THE USE OF FILTRATION MATERIALS TO REMOVE AMMONIA FROM WATER

VYUŽITIE FILTRAČNÝCH MATERIÁLOV NA ODSTRAŇOVANIE AMONIAKU Z VODY

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Abstract

Ammonia nitrogen is the primary product of the decomposition of plant and animal organic nitrogenous materials. Ammonium salts are part of some nitrogen fertilizers and are getting into ground and surface waters from agricultural areas. Secondary ammonium compounds can be formed directly in waters by reducing nitrates. This article is focused just on removing ammonia from drinking water, while the current research emphasizes the use of natural materials, especially for the removal of inorganic contaminants from water. The aim of this work consists in verifying the possibility of using natural filter materials - zeolites in ammonia removal and other filter materials, verify sorption (ion exchange) properties and efficiency of available types of materials under laboratory conditions.

Abstrakt

Amoniak je primárnym produktom rozkladu rastlinných a živočíšnych organických dusíkatých látok. Amónne soli sú súčasťou niektorých dusíkatých hnojív a z poľnohospodárskej plochy sa dostávajú do podzemných a povrchových vôd. Sekundárne amóniové zlúčeniny môžu byť vytvorené priamo vo vodách redukciou dusičnanov. Tento článok je zameraný práve na odstránenie amoniaku z pitnej vody, pričom súčasný výskum kladie dôraz na použitie prírodných materiálov, najmä pre odstránenie anorganických kontaminantov z vody. Cieľom tejto práce bolo overiť možnosti použitia prírodných filtračných materiálov - zeolitov a iných filtračných materiálov pri odstraňovaní amoniaku z vody, overiť sorpčné (ionovýmenné) vlastnosti a účinnosť dostupných typov materiálov v laboratórnych podmienkach.

Key words: ammonia removal, zeolite, lewatit, filtralite, klinomangan, Read-As

1 INTRODUCTION

Considering currently legal requirements to ensure the supply of "safe drinking water" in terms of water treatment technology, it is necessary to optimize the water treatment process, thus to use modern technologies which can cope with changing water quality. In many cases, water treatment is the only possible way to supply the population with quality drinking water. The issue of water treatment, however, concerns of different industries, not only the problems of drinking water, but also water for industrial, pharmaceutical and other purposes. The article focuses on verifying the possibility of using selected filter materials in removing ammonia from water.

The study by Woo-Hang deals with the evaluation of the feasibility of ammonia removal by zeolite adsorption in drinking water treatment process. They tested the feasibility using two methods: powdered zeolite dosing to coagulation tank and substituting sand for granular zeolite in a sand filter. When the zeolite dosage was above 100mg/l, the ammonia concentration decreased below 0.5mg/l of NH_4^+-N in a powdered zeolite test, but the turbidity increased to 30NTU. In a granular zeolite test, ammonia was not detected in treated water until 8 days. This result suggest that the use of granular zeolite in sand filter could remove ammonia in winter [1].

Balci and DinÇel examined the ammonium removal from the solution using Turkish sepiolite having an average particle size of 2.05 mm. Ammonium uptake experiments for two different adsorbent quantities were performed for solutions with initial solution concentrations in a range of 8.333–222.23 mmol NH_4^+/l, and equilibrium isotherms were performed at 25°C. The removal of ammonium was observed around 60%, 90% of which were achieved within 500 s [2].

The objective of a Chmielewská study consisted in determining the feasibility at the laboratory and to evaluating a pilot potential in field operation for ammonium removal from drinking water with enhanced ammonium concentration by applying Slovakian clinoptilolite-rich tuff deposited nearby Nižný Hrabovec, a village in the Eastern Slovakia. Detailed technological parameters (specific and shipping weights, porosity, sphericity, abrasion) were determined for the Slovakian clinoptilolite-rich tuff and compared with some other foreign samples including common water treatment adsorption materials like active coke and silica sand [3].

