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Biofilters efficiency in removing ammonium from water intended for human consumption

Eficiența biofiltrelor în eliminarea amoniului din apa destinată consumului uman

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Abstract. Nitrogen is the most abundant element of Earth atmosphere and is a component part of all living organism, being found in DNA and RNA. In most ecosystems, ammonium is the main source of nitrogen for plants. In natural water sources, ammonium concentrations are usually low, but due to industrial, agricultural and other human activities, its concentration is quite high. In water supply systems it leads to degradation of water quality and to formation of other by-products with effects on human health. Existing technologies used to reduce the ammonium concentration in water are ion exchangers, biological filtration, air stripping, breakpoint chlorination and reverse osmosis. This article is based on experimental tests aimed to determine the efficiency of biofilters in reducing the ammonium concentrations in water, depending on the carrier/filtration media used in the biofilters, depending on the ammonium concentration in raw water and depending the contact time between water and the attached biomass, but also the impact of the sudden change of ammonium concentration in the raw water on the biofilters efficiency.

Key words: nitrogen, biofilters

1. General data

Nitrogen is an essential nutrient for plants and animals [1]. In nature, nitrogen is found in many forms, and its transformation from one form to another is described by the nitrogen cycle. The atmosphere is made up of 79% nitrogen gas. Although the atmosphere is practically an inexhaustible reservoir of nitrogen gas, nitrogen must be first combined with hydrogen or oxygen in order to be assimilated by plants and then to be consumed by animals [2].

In living organisms, nitrogen is found in amino acids, in proteins, in nucleic acids (DNA and RNA) and in the energy transfer molecule, adenosine triphosphate. The human body contains 3% nitrogen by mass.

Ammonia is a colorless gas, which has a pungent odor at room temperature and which under pressure can become liquid. Ammonia dissolves easily in water, in which most of the ammonia (depending on temperature, pH and amount of dissolved matter) is converted to ammonium cations by protonation. In water, a balance is established between dissolved ammonia and ammonium [3].

Ammonium and nitrate are the primary sources of nitrogen for plants, but the assimilation of ammonium requires less energy than the assimilation of nitrate and for this reason ammonium is the first element absorbed when plants are nitrogen deficient. However, large amounts of ammonium can be toxic, so the absorption of ammonium must be controlled to avoid its toxicity to plants [4].

Ammonium itself has no toxic effects on the human or animal health and would not limit the use of water, its sanitary importance consists in the fact that it indicates the pollution of water with other chemical or especially bacteriological elements that can have harmful effects on human health [3] [5].

Ammonium concentrations in non-degraded natural water sources are usually low, but ammonium concentrations from intensive agriculture, industrial and other human activities have a negative impact on water sources.

In particular, in the case of surface water sources, the presence of ammonium in very high concentrations decreases the dissolved oxygen concentration and leads to toxicity of the aquatic environment, increased corrosion rate in the soil, algae development and finally leads to eutrophication of lakes. Eutrophication is a global problem and has been identified as a major problem for water source management [6].

In the drinking water treatment plants, the excess concentrations of ammonium are undesirable because the presence of excess ammonium in water leads to an increase in the consumption of chlorine used in disinfection and thus to a reduction of the efficiency in the disinfection stage of the treatment plant. Ammonium can interfere with manganese removal filters, as oxygen would be used largely for ammonium nitrification, resulting in an earthy taste [7].

Its presence in the distribution networks leads to the development of bacteria on the pipes walls of which the distribution network is composed and to the oxidation of ammonium to nitrites and nitrates.

Nitrites and nitrates formed by oxidative conditions in distribution networks are toxic compounds that lead to the oxidation of divalent iron from hemoglobin to trivalent iron, preventing the transport of oxygen to tissues and leading to methemoglobinemia, especially at infants [3] [8].

Ammonium leads to the degradation of water quality by changing its taste and smell, which can create problems of water acceptability to consumers [3].

In Romania, the maximum allowed concentration for ammonium in drinking water, according to Law 458/2002 is 0.50 mg/l. Also, the 2184 /2020/EU Directive impose a maximum concentration of 0.5 mg/l for ammonium [9][10].

Over time, several methods have been developed to reduce the ammonium concentration in water, namely: physical, chemical, biological methods or a combination of these methods. These mainly include: ion exchangers, biological filtration, air stripping, breakpoint chlorination and reverse osmosis [11].

