have negative effects. Although ammonium itself does not directly affect human health, it oxidizes into nitrites and nitrates when in contact with oxygen, and these compounds can be toxic. Similarly, while calcium and magnesium ions do not pose a health risk, they can damage pipes and shorten the lifespan of water-using electrical devices. Sapropel can be used as a biosorbent to remove these cations from aqueous solutions. It is inexpensive, eco-friendly, naturally occurring, easy to use, and sustainable. Various doses of sapropel, ranging from 1 to 50 g/L and mixing duration from 15 to 120 min., have been tested. Research has shown that using sapropel as a sorbent reduces the concentration of magnesium ions by 20%, calcium ions by 15.5%, ammonium – 57%. The optimal combination for ammonium ions was found 40 g/L of sapropel and ≥ 30 min of mixing.

Keywords: sapropel, water, ammonium ions, calcium ions, magnesium ions.

1. Introduction

Water contains various dissolved salts, including cations such as calcium, magnesium, and ammonium. Although calcium and magnesium are beneficial to human health (Wang et al., 2024), and ammonium ions are an indicator compound that has no direct impact on human health, nitrogen in the form of ammonium does not remain in the water for long and, if there is sufficient contact with oxygen, it quickly oxidizes in water to nitrites and then to nitrates (Sakalauskas et al., 2016; Radu & Racoviteanu, 2021). Meanwhile, nitrites and nitrates are carcinogenic compounds that cause various diseases (Crowe et al., 2019; Neth et al., 2020; Altamimi & Al-Eissa, 2021). Calcium ions contribute to the formation and accumulation of deposits (CaCO_3) inside pipes, which reduces their diameter and can increase hydraulic resistance, while magnesium ions can alter the stability of deposits, thereby negatively affecting the longevity and efficiency of pipelines (Peng et al., 2010).

Calcium and magnesium ions are removed from water in various ways, but nowadays, inexpensive, effective, and sustainable materials and technologies are being sought that can remove various elements. Currently, an artificial sorbent such as alginate-citrate composite aerogel effectively removes calcium and magnesium

ions from water, achieving 96% efficiency (Wang et al., 2021). Modified Na-X and Na-Y zeolites effectively sorb calcium and magnesium ions from hard water, achieving high cation exchange capacity, and the sorption efficiency depends on the pore structure of the zeolites and their chemical modification (Le Van Mao et al., 1994). Natural sorbents such as clinoptilolite are also used, which effectively sorbs magnesium ions from water, achieving a sorption capacity of about 2.5 mg Mg^{2+} per gram of sorbent (Tomić et al., 2012). However, there are no studies on these compounds using a natural biosorbent – sapropel.

Sapropel is a sediment rich in organic and inorganic substances that forms at the bottom of lakes due to the accumulation of biological remains (Bakšienė & Ciūnys, 2012). Sapropel is a homogeneous gel-like mass with a pH ranging from 7 to 8 (Pavlovskaja et al., 2020). Sapropel contains various macro- and microelements, as well as biologically active substances such as enzymes (Daux et al., 2006; Thomson et al., 2006; Bakšienė & Ciūnys, 2012). According to Klavina et al. (2020), sapropel is rich in biologically active organic compounds, especially humic and fulvic acids. It has been found that fulvic acids, due to their carboxyl, phenolic, and carbonyl functional groups, have a high ability to bind with metal ions,

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therefore such substances can act as natural complexing agents in aquatic ecosystems (Sharma & Anthal, 2022). Tipping and Hurley (1992) found that humic substances sorb cations through specific complexation to carboxyl and phenolic groups and non-specific electrostatic ion accumulation (Donnan-type mechanism), and the strength of these interactions is determined by pH, ionic strength, the loading density of humic substances, and the chemical properties of metals; the sequence of cation binding strength is as follows: $Mg^{2+} < Ca^{2+} < Mn^{2+} < Cd^{2+} < Co^{2+} < Ni^{2+} \approx Zn^{2+} < Pb^{2+} < Cu^{2+} < VO^{2+}$. Other scientists have found that magnesium (Mg^{2+}) binds to humic acids mainly at carboxyl groups, and the strength of the bond depends on the pH and structure of the humic acid (Christl, 2018). One of the most important properties of wet sapropel is the colloidal structure, which causes organic colloidal particles to absorb large amounts of water, resulting in high moisture content and low filtration rate (Obuka et al., 2015). According to these properties, sapropel can be used as a biosorbent. Studies have shown that sapropel can be used for the sorption of heavy metals: the efficiency of lead removal reached 81.6%, while the efficiency of zinc sorption from solution was even higher – 97.57% (Birgėlaitė et al., 2016). Other authors have found that sapropel removes metals from aqueous solutions with the following efficiency: Cr (31%), Cu (84%), Zn (84%), Cd (93%), Pb (98.6%) (Albrektienė-Plačakė et al., 2023; Albrektienė-Plačakė & Paliulis, 2024). Sapropel can be widely used not only for water purification, but also as a fertilizer, feed additive, cosmetic ingredient, or building material (Bakšienė & Ciūnys, 2007; Blecic et al., 2014; Obuka et al., 2015; Voišnienė et al., 2024). The use of sapropel helps to promote sustainable resource management and contributes to the implementation of circular economy principles.