It is necessary to note the researches dealing with wastewater treatment who work with higher concentrations of ammonia nitrogen, but with similar materials and the same principle. Several works related to the wastewater treatment using bentonite, sepiolite and clinoptilolite from many different deposits are cited in the literature. Suzuki and Ha [4], investigated the ion exchange and the equilibrium for NH_4^+–Na^+, NH_4^+–K^+, and NH_4^+–Ca_2^+ systems in the channels of clinoptilolite at room temperature. It was observed that clinoptilolite has a higher selectivity coefficient for ammonium relative to sodium and calcium ions. The effective diffusion coefficient for ammonium and sodium ions in clinoptilolite was observed at around $4–5\times10−12\ m^2/s$. Bernal and Lopez [5] studied ammonia and ammonium removal using clinoptilolite and sepiolite. They showed that sepiolite also had a comparable ammonium removal capacity. Booker et al. [6] observed that the NH_4–N concentration decreased below 1 mg/l during the treatment of sewage with the concentration ranging from 25 to 50 mg NH_4–N/l using natural zeolite. Veigh and Weatherley [7] observed around 90% removal of influent ammonia in the continuous packed bed operation using clinoptilolite. Rozic et al. [8] obtained around 50% ammoniacal nitrogen removal at a low solution concentration using zeolite and natural clays. They also observed a decrease of removal efficiency upon increasing initial concentration.

The main aim of this work consists in verifying the possibilities of using natural filter materials - zeolites in ammonia removal and other filter materials, verifying sorption (ion exchange) properties and efficiency of available types of materials which were originally developed to remove other undesirable substances in the process of water treatment such as arsenic, heavy metals, reduction of water hardness under the laboratory conditions.

1.1 Occurrence and properties of ammonia

Simple ammonium salts do not form minerals in nature. They mostly come from secondary sources as wastes and from farming. Ammonia nitrogen is a primary product of the decomposition of animal and plant organic nitrogenous materials. Ammonium compounds can be formed secondarily in natural waters by a chemical reaction of nitrates and biochemical decomposition.

Its concentrations in natural water are below $0.2\ mg.l^{-1}$. Groundwaters associated with petroleum waters sometimes contain more than $100\ mg.l^{-1}$ of ammonia nitrogen. This is explained by the decomposition of organic matter under anoxic conditions where nitrification processes cannot take place.

The term ammonium includes both unionized (NH_3) and ionized (NH_4) forms. Ammonia dissolution in water gives rise to hydrate $NH_3.H_2O$ which directly dissociated to NH_4^+ and OH^- ions. In groundwater evaluation, ammonium is an indicator of possible groundwater pollution. Its values are particularly significant in association with a demonstrated bacteriological pollution, but it is very important to rule out in advance its inorganic origin or decomposition of nitrogenous organic matter of vegetable origin. Toxic effect of ammonia nitrogen on fish are considerably controlled by water pH, because it is not the NH_4^+ ion which has toxic effect, but the undissociated ammonia which more easily penetrates through cell membranes. Ammonium is the principal component of mammal metabolism. Toxic effects have been noted only if its content attains about $200\ mg.kg^{-1}$ of body weight. [9]