Nitrification is the biological process of ammonium oxidation to nitrate with the intermediate form nitrite. Nitrification is performed by species of bacteria called

Nitrosomonas and Nitrobacter [12]. These two species of bacteria are distinguished from each other by the ability to oxidize only certain nitrogen compounds. Nitrosomonas oxidizes ammonium to nitrite and Nitrobacter oxidizes nitrite to nitrate. Both species of bacteria are classified as autotrophic organisms (they do not need a source of organic carbon, they use carbon dioxide for synthesis) [13] [14] [15].

The stoichiometric equations of nitrification for Nitrosomonas and Nitrobacter are [11]:

$$NH_4^+ + 1.5\ O_2 \rightarrow 2H^+ + H_2O + NO_2^-\ \text{(Nitrosomonas)} \quad (1)$$

$$NO_2^+ + 0.5\ O_2 \rightarrow NO_3^-\ \text{(Nitrobacter)} \quad (2)$$

$$NH_4^+ + 2\ O_2 \rightarrow NO_3^- + 2H^+ + H_2O \quad (3)$$

The above equation represents the compound equation of ammonium oxidation to nitrate made by Nitrosomonas and Nitrobacter.

Nitrification process is influenced by certain factors such as temperature, dissolved oxygen concentration, pH and inhibitor concentration [13] [14].

The biological processes used to reduce ammonium in water for human consumption are mainly processes with biomass attached. Biofilters are characterized by the presence of microorganisms that adhere to the filtration media in the form of a fixed film (biofilm) [16].

For any filtration media used, all biofilters follow the same principle, namely: biological degradation of pollutants by microorganisms fixed on the filtration media surface.

2. Materials and Methods

2.1. Purpose of experimental tests

In order to reduce the ammonium concentration in water, in the performed experimental tests, it was proposed and used a biological processes with attached biomass.

The main purpose of the experimental tests was to determine the efficiency of biofilters for reducing ammonium concentrations in water, depending on the filtration media used in the biofilter, on the ammonium concentration in the raw water and on the contact time between water and attached biomass. In addition to this main purpose, the experimental tests aimed to determine the impact of sudden change of the ammonium concentration in raw water on the biofilter efficiency in reducing its concentration in water, compared to the biofilter efficiency obtained after a period of one week in which synthetic raw water with the same ammonium concentration was introduced into the biofilters.

2.2. Experimental stand

The experimental tests were performed at laboratory level on an experimental stand that is part of the Colentina Laboratory Complex, a complex belonging to the Technical University of Civil Engineering of Bucharest.

The experimental stand is represented by a pilot installation, consisting of 3 biofilters with fixed bed and ascending flow.

The following figure shows the technological scheme of the experimental stand of biofilters and its component objects.

