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AMMONIA NITROGEN REMOVAL IN A FILTRATION PROCESS BY ZEOLITE MODIFIED MANGANESE BED

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USUWANIE AZOTU AMONOWEGO W PROCESIE FILTRACJI PRZEZ ZŁOŻE ZEOLITU MODYFIKOWANEGO MANGANEM

W publikacji przedstawiono badania nad wykorzystaniem zeolitu naturalnego modyfikowanego do doczyszczania ścieków komunalnych. Badania przeprowadzono w skali laboratoryjnej w układzie sekwencyjnych reaktorów porcjowych typu SBR oraz złoża filtracyjnego, którego wypełnienie stanowił zeolit modyfikowany manganem. Procesowi poddawano ścieki syntetyczne przygotowane w oparciu o niezbędne składniki organiczne oraz mineralne. Ścieki po procesie biologicznego oczyszczania osadem czynnym w reaktorze kierowano na kolumnę filtracyjną. Ze względu na cykliczność pracy reaktora ścieki oczyszczano na złożu w sposób porcjowy, utrzymując je w formie zalanej. Badania prowadzono kilkuetapowo: bez regeneracji złoża, z regeneracją okresową oraz regeneracją cykliczną po każdorazowym cyklu pracy reaktora SBR. Uzyskane wyniki usunięcia związków azotu, fosforu oraz węgla zweryfikowano na ściekach rzeczywistych. W tym celu na kolumnę filtracyjną podawano ścieki biologicznie oczyszczone z osadnika wtórnego oczyszczalni miejskiej w sposób ciągły, regenerując różnymi metodami po wyczerpaniu właściwości katalitycznych złoża. Przeprowadzona weryfikacja wykazała dużą skuteczność zastosowanego układu doczyszczania ścieków w zakresie usuwania azotu amonowego oraz związków węgla i fosforu. Obserwowano również obniżenie przewodności właściwej ścieków oraz niewielki wzrost stężenia azotu azotanowego.

Summary

The paper presents research on using natural zeolite modified for cleaning municipal sewage thoroughly. The research was carried out in laboratory scale in system composed of Sequencing Batch Reactors and filter bed with zeolite modified with manganese ions. Synthetic sewage prepared on the basis of organic and mineral components was used for cleaning process. After biological treatment with activated sludge in the reactor, the sewage was directed to filtration column. Due to the fact that reactor was operating in cycles; the sewage was treated on the bed in portioned way and kept submerged. The research was carried out in a few stages: without regeneration of the bed, with periodic regeneration and cyclical after each cycle of SBR reactor operation. Obtained results of nitrogen, phosphorous and carbon compounds removal were verified on real sewage. For that purpose biologically treated sewage was directed in a continuous way to a filtration column from secondary settling tank of sewage treatment plant, regenerating bed with different methods after exhausting its catalytic properties. Verification showed great effectiveness of used configuration for removal ammonia nitrogen, carbon and phosphorous compound from sewage. Decrease in electrical conductivity of sewage and a slight increase of nitrate nitrogen concentration was observed. The research showed great effectiveness of proposed filtration configuration for sewage treatment and a possibility of bed regeneration with keeping its work effectiveness.

INTRODUCTION

Sequencing batch reactors are machines of simple construction, in which one working cycle consists of phases such as filling, mixing, aeration, settling and decanting of treated sewage. Depending on processing phase's configuration apart from mineralization of organic substance, nitrification, denitrification and biological phosphorus removal may occur.

At present, total sewage purification is sought based on removal not only of organic matter but also products of nitrification and phosphorus. Results of those processes in one reactor may be, however, in particular conditions, difficult to achieve. For example, extension of time of aeration in one working cycle of reactor is connected with shortening of anaerobic phase, decisive for the process of biological phosphorus removal and during which denitrification occurs. However, considerable extension of total time of anaerobic phase makes effectiveness of nitrification process difficult. That is why in some cases, sewage treated in SBR should be additionally purified, for example in filtration process, sorption, etc. Sewage drained from SBR, with a high concentration of ammonium nitrogen may be additionally purified in filtration process through zeolite bed, showing at the same time catalytic and sorptive qualities [1, 3, 4].

Zeolites are aluminosilicates consisting of aluminum and siliceous tetrahedrons (SiO_4, AlO_4), which give them electronegative character and high affinity to cations, including ammonium ions. Therefore, zeolite bed can support the nitrification process taking place in the reactor. The disadvantage of that method is a limited sorption capacity of zeolite and the necessity of exchanging or regeneration of the bed. Working cycle of zeolite can be extended in time by its modification, for example with manganese ions, forming a layer of MnO_2 on the surface and in micropores.

Manganese dioxide is amphoteric compound showing catalytic abilities and it reacts with acids and bases. The MnO_2 configuration is very complex – it forms various polymorphic modifications, which heavily diverge from stechiometrical formula, for example á-MnO_2 (including 91.5%) or ă-MnO_2 (with real configuration $MnO_{1.93}$). In its structure some ions of Mn^{4+} are replaced with ions of Mn^{2+} or some other, what gives the skeleton excess of negative charge. This charge can initiate redox reactions or build in some other positive ions inside the net structure. That is why the zeolite modified with manganese shows more catalytic abilities than natural zeolite [2]. Its additional advantage is also a possibility of regeneration with $KMnO_4$ solution, which simultaneously acts as a strong oxidant and disinfect for the filter bed, not allowing for development of microorganisms and secondary pollution as well as contamination of sewage. Afterwards regeneration solution may be used for sludge deactivation.