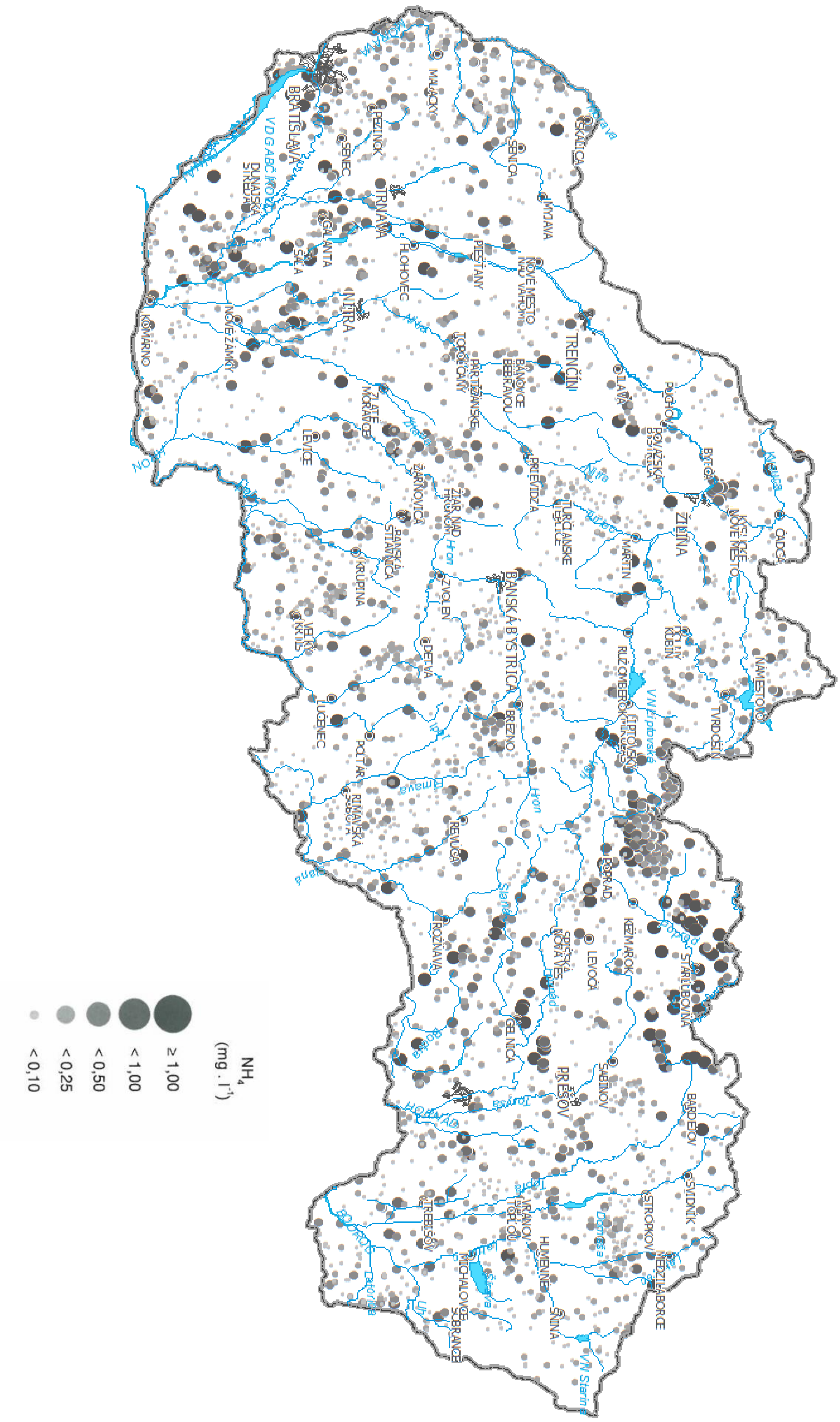

Fig. 1 Occurrence of ammonia SR [9]

WHO does not give its limit value. SR Government Regulation no. 496/2010 Coll. about *Drinking Water* permits the maximum NH_4^+ concentration of 0.5 mg.l^{-1}. An average NH_4^+ content in groundwater samples is 0.075±0.53 mg.l^{-1} as shown in Fig.1. Ammonium in groundwater is fairly evenly distributed, 98% of samples contain it in an amount below the limit value permitted by the SR Government Regulation no. 496/2010 Coll. In high-altitude areas, called headwaters, notably in their summit section with little soil and biomass, winter precipitation is the chief source of NH_4^+. Elsewhere, however, the vast majority of occurrences are man-made-agriculture and missing sewerage are their main sources [9].