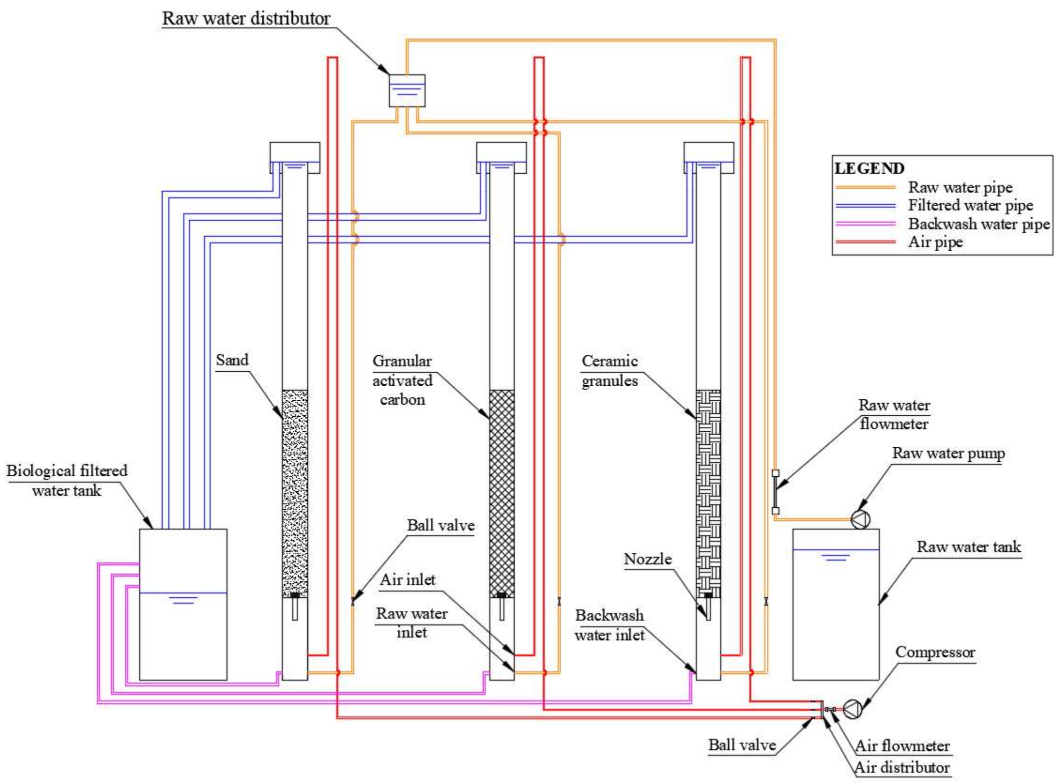

Figure 1. Technological scheme of biological filtration installation and component objects.

Considering that the experimental stand consists of 3 biofilters and because 3 filtration media were available, in the experimental tests, in each biofilter only one type of filtration media was used. The filtration media used and analyzed in the experimental tests are: sand, ceramic granules and granular activated carbon. These are shown in the following figure.

Figure 2. Filtration media.

2.3. Raw water

2.3.1. Raw water from well. The experimental stand is based on biological processes with biomass attached, and to help the start of the biological process and maintain its stability until the end of experimental tests, it was decided to use untreated water with bacteriological development potential.

The raw water used in the experimental tests comes from a well near Bucharest.

The following table presents the quality parameters of the raw water from the well and used in the experimental tests.

Table 1. Raw well water quality parameters.

Nr.	Water quality parameter	Minimum values	Average values	Maximum values
1	Ammonium [mg/l]	0.17	0.63	1.12
2	Nitrates [mg/l]	91.80	146.40	230.10
3	Nitrites[mg/l]	0.040	0.285	0.575
4	Phosphorus [mg/l]	0.029	0.032	0.036
5	pH	6.71	7.78	8.24
6	Conductivity [μS/cm]	845.00	1089.38	1327.00

2.3.2. Syntetic raw water In order to increase and vary the ammonium concentration in the raw water, up to the ammonium concentration necessary to perform the test, synthetic raw water was prepared by adding ammonium chloride NH_4Cl in the raw water from the well.

To obtain the desired ammonium concentration in raw water, has been calculated the required dose of ammonium chloride to be added in water.

2.4. Water parameters

To determine the raw water and biologically filtered water quality parameters, the existing glassware and equipments of the Colentina laboratory was used.

Ammonium, nitrates, nitrites and phosphorus concentrations were determined with the Hach spectrophotometer DR3900, using the reagents and methods of determination specified in the spectrophotometer user manual.

Determinations of pH, conductivity, temperature and dissolved oxygen was performed using a multimeter with two channels Hach HQ440D and a pH-meter Marvel pH526, using the provided sensors.

2.5. Initiation of the biological process

Unlike other existing technologies for reducing the ammonium concentration in water, biofilters cannot be used immediately, and require a period of priming of the biological process. In order to perform the experimental tests, a period of the biological process priming was needed, respectively a period for Nitrosomonas and Nitrobacter growth.

According to the analyzes performed, after the water was transported 3-4 times per week constantly, without even a week of interruption due to traffic restrictions generated by SARS-COV 2 virus, after about a month later, the biological process was primed.

2.6. Experimental tests

In the experimental tests were proposed and performed 3 test phases, in which the efficiency of biofilters in reducing the ammonium concentration in water was analyzed.

The tests performed in phases I and II, aimed to evaluate the efficiency of the biological process on each filtration media, depending on the ammonium concentration in raw water and depending on the contact time between water and biomass. The tests performed in phase III aimed to increase the efficiency of the biological process in reducing the ammonium concentration in raw water by evaluating the influence of external sources of nutrients in case of their addition to water.