Considering the discussed properties of sapropel and the importance of cations for human health and their influence on technology, it becomes relevant to investigate the ability of sapropel to sorb ions present in water. Therefore, the aim of this work is to evaluate the ability of sapropel, as a natural biosorbent, to sorb cations (Ca^{2+} , Mg^{2+} , NH_4^+) in water and to determine its potential for improving water quality and protecting water management systems from sedimentation, thus contributing to sustainable resource management and the implementation of circular economy principles.

2. Methods and materials

Sapropel taken from a depth of 2–3 meters in Lake Apslavo, located in the village of Stabulankliai, Leliūnai parish, Utena district, Lithuania, was used for the research. The sapropel was washed twice with distilled water and then dried for 3 hours in a thermostat at 110 °C. The dried sapropel was crushed with a porcelain mortar and sieved through a 0.2 mm sieve. The chemical composition of the dried sapropel was analyzed using an X-ray

fluorescence spectrometer, and the carbon content was determined using a CS-2000 carbon and sulfur analyzer. The microstructure of the sapropel was examined using a SEM JEOL JSM-7600F scanning electron microscope.

Three cations (Ca^{2+} , Mg^{2+} and NH_4^+), were selected for this study, which are not harmful to human health but are all found in groundwater. The concentrations of these cations in aqueous solutions were selected according to the actual concentrations prevailing in groundwater in Lithuania: 4,5 mg/L Ca^{2+} , 2 mg/L Mg^{2+} , 0,7 mg/L NH_4^+ . Water samples were prepared using standard $Ca(NO_3)_2$, $Mg(NO_3)_2$, NH_4Cl solutions with a cation concentration of 1 g/L and deionized water (pH ~ 5.5).

10 different doses of sapropel (1, 2, 3, 4, 5, 10, 20, 30, 40, 50 g/L) were used to remove calcium, magnesium, and ammonium ions from aqueous solutions. One-liter water samples contaminated with 4,5 mg/L Ca^{2+} , 2 mg/L Mg^{2+} , 0,7 mg/L NH_4^+ , were mixed for 15, 30, 60, 90, and 120 minutes at a speed of 150 rpm. After mixing for different periods of time with different doses of sapropel, 100 mL water samples were taken from each container to determine the cations, and the water sample was filtered through a 0.45 μm membrane filter. The concentrations of calcium and magnesium ions in the water solutions were determined according to “LST ISO 6059:1998/P:2008 Water quality – Determination of the sum of calcium and magnesium – EDTA titrimetric method” (Lietuvos standartizacijos departamentas, 2008), and ammonium ions according to “LST ISO 7150-1:1998 Water quality – Determination of ammonium. Part 1: Manual spectrometric method” (Lietuvos standartizacijos departamentas, 1998).

Linear regression was used for statistical data analysis to evaluate the effect of sorbent dose and mixing time on the concentration of cations in the solution. Statistical analysis was performed using IBM SPSS Statistics software.

3. Results

Determining the composition and properties of the selected material is essential when conducting research. Therefore, the elements that make up sapropel were investigated. The chemical composition of sapropel is shown in Table 1.

According to the data presented in Table 1, it can be seen that the largest part of sapropel consists of oxygen (O) – 56.3% and silicon (Si) – 30.4%. A significant part of sapropel consists of carbon (C) – 6.6%. This indicates that sapropel contains organic compounds. The metals dominant in sapropel are aluminum (Al) – 2.6% and potassium (K) – 1.4%. Sapropel contains 0.890% calcium (Ca) and 0.28% magnesium (Mg).