2 MATERIALS AND METHODS

For this experiment was produced the stock solution with the concentration of NH_4^+ 6.8 mg.l^{-1} at pH 7.84. To each beaker was dosed 60 ml the stock solution (model raw water) and 0.6 g of each material. The experiment was conducted in a stationary mode with occasional stirring at laboratory temperature 23.4 °C. The samples was collected after 1, 4, 12 and 24 hours. Based on previous research were selected following available materials with proven efficiency for removing ammonia nitrogen and other undesirable substances:

1. Zeolite - mined in Nizhny Hrabovec, supplier Zeocem, a.s. Bystré, Slovakia; particle size: 0.5 to 1.0 mm (Fig. 2)
2. Filtralite - developed in Norway, supplier Envi-Pur, s.r.o. Prague (Fig. 3)
3. Cation exchange resin - Ostion KS (Fig. 4)
4. Read-As - supplier Global Water Kft., Hungary(Fig. 5)
5. Zeolite - mined in Nizhny Hrabovec, supplier Zeocem, a.s. Bystré, Slovakia; particle size: 1.0 to 2.5 mm (Fig. 6)
6. Lewatit TP 207 - supplier SAMPLE Bratislava, s.r.o., Slovakia, producer Lanxess AG (Fig. 7)
7. Klinomangan -BBC-NA1000-0.5-1.2 - producer Geoproduct Kft., Hungary (Fig. 8)
8. Lewatit S 108 - supplier SAMPLE Bratislava, s.r.o., Slovakia, producer Lanxess AG (Fig. 9)

Fig.2 Zeolit 0,5-1,0 mm

Fig.3 Filtralite

Fig.4 Ostion KS

Fig.5 Read-As

Fig.6 Zeolit 1,0-2,5 mm

Fig.7 Lewatit TP 207

Fig.8 Klinomangan

Fig.9 Sk Lewatit S 108

Kinetics of adsorption was carried out in a stationary mode with occasional mixing at a constant room temperature of 23 °C, and was used 0.6 g of adsorbent and 60 ml of a model solution with the initial ammonia concentration of 6.8 mg $.l^{-1}$.

Zeolite (ZeoCem Eco) is used for drinking water treatment as a filter inset. It reduces material cost by saving the costs for chemicals. It reduces iron and manganese, antimony. Zeolites also effectively remove ammonium ions from drinking water and wastewater so they can be used both for chemical filtration and as a substrate for biological treatment. As a biofilter for water purification, they provide an appropriate environment for the growth of bacterial cultures which oxidize NH_4 to NO_3^-. The structure of clinoptilolite includes a three-dimensional grid which consists of Silicate tetrahedrite $(SiO_4)^{4-}$ each interconnected via oxygen atoms; the atoms of silicon is replaced by aluminum $(AlO_4)^{5-}$ way, creating a characteristic spatial structure with a significant incidence of cavities interconnected by channels, in which metal cations, or water molecules are stored. The total volume of these cavities is from 24 to 32 % [10].

Filtralite filter media is made by burning of clay at about 1200º C, followed by crushing and sieving. Filtralite are inert ceramic particles with a dense shell surrounding a porous core. The chemical and physical composition gives the beads several properties used for many different purposes included filter materials. Filtralite's large pore volumes and surface areas make it an ideal media for biological treatment of drinking water. Biofilm processes are excellent for treating raw water containing ammonia, manganese, iron etc. Tests have shown that Filtralite works perfectly in these kinds of processes. In conventional filtration processes Filtralite can be used in different ways. In dual media filters Filtralite can be used in combination with sand. Tests have shown that Filtralite operates as well as or better than traditional filter media in these types of filters. Filtralite can not only be used in traditional drinking water treatment, but also in treatment of saline water. In desalination plants one of the most important parts is the pretreatment process. Filtralite is an excellent filter media for use in pre-treatment filters in desalination plants [11].

READ-As was provided by the company Global Water KFT. The material, granulated ceric oxide, has been developed by the Nihin Kaisui Co., Ltd. Company in Japan. READ-As is a material produced for removal of arsenic from water under a large scale of conditions and it is sorbs efficiently both arsenate and arsenate (III). Neither oxidation of arsenate (III) to arsenate, nor adjusting the pH of water before and after sorption is needed. It is possible to regenerate this material by adding natrium hydroxide followed by sodium hypochlorite and washing it with water at the end. The regenerated material needs to be neutralized by hydrochloric acid and must be washed with water before the next use. Depending on the amount of sorbent and a chemical composition of water, the regeneration should be performed after 4-12 months. After 7-10 years, it is necessary to change the whole filling of the filter as the material could be dangerous [12].