The following table shows the raw water flows and ammonium concentrations in experimental tests.

Table 2. Experimental phases

Phase	Test	$Q_{raw\ water}$ [l/h]	$Q_{raw\ water/filter}$ [l/h]	$C_{ammonium}$ [mg/l]	Remarks
I	1	60	20	≅2	Without nutrients
	2	60	20	≅5	
	3	60	20	≅10	
II	1	105	35	≅2	
	2	105	35	≅5	
	3	105	35	≅10	
III	1	60	20	≅5	With nutrients

Even if the amount of ammonium chloride added into the water was calculated to obtain the desired concentration, in reality the concentrations obtained were approximately equal to those desired. The names used for ammonium concentrations in this article are:

- low concentrations: 0.5-4 mg/l;
- average concentrations: 4-8 mg/l;
- high concentrations: 8-12 mg/l.

3. Results and Discutions

The pilot plant was monitored by measurements of factors influencing the biological process, such as temperature, pH and dissolved oxygen.

The nitrification process takes place at temperatures between 4 and 45°C, with an optimal temperature of 35°C for Nitrosomonas and an optimal temperature of 35-42°C for Nitrobacter [14].

For an optimal functioning of the nitrification process, it is recommended that the minimum level of dissolved oxygen concentration be 2 mg/l [14].

For the operation of the nitrification process, it is recommended that the pH be maintained in the range of 6.5-8, with an optimal pH of 7.2 [14].

3.1. Biofilter's efficiency in phase I

The first phase consisted of 3 tests, in which, in each biofilter, raw water was introduced with a flow rate of 20 l/h and with 3 different ammonium concentration (one concentration for each test).

The following table presents the results of experimental tests obtained for biologically filtered water on the three biofilters in phase I, after normal operation for one week with approximately constant ammonium concentration in raw water.

Table 3. Raw water and biologically filtered water quality parameters in phase I - operation for one week with approximately equal ammonium concentration in raw water

Item	Water sample	NH_4^+ [mg/l]	Efficiency [%]	P [mg/l]	NO_3^- [mg/l]	NO_2^- [mg/l]	PH	Cond. [µS/cm]	DO[e] [mg/l]	T [°C]
$C_{1\text{-}NH_4^+}$= 1.76 mg/l										
1	SRW[a]	1.76	-	0.032	91.8	0.08	6.71	945	7.31	23.3
2	BFWS[b]	0.38	78%	0.029	95.3	0.049	7.68	933	5.11	24.3
3	BFWCG[c]	0.24	86%	0.028	96.1	0.05	7.51	871	5.12	24.1
4	BFWAC[d]	0.59	66%	0.028	95.1	0.044	7.56	965	4.81	24.3
$C_{2\text{-}NH_4^+}$= 4.68 mg/l										
5	SRW[a]	4.68	-	0.032	163.3	0.204	7.82	1004	6.88	22.8
6	BFWS[b]	0.69	85%	0.028	173.9	0.41	7.21	936	4.74	23.4
7	BFWCG[c]	0.51	89%	0.026	175.0	0.522	7.46	937	4.74	23.3
8	BFWAC[d]	0.95	80%	0.027	174.3	0.486	7.27	1004	4.64	23.2
$C_{3\text{-}NH_4^+}$=10.69 mg/l										
9	SRW[a]	10.69	-	0.03	230.1	0.575	8.24	1232	7.00	22.1
10	BFWS[b]	3.79	65%	0.022	251.5	0.581	8.25	1158	5.14	22.4
11	BFWCG[c]	0.56	95%	0.024	262.7	0.586	8.06	1168	4.95	22.5
12	BFWAC[d]	2.9	73%	0.025	255.1	0.583	8.06	1186	4.70	22.4

[a] Synthetic raw water.
[b] Biologically filtered water on sand.
[c] Biologically filtered water on ceramic granules.
[d] Biologically filtered water on granular activated carbon.
[e] Dissolved oxygen.

Analyzing the results obtained for ammonium and nitrates, it can be seen that the ammonium concentration in biologically filtered water decreases and the concentration of nitrates increases, resulting that the biological nitrification process works, ammonium being oxidized to nitrate.

The following figure shows the efficiencies obtained on each biofilter in phase I in order to reduce the ammonium concentration in the water, when installation was operated for one week with approximately equal ammonium concentration in water.