Figure 1 shows the microstructure of sapropel. It was found that sapropel particles are irregular in shape and unevenly distributed. SEM images reveal a heterogeneous structure with particles of varying sizes, reaching

Table 1. The chemical composition data of sapropel

Element	w/w,%	Element	w/w,%
O	56.3	Cr	0.027
Si	30.4	Zr	0.024
C	6.6	Ba	0.013
Al	2.6	Cl	0.011
K	1.4	Mn	0.010
Ca	0.89	Pb	0.007
Fe	0.66	Rb	0.005
Na	0.36	Sr	0.005
Mg	0.28	Ni	0.003
S	0.17	Zn	0.003
Ti	0.15	Y	0.002
P	0.04	Cu	0.001

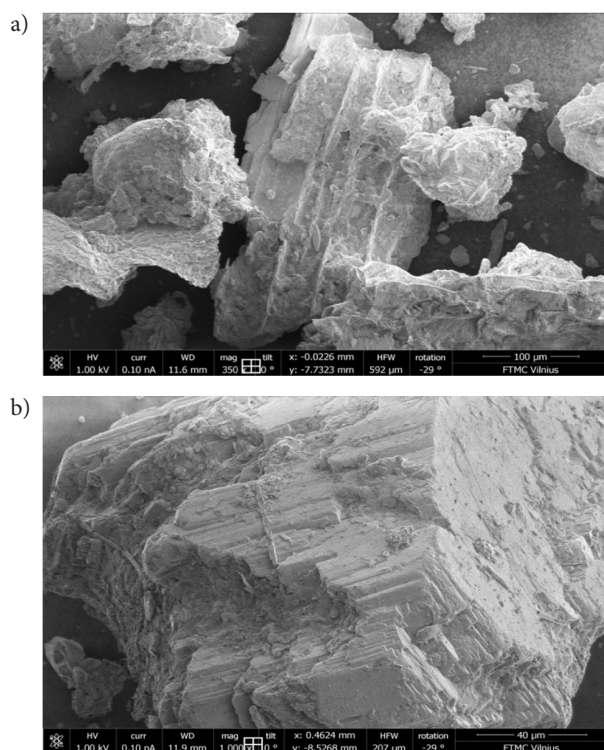
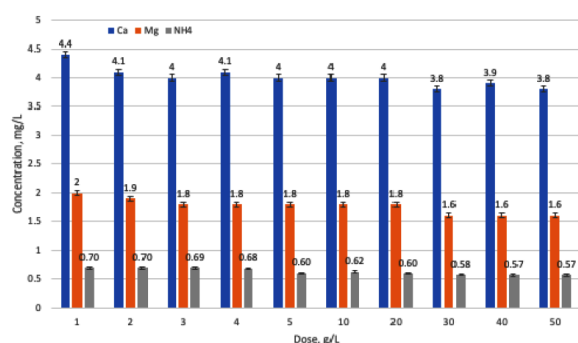
Figure 1. SEM images of sapropel microstructure at different magnifications: a – $\times 350$; b – $\times 1000$ 

Figure 2. The dependence of cations' concentration on the doses of sapropel when mixing duration is 15 min

up to 100 μm , with uneven surfaces. Smaller particles fill the voids between larger fragments.

In Figure 2, the efficiency of cations' concentration depending on dose of sapropel is provided in 15 min mixing duration. The initial concentrations of cations are 4.5 mg/L Ca^{2+} , 2 mg/L Mg^{2+} , 0.7 mg/L NH_4^+ .

Removing calcium ions, whose initial concentration in an aqueous solution is 4.5 mg/L, shows that using the lowest dose of sapropel, 1 g/L, reduces the concentration of calcium ions only minimally, to 4.4 mg/L. When increasing the doses and adding 2, 3, 4, 5, 10, and 20 g/L of sapropel to the aqueous solution, the concentration of calcium ions changes little: between 4.0 and 4.1 mg/L, and the removal efficiency reaches 11%. When 30, 40, and 50 g/L doses of sapropel were added, the calcium ion concentration decreased to 3.8–3.9 mg/L, and the removal efficiency reached 15.5%. Very similar results are seen when removing magnesium ions from an aqueous solution with an initial ion concentration of 2 mg/L. When using the lowest dose of sapropel, 1 g/L, the concentration of magnesium ions does not decrease and remains the same. When the doses are increased and 2, 3, 4, 5, 10, and 20 g/L doses of sapropel are added to the aqueous solution, the concentration of magnesium ions changes little: between 1.8 and 1.9 mg/L, and the removal efficiency reaches 10%. When 30, 40, and 50 g/L doses of sapropel are added, the magnesium ion concentration decreases to 1.6 mg/L, and the removal efficiency reaches 20%.