Klinomangan-BBC-NA1000-0.5-1.2 is a natural filtration medium with high content of dispersed manganese dioxide. A special technique was used for binding of manganese oxide to the surface and in micropores of zeolite, thanks to which only a low concentration of colloidal particles is washed out while the medium is in water. Manganese is present in Klinomangan in two forms: in from of manganese oxide and in ionic form in the crystal structure of zeolite. The manganese ions bound in zeolite are replaceable, which means that they can be replaced by alkaline cations. They may occure in filtered water, if the salt concentration of the water is very high. Klinomangan is an excellent adsorbent of ammonium ions. The elimination of the ammonium ions, iron and manganese runs simultaneously in one technological step. The regeneration solution, NaCl (5%) is used. The regeneration by means of NaCl restores only the absorption capacity of ammonium ions. Then it should be followed by regeneration using the Perma preparation, which restores the original oxidative capacity of the medium [13].

Ion exchange resins (Lewatit) are used to remove dissolved particles from liquids. Therefore, their functional groups are bonded to polymer beads. These functionalized polymers adsorb particular anions or cations from the liquid and discharge others. Water treatment is the best-known and biggest field of application of ion exchange resins. In household, such applications are used, among others, to soften water. They ensure that the calcium ions responsible for water hardness are replaced by sodium ions. Ion exchange resins are also used in industry, for example in power generation. There, Lewatit is used in the production of ultra-pure boiler feed water and steam to avoid incrustations and corrosion. This improves the efficiency, operating reliability and lifetime of power plants. In the cleaning of industrial effluent and treatment of groundwater, the removal of toxic substances plays a key role. A range of special Lewatit resins are able to selectively remove, for example, heavy metal ions and organic pollutants from (waste)water. Other fields of application for ion exchange resins include semi-conductor production, galvanic industry (recovery and treatment of metals such as gold, copper, nickel or cobalt in hydrometallurgy), pharmaceuticals, and the food and beverage industry. Lewatit ion exchange resins are also reliable catalysts which are crucial for many chemical reactions [14].

3 RESULTS AND DISCUSSION

The experimental work weas focused mainly on the removal of ammonium ions from drinking water; the limit value for drinking water is 0.5 mg.l^{-1}. The results achieved by volumetric analysis method are presented in tabular form (Tab. 3.1-3.9).

Tab. 3.1 Concentration of ammonia after the adsorption in mg/l

Material	Ammonia [mg/l]			
	1 h	4 h	12 h	24 h
Zeolite 0.5-1.0 mm	6.40	4.90	3.05	2.15
Filtralite	6.80	6.70	6.75	6.75
Ostion	3.65	1.45	1.25	0.50
Read-As	6.80	6.75	6.50	6.20
Zeolite 1.0-2.5 mm	6.55	5.25	4.80	3.70
Lewatit TP 107	6.80	6.60	6,.35	6.30
Klinomangán	6.60	5.10	2.85	2.10
Lewatit S 108	6.05	3.90	1.50	1.35

Tab. 3.2 Removal of ammonia from water by Zeolite 0.5-1.0 mm

Variable	Unite	*Zeolit 0,5-1,0 mm*			
t	[h]	1	4	12	24
c_m	[mg/l]	6.40	4.90	3.05	2.15
η	[%]	5.88	27.94	55.15	68.38
a_t	[μg/g]	40	190	380	470

Tab. 3.3 Removal of ammonia from water by Filtralite

Variable	Unite	*Filtralite*			
t	[h]	1	4	12	24
c_m	[mg/l]	6.80	6.70	6.75	6.75
η	[%]	0.00	1.47	0.74	0.74
a_t	[μg/g]	0.00	10	0.00	0.00

Tab. 3.4 Removal of ammonia from water by Ostion KS

Variable	Unite	*Ostion KS*			
t	[h]	1	4	12	24
c_m	[mg/l]	3.65	1.45	1.25	0.50
η	[%]	46.32	78.68	81.62	92.65
a_t	[μg/g]	320	540	560	630