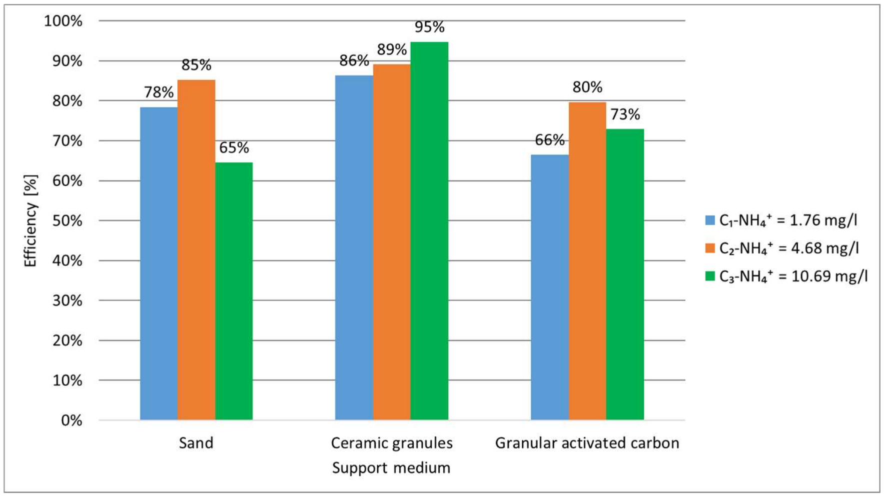

Figure 3. Biofilter efficiency of reducing the ammonium concentration depending on the filtration media used and the ammonium concentration in the raw water introduced in the biofilters - phase I.

As it can be seen in the figure above, in phase I, for any ammonium concentration in raw water introduced in biofilters, the highest efficiencies for ammonium concentration reduction was obtained for the biofilter having ceramic granules as filtration media. For this biofilter, it is observed that if ammonium concentration in the raw water is higher, the efficiency of the biofilter in reducing ammonium increases.

The biofilter that uses sand as filtration media, has higher efficiencies in reducing ammonium at low and medium concentrations, compared to the biofilter with granular activated carbon. At high concentrations, the granular activated carbon biofilter is more efficient than the sand biofilter.

For both, sand biofilter and the granular activated carbon biofilter, the best efficiency is obtained at average ammonium concentrations in raw water.

3.2. Biofilter's efficiency in phase II

The second phase consisted of 3 tests, in which, in each biofilter, raw water was introduced with a flow rate of 35 l/h and with 3 different ammonium concentration (one concentration for each test).

The following table shows the results of experimental tests obtained for biologically filtered water on the three biofilters in phase II, after normal operation for one week with approximately constant ammonium concentration in water.

Table 4. Raw water and biologically filtered water quality parameters in phase II - operation for one week with approximately equal ammonium concentration in raw water

Item	Water sample	NH_4^+ [mg/l]	Efficiency [%]	P [mg/l]	NO_3^- [mg/l]	NO_2^- [mg/l]	PH	Cond. [µS/cm]	DO [mg/l]	T [ºC]
$C_{1\text{-}NH_4^+}$=2.81 mg/l										
1	SRW[a]	2.81	-	0.035	145.2	0.04	7.76	1321	7.70	22.1
2	BFWS[b]	1.39	51%	0.027	149.3	0.18	7.95	1196	5.45	22.1
3	BFWCG[c]	0.62	78%	0.029	150.7	0.08	8.09	1137	5.35	22.2
4	BFWAC[d]	0.77	73%	0.028	149.9	0.038	8.04	1139	5.40	22.1
$C_{2\text{-}NH_4^+}$=5.15 mg/l										
5	SRW[a]	5.15	-	0.032	99.6	0.261	8.08	890	7.23	23.7
6	BFWS[b]	3.41	34%	0.028	104.4	0.374	8.11	845	4.83	23.5
7	BFWCG[c]	1.83	64%	0.026	108.2	0.555	8.15	815	5.00	23.7
8	BFWAC[d]	1.87	64%	0.027	109.0	0.144	8.19	879	4.56	23.9
$C_{3\text{-}NH_4^+}$=11.03 mg/l										
9	SRW[a]	11.03	-	0.029	148.4	0.551	8.11	845	7.16	24.6
10	BFWS[b]	6.59	40%	0.027	161.5	0.58	8.05	851	4.86	24.7
11	BFWCG[c]	1.94	82%	0.027	176.7	0.586	7.7	858	4.76	24.6
12	BFWAC[d]	4.97	55%	0.03	167.2	0.582	7.98	861	4.58	24.9

Similar to phase 1, the nitrate concentration increases and the ammonium concentration decreases, resulting that the biological nitrification process works, ammonium being oxidized to nitrate.