Different results are observed when removing ammonium ions, whose initial concentration was 0.7 mg/L, from an aqueous solution. Adding smaller doses of sapropel (1, 2, 3, 4, and 5 g/L) does not significantly affect the efficiency of ammonium cation removal. Only when 5 g/L of sapropel was added did the concentration of ammonium ions in the solution decrease by 14.3%. When larger doses of sapropel (10, 20, 30, 40, and 50 g/L) were added to the solution, the concentration of ammonium ions decreased slightly by 18.6% (40 and 50 g/L), but when comparing the concentration of ammonium ions after adding 5, 10, 20, 30, 40, and 50 g/L of sapropel, the results varied within a range of 5%. When using all sapropel doses, ammonium ions are not removed to the permissible level of 0.5 mg/L according to the World Health Organization (2011).

After analyzing the results of the study on how cations are removed from aqueous solutions by mixing them for 15 minutes and using different doses of sapropel, the following studies are presented on the effect of mixing time on the removal of cations from aqueous solutions. Since a previous study found that cations are most effectively removed when sapropel doses are 40 and 50 g/L, a more rational sapropel dose, i.e., 40 g/L, which is more economically beneficial, was used in the following study. In Figure 3, the efficiency of cations' concentration depending on the mixing time is provided for dose 40 g/L of sapropel. The initial concentrations of cations are 4.5 mg/L Ca^{2+} , 2 mg/L Mg^{2+} , 0.7 mg/L NH_4^+ .

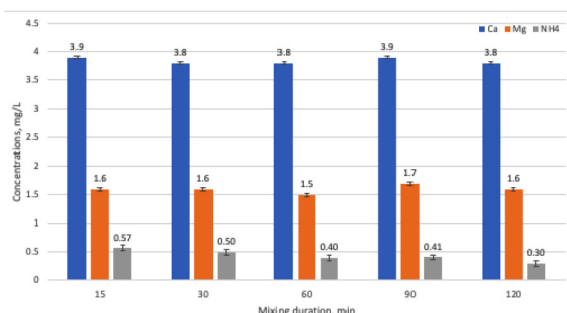


Figure 3. The dependence of cations' concentration on sorption contact time when the doses of sapropel is 40 g/L

When using a dose of 40 g/L sapropel and removing calcium ions with an initial concentration of 4.5 mg/L in an aqueous solution, it can be seen that very minimal changes occur when using different mixing times. When mixing times are extended, the changes observed are insignificant and correspond to measurement errors. The same results are observed when removing magnesium ions from an aqueous solution with an initial ion concentration of 2 mg/L. However, when removing ammonium ions with an initial concentration of 0.7 mg/L from an aqueous solution, the concentration of ammonium ions in water decreases as the mixing time is extended. The highest efficiency of ammonium ions in an aqueous solution was achieved by mixing for 120 min – 57%. It can also be seen that when mixing aqueous solutions for only 30 minutes or longer, ammonium ions are already removed to the permissible level of 0.5 mg/L according to the World Health Organization (2011).

Since it can be seen that when removing ammonium ions from aqueous solutions, using a dose of 40 g/L of sapropel, the ammonium concentration in the aqueous solution is lower than the permissible limit after just 30 minutes of mixing, it should be checked whether similar results can be achieved using lower doses of sapropel. In Figure 4, the efficiency of ammonium ions' concentration depending on the mixing time is provided for dose 10 g/L of sapropel. The initial concentrations of cations is 0.7 mg/L NH_4^+ .

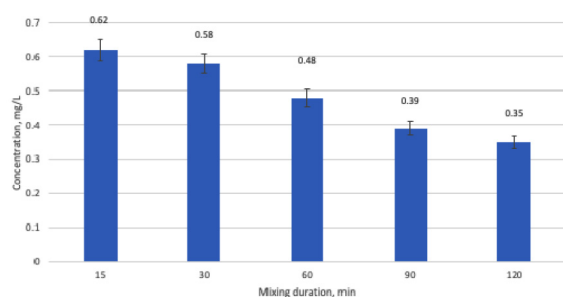


Figure 4. The dependence of ammonium ions' concentration on sorption contact time when the doses of sapropel is 10 g/L