Tab. 3.5 Removal of ammonia from water by Read-As

Variable	Unite	*Read-As*			
t	[h]	1	4	12	24
c_m	[mg/l]	6.80	6.75	6.50	6.20
η	[%]	0.00	0.74	4.41	8.82
a_t	[μg/g]	0.00	0.00	30	60

Tab. 3.6 Removal of ammonia from water by Zeolite 1.0-2.5 mm

Variable	Unite	*Zeolit 1.0-2.5 mm*			
t	[h]	1	4	12	24
c_m	[mg/l]	6.55	5.25	4.80	3.70
η	[%]	3.68	22.79	29.41	45.59
a_t	[μg/g]	30	160	200	310

Tab. 3.7 Removal of ammonia from water by Lewatit TP 107

Variable	Unite	*Lewatit TP 107*			
t	[h]	1	4	12	24
c_m	[mg/l]	6.80	6.60	6.35	6.30
η	[%]	0.00	2.94	6.62	7.35
a_t	[μg/g]	0,00	20	50	50

Tab. 3.8 Removal of ammonia from water by Klinomangan

Variable	Unite	*Klinomangán*			
t	[h]	1	4	12	24
c_m	[mg/l]	6.60	5.10	2.85	2.10
η	[%]	2.94	25.00	58.09	69.12
a_t	[μg/g]	20	170	400	470

Tab. 3.9 Removal of ammonia from water by Lewatit S 108

Variable	Unite	*Lewatit S 108*			
t	[h]	1	4	12	24
c_m	[mg/l]	6.05	3.90	1.50	1.35
η	[%]	11.03	42.65	77.94	80.15
a_t	[μg/g]	80	290	530	550

The instantaneous adsorption capacity at (1) and adsorption efficiency η (2) is calculated according to the formulas:

$$a_t = \frac{(c_o - c_m)V}{m} \qquad (1)$$

$$\eta = \frac{(c_o - c_m)100}{c_o} \qquad (2)$$

where:

c_o – concentration of ammonia before adsorption (6.8 mg/l) $\left[\frac{mg}{l}\right]$,

c_m – concentration of the ammonia after adsorption at the time t $\left[\frac{mg}{l}\right]$,

V – volume of the aqueous solution (0.060 l) $[l]$,

m – weight of sorbent material (0.6 g) $[g]$,

η – adsorption efficiency $[\%]$,

a_t – instantaneous adsorption capacity; adsorbed amount per unit weight of sorbent material at time t $\left[\frac{mg}{g} / \frac{\mu g}{g}\right]$,