The following figure shows the efficiencies obtained on each biofilter in phase II in order to reduce the ammonium concentration in water, when installation was operated for one week with approximately equal ammonium concentration in water.

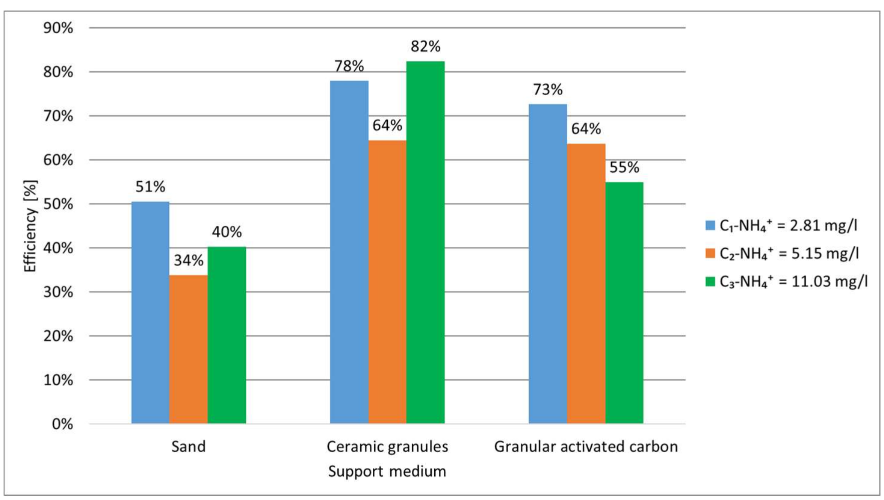

Figure 4. Biofilter efficiency of reducing the ammonium concentration depending on the filtration media used and the ammonium concentration in the raw water introduced in the biofilters - phase II.

As it can be seen in the figure above, in phase II, for any ammonium concentration in raw water introduced in biofilters, the highest efficiencies in reducing ammonium were obtained for the biofilter having as filtration media ceramic granules, followed by the biofilter with granular activated carbon and sand biofilter. For this biofilter with ceramic granules, it is observed that in case of high concentrations of ammonium, the best efficiency in reducing ammonium in water was obtained, followed by efficiency for low concentrations and efficiency for average concentrations.

The biofilter that uses sand as a filtration media, has the lowest efficiencies in reducing ammonium it is observed that if the concentration increases, the efficiency of this biofilter decreases.

In the case of the biofilter that has granular activated carbon as filtration media, it is observed that if ammonium concentration in the raw water is higher, the efficiency of the biofilter in reducing the ammonium decreases.

As it can be seen in the previous table, a reduction of the contact time between water and the attached biomass, by introducing a higher flow through the installation, the efficiency decreases for all biofilters, but the least affected is the biofilter with ceramic granules, being also the most efficient biofilter in reducing the ammonium concentration at low or high contact time.

For both phases, in case of sudden change of ammonium concentration in raw water for example from 5 mg/l to 10 mg/l there was a small decrease in the efficiency of biofilters, but the most relevant results are those at the end of the week, after normal operation of biofilters.

3.3. Biofilter's efficiency in phase III

In the tests performed in phases I and II, approximately 95% efficiencies were obtained in the reduction of ammonium concentration for the biological filter with ceramic granules, but for any ammonium concentration in the raw water, after biological filtration, the ammonium concentration does not fall below the minimum value of 0.24 mg/l.

In the first phases it was also observed that the concentration of phosphorus in water is very low, phosphorus being a necessary nutrient in the nitrification process. For this reason, in this phase, a new test was performed, with the addition of phosphoric acid (0.26 mg/l) in the synthetic raw water, to check if a better efficiency in reducing ammonium is obtained.