When removing ammonium ions from aqueous solutions and using a lower dose of sapropel – 10 g/L, it

can be seen that when the sorbent is stirred for 15 and 30 minutes, the concentration of ammonium ions decreases by 11.4–17.1%, but exceeds the permissible limit of 0.5 mg/L. When mixed for 60 minutes, the concentration of ammonium ions decreases even further, with a removal efficiency of 31.4%. When mixed for longer, the concentration of ammonium ions in the solution decreases significantly, and when mixed for the longest time – 120 minutes – the ammonium ion removal efficiency reaches 50%. Comparing doses of 10 and 40 g/L of sapropel, it can be seen that when using a higher dose and stirring for 30 minutes, the concentration of ammonium ions in the aqueous solution decreases by 28.6% and is 0.5 mg/L, which does not exceed the permissible limit, while when the sorbent is mixed for 60 minutes, ammonium ions are removed by 42.8% and their concentration (0.4 mg/L) in the aqueous solution is lower than the permissible limit. Meanwhile, when using a lower dose of 10 g/L and mixing for 30 minutes, the concentration of ammonium ions in the aqueous solution decreases by 17.1% and the concentration of ammonium ions is 0.58 mg/L, which exceeds the permissible limits. When the sorbent is stirred for 60 minutes, 31.4% of the ammonium ions are removed and their concentration (0.48 mg/L) in the aqueous solution is below the permissible limit. And when mixed for another 30 minutes, the concentration of ammonium ions decreases to 0.39 mg/L, and the removal efficiency reaches 44.2%. Therefore, when selecting the sapropel dosage and mixing times for removing ammonium ions from aqueous solutions, economic and design aspects should be taken into account when choosing a lower or higher sapropel dosage and a shorter or longer mixing time.

4. Discussion

Research results show that sapropel is not very effective in removing calcium and magnesium ions from aqueous solutions, because even when using the highest doses of sapropel (50 g/L), the efficiency of calcium ion removal is only about 15.5%, and that of magnesium ions is about 20%. According to the conclusions of the study by Tipping and Hurley (1992), the ability of humic substances to sorb cations depends on various factors, including the chemical properties of metals. These scientists presented the following sequence of cation binding strengths: $\text{Mg}^{2+} < \text{Ca}^{2+} < \text{Mn}^{2+} < \text{Cd}^{2+} < \text{Co}^{2+} < \text{Ni}^{2+} \approx \text{Zn}^{2+} < \text{Pb}^{2+} < \text{Cu}^{2+} < \text{VO}^{2+}$, which shows that magnesium ions are the most difficult to absorb from humic substances. Comparing the sorption capacity of natural sapropel with artificial (alginate-citrate aerogel 96% efficiency (Wang et al., 2021) or modified sorbents (Na-X and Na-Y zeolites) (Le Van Mao et al., 1994), it can be seen that natural sapropel, despite its humic substances and colloidal properties, has limited sorption capacity for calcium and magnesium ions.

Unlike calcium and magnesium, the sorption of ammonium ions was significantly more effective and highly dependent on the sapropel dose and mixing time. When using higher doses of sapropel (40 g/L) and a long mixing time (≥ 30 min), the concentration of ammonium ions in water decreased to the permissible WHO standard (0.5 mg/L), and with longer mixing – even to 0.39 mg/L.

Statistical data analysis using linear regression was performed to evaluate the effect of sorbent dose and mixing time on the concentration of ammonium ions in the solution. The analysis showed that the concentration of ammonium ions depends on at least one explanatory variable in a statistically significant manner ($p < 0.001$). Both independent variables, sorbent dose and mixing time, were found to be statistically significant predictors of ammonium ion concentration ($p < 0.001$ for both variables), with sorbent dose exhibiting a stronger effect compared to mixing time. The predictive model describing this relationship is expressed by the following equation:

$$\text{NH}_4^+ \text{ concentration} = 0.74 - 0.006 \cdot \text{dose} - 0.002 \cdot \text{time}.$$

The model demonstrated a high goodness of fit, explaining 84.1% of the variability in ammonium ion concentration ($R^2 = 0.841$), while the adjusted coefficient of determination (adjusted $R^2 = 0.693$) confirmed that the model retains substantial explanatory power after accounting for the number of predictors.

Data showed that the sorption of calcium and magnesium ions was practically independent of the mixing time, while the sorption of ammonium ions was highly dependent on the mixing duration. The economically optimal strategy for removing ammonium ions would be to use a dose of 40 g/L of sapropel and mix the solution for 30–60 min, as this allows WHO standards to be met while maintaining efficiency and reducing sorbent use.

5. Conclusions

Studies have shown that the efficiency of calcium and magnesium ion removal using sapropel is only 15–20% when using the highest doses of 30, 40, and 50 g/L of sapropel, regardless of the mixing time. These results show that sapropel is not suitable for water softening.

When removing ammonium ions from aqueous solutions, it was found that their removal efficiency depends on the sapropel dose and mixing time. Maximum ammonium ion removal reaches 57% when mixing for 120 min. The optimal combination was found to be 40 g/L of sapropel and ≥ 30 min of mixing, when ammonium ions are removed to the permissible WHO standard of 0.5 mg/L.

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