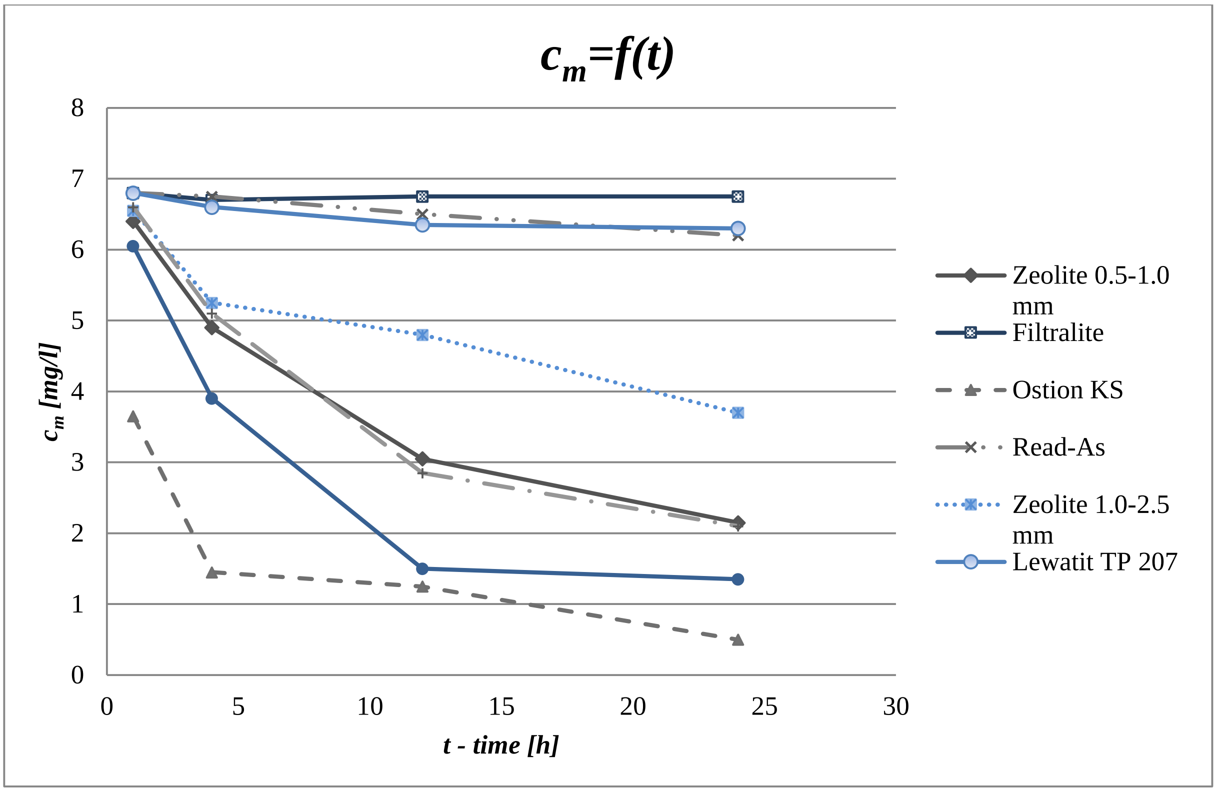

Fig.10 Dependence of ammonia concentration from adsorption time

As shown in Tab.3.4 and Tab.3.9, the highest adsorption capacity for ammonia removal was achieved by the cation exchangers Ostion KS and Lewatit S 108 which presented their ion exchange properties as they are high molecular compound (polymer resin) able to exchange sodium for different cation with high efficiency.

Klinomangan and natural adsorbent zeolite clinoptilolite - mined in Nižný Hrabovec, are the second most effective adsorbent depending on their particle size; a finer particle size has higher efficiency (Tab. 3.2, Tab. 3.8). Broad application possibilities of zeolites (e.g. in construction, agriculture, water purification and treatment, pharmacology) are the result of their unique physicochemical properties such as ion exchange and sorption capacity, relatively large specific surface or economic access. The three-dimensional network of the tetrahedron structure of SiO_4^{4-} and AlO_4^{5-} and the permanent negative charge in the structure makes it possible to bind exchangeable cations [15, 16, 17].

The graph (Fig.10) shows the change in ammonia concentration with adsorption time, which means that the concentration of both substances decrease with time during the ongoing adsorption. It is expected that under a longer influence of adsorbent in a model solution, the ammonia concentration will not significantly decrease in the case of non-active materials (it follows from the plot $c_m = f(t)$) and sorption properties of these selected materials will be exhausted under the given laboratory conditions. In case of an active material, the dependence $c_m = f(t)$ shows a constant decrease in the ammonia concentration unless the sorption properties of the material are exhausted.

It is necessary to emphasize the inclusion of the cation exchangers in the experiment as a reference material due to its ion exchange properties and proven efficiency. From previous research and from the actual experiment, it is clear that the use of zeolites and their modified forms achieves high efficiency in the removal of ammonia (and other undesirable substances). In the case of materials, primarily developed for the removal of different undesirable substances from water than ammonia, their inclusion in the experiment didn't prove validity.

4 CONCLUSIONS

The study of adsorption properties of selected natural and synthetic adsorbents was performed in order to determine and compare the adsorption efficiency of the NH_4^+ removal from a model solution in the stationary laboratory experiment. It is clear, from the obtained results, that several of the materials can be used for ammonia removal; natural materials - clinoptilolite zeolites are able to remove NH_4^+ with an efficiency of about 45-68%, klinomangan with efficiency of about 69% and cation exchangers with their ion exchange properties with the efficiency of about 80-92%, however not to the legislative required value 0.5 mg l^{-1}. Other tested materials did not demonstrate the efficiency.