In order to be able to compare the results obtained with the previous tests, the same water flow (20 l/h on each filter) was used and a concentration approximately equal to 5 mg/l, respectively the raw water flow and the ammonium concentration in the water, were approximately equal to those in test 2, from phase I.

The following figure shows the efficiencies obtained for biological filtration in phase III.

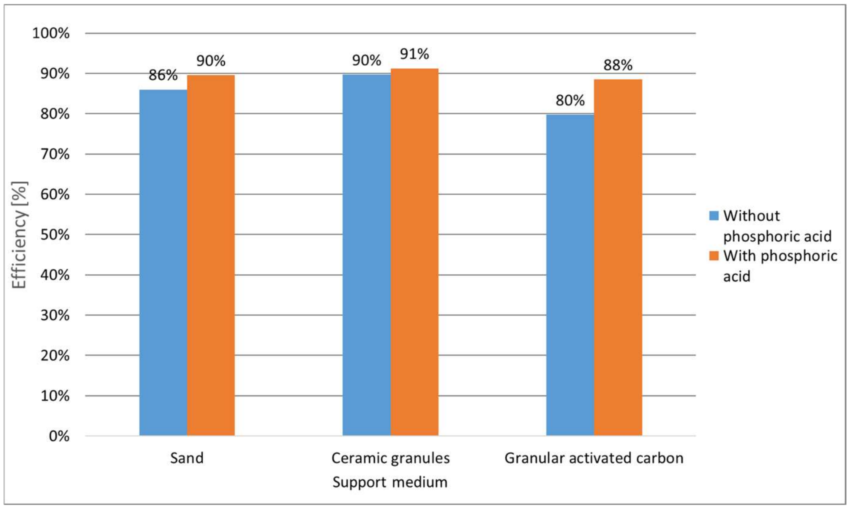

Figure 5. Biofilters efficiency of reducing the ammonium concentration depending on the filtration media used - phase III.

Comparing the results obtained regarding the efficiencies of the biological process in reducing ammonium for normal operation and operation with the addition of phosphoric acid, it is observed that similar efficiencies are obtained in both cases, with one small exception, a small increase in ammonium concentration reduction efficiency for raw water with phosphoric acid.

4. Conclusions

According to the results obtained from the experimental tests, the following can be stated:

- The biological nitrification process worked during the experimental tests, considering that a reduction of ammonium concentration was obtained simultaneously with an increase of nitrate concentration in treated water, indicating oxidation of ammonium to nitrate;
- The results obtained after the operation of the installation for a week with an approximately constant ammonium concentration in the synthetic raw water, indicate that in all tests in the three phases, the biofilters that use ceramic as a filtration media have the highest efficiency (over 64%) in reducing ammonium, for all ammonium concentration in the raw water introduced into the biofilters;
- The biological process is affected by the contact time reduction between water and the attached biomass, because according to the results, the efficiencies of all biofilters decreased as the contact time decreased. However, the biofilter with ceramic granules was the least affected by the reduction of the contact time, being the most efficient biofilter in reducing the concentration of ammonium in the raw water in all the analyzed cases.
- In case of sudden change of ammonium concentration in raw water for example from 5 mg/l to 10 mg/l there is a decrease in the efficiency of biofilters, but the most relevant results are those at the end of the week, after normal operation of biofilters.

• The efficiency of the biological process is not significantly improved by the addition of phosphoric acid.

• Regarding the conformation of ammonium concentrations in biologically filtered water, at the maximum allowable concentration according to Law 458/2002, it can be said that in the case of biofilter with ceramic granules, at longer contact time between water and attached biomass, the ammonium concentration in filtered water was below 0.5 mg/l at concentration of 2 mg/l in raw water, and at a concentrations of 5 and 10 mg/l in raw water a maximum concentration of 0.59 mg/l in filtered water was obtained, close to the limit. For the other biofilters, good efficiencies were obtained at high contact time and low ammonium concentrations in raw water, but when the contact time was reduced, the efficiencies obtained were very low compared to the biofilter with ceramic granules.

• Biofilters can be used successfully in reducing ammonium concentrations in water, require low operating costs, and in the case of concentrations close to the limit, a secondary treatment with chlorination at breakpoint can be used, but the required doses of chlorine would be much lower than a direct treatment with chlorine (without biofilters).

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