The next task will be to study the influence of selected parameters on the adsorption process in water treatment and transformation of the stationary experiments into dynamic regime consisting of a set of experimental columns, 5 cm in diameter.

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RESUMÉ

Vzhľadom na dnes oprávnenú požiadavku zaistenia dodávky „bezpečnej pitnej vody“ z hľadiska technológie úpravy vody, je potrebné optimalizovať proces úpravy vody, resp. začať prevádzkovať nové moderné technológie, ktoré sa vedia s meniacou sa kvalitou vody vysporiadať. V mnohých prípadoch je úprava vody jediným možným spôsobom, ako zásobovať obyvateľstvo kvalitnou pitnou vodou. Problematika úpravy vody sa však týka rôznych odvetví, nielen problematiky pitnej vody, ale aj vody pre priemyselné, farmaceutické a iné

účely. Tento článok je zameraný na overenie možnosti využitia vybraných filtračných materiálov pri odstraňovaní amoniaku z vody.

Amoniakálny dusík je primárnym produktom rozkladu rastlinných a živočíšnych organických dusíkatých látok. Amónne soli sú súčasťou niektorých dusíkatých hnojív a z poľnohospodárskych plôch sa dostávajú do podzemných a povrchových vôd. Sekundárne môžu amónne zlúčeniny vznikať priamo vo vodách redukciou dusičnanov. Tento jav nastáva v podzemných vodách vo väčších hĺbkach, ktoré obsahujú zvýšené koncentrácie železa a mangánu, prípadne aj sulfán a jeho iónové formy. Amoniakálny dusík obsahujú odpadové vody z plynární, koksární a galvanizovní. Môžu ho obsahovať aj pitné vody dezinfikované chlóramináciou. Do atmosférických vôd sa dostáva z priemyselných exhalátov. Pri rozpúšťaní amoniaku vo vode vzniká hydrát $NH_3.H_2O$, ktorý priamo disociuje na ióny NH_4^+ a OH^-. Zvyšujúca teplota podporuje disociáciu NH_4^+ na NH_3. Podzemné vody a čisté povrchové vody majú koncentráciu dusíka (NH_3 + NH_4^+) asi do 0,1 $mg.l^{-1}$. Väčšie koncentrácie dusíka v podzemných vodách sa vysvetľujú chemickou redukciou dusičnanov, pokiaľ nie sú organického pôvodu. [18]

Amoniak môže byť detegovaný v pitnej vode prostredníctvom série dobre zavedených metód. Voľba použitej metódy bude závisieť od prítomnosti interferencií. V komunálnych odpadových vodách, môže byť amoniak odstránený pomocou biologického čistenia (riadená nitrifikácia) a fyzikálno-chemických procesov, ako je breakpoint chlorácia, iónová výmena a membránová filtrácia.

V rámci experimentu boli testované viaceré materiály: zeolity (Zeocem, a.s. Bystré) Filtralite (Envi-Pur, s.r.o.), ionexy Lewatit (Lanxess AG), Read-As (Global Water Kft.) a Klinomangán (Geoproduct Kft.). Štúdium adsorpčných vlastností na vybraných prírodných a syntetických adsorbentoch bolo vykonané s cieľom zistiť a porovnať ich adsorpčné účinnosti pri odstraňovaní NH_4^+ z modelového roztoku v laboratórnych podmienkach, zatiaľ len stacionárnym pokusom. Zo získaných výsledkov je zrejmé, že niektoré materiály sú použiteľné pri odstraňovaní amoniaku; prírodný materiál - zeolity typu klinoptilolit sú schopné odstraňovať NH_4^+ s účinnosťou cca 45-68%, klinomangán s účinnosťou cca 69% a katexy so svojimi ionovýmennými vlastnosťami s účinnosťou cca 80-92%, avšak nie na legislatívne požadovanú hodnotu 0.5 $mmol.l^{-1}$. Ostatné testované materiály nepreukázali účinnosť.