Using filter bed with zeolite bed modified with manganese ions in order to treat sewage from SBR may be an effective solution to remove excessive amounts of ammonia nitrogen, occurring in treated sewage with a short aeration cycle. Therefore, the purpose of this work was to test efficiency of system of treatment consisting of SBR and filter, filled with zeolite modified with MnO_2.

MATERIALS AND METHODS

Tests were carried out in two stages. In the first stage the process of additional purification of synthetic sewage disposed from laboratory reactor SBR was tested. In the second stage real sewage was additionally purified. Additional purification was based on sewage filtration through modified zeolite bed, occasionally subject to regeneration process.

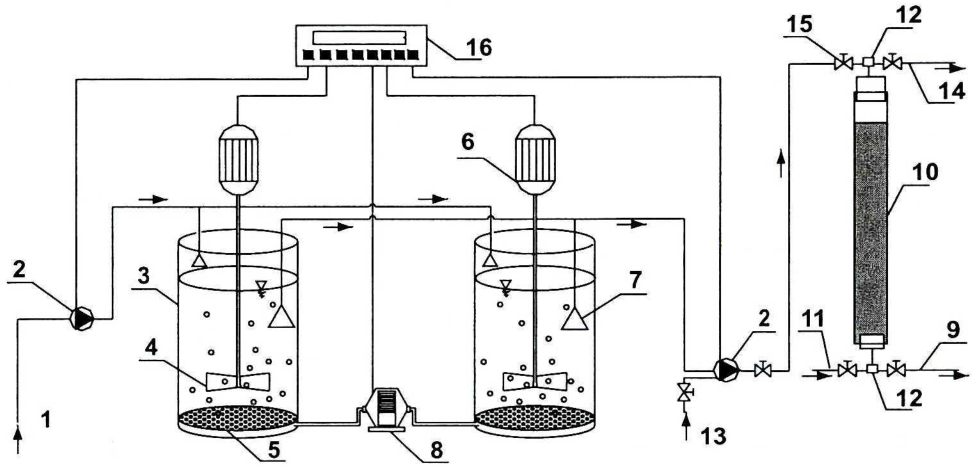


Fig. 1. Scheme of experimental laboratory-scale SBR system
1 – feeding of raw sewage, 2 – peristaltic pump, 3 – sequencing batch reactor, 4 – mixer, 5 – diffuser of air, 6 – engine, 7 – decanter, 8 – aerator, 9 – filtrate, 10 – filtering column, 11 – feeding of fluffing water, 12 – three-way valve, 13 – feeding of rinsing water and $KMnO_4$, solution, 14 – backwash water, 15 – valve, 16 – electronic time switch

The first stage designed technological system consisted of reactors type SBR and zeolite filter (Fig. 1). The amount of sewage disposed from reactors and subject to filtration was 50% of their total volume. Filtering column was made of organic glass with the height of 1.5 m and inside diameter 2.5 cm. Filtration speed was 4 m/h. The column was filled with zeolite modified with manganese. The height of bed equaled 70 cm, but the height of supportive layer was 30 cm. Graining of bed was between 0.5 to 1 mm. The filter bed was periodically fluffed with tap water and then concurrently regenerated with 0.3% of $KMnO_4$ solution with the speed of 4 m/h. Excessive regeneration agent was washed out with water flowing concurrently. Time of fluffing, regeneration and rinsing equaled 30 min. Sewage directed to SBR was obtained according to the norm PN-72/C-04550.09.

In the second stage real sewage was treated in a sewage – treatment plant "Jamno" in Koszalin (Tab. 7). Filtration was made in the same conditions as in the first stage. The bed was regenerated by two solutions: 3 or 6% solution H_2O_2 and 0.3% solution $KMnO_4$. In order to obtain higher concentrations of ammonium ions (from 6.12 to 8.76 mg/dm^3) in sewage being filtrated, appropriate exact amounts of ammonium salt were added.

Both the first and second stage of tests whereby subject to qualitative analysis was filtered sewage, directed to the filtration bed. The obtained results were the base for evaluation of efficiency and effectiveness of the operating of the technological system applied.

RESULTS AND DISCUSSION

I stage of tests

Tests were carried out in two series. In the first, sewage, including a little of phosphates < 1 mg P/dm^3 was additionally purified (Tab. 1). In the second series sewage, including phosphates amounting to 19 mg P/dm^3 was purified (Tab. 4). In both series of tests sewage disposed from reactors was filtrated by regenerated zeolite bed, maintaining this way regular condition of tests.

Table 1. Physical and chemical characteristics of sewage directed to filtering column in 1st series of tests

Parameter	Unit	Day				
		1	2	3	4	5
pH	–	7.82	7.74	7.86	7.85	7.88
Electrical conductivity	mS/cm	0.788	0.858	0.872	0.887	0.923
Ammonia nitrogen	mg/dm^3	34.42	36.88	33.74	36.26	35.27
Nitrate nitrogen	mg/dm^3	0.37	0.20	0.11	0.50	0.34
Orthophosphates	mg P/dm^3	0.72	0.52	0.38	0.42	0.37
COD	mg O_2 /dm^3	34	29	17	13	17

1st series of tests

Sewage directed to filter bed was characterized with higher amount of ammonia nitrogen (Tab. 1), whose concentration during 5 days of observation was between 33.74 to 36.88 mg/dm^3 and including small amount of phosphate (between 0.26 to 0.83 mg P/dm^3). Filtrating sewage through zeolite bed improved its quality. Filtrate reaction practically did not change, pH decreased average of 0.3–0.4 of the unit.

Table 2. Physical and chemical characteristics of filtrate obtained in 1st series of tests

Parameter	Unit	Day				
		1	2	3	4	5
pH	–	7.48	7.55	7.64	7.61	7.70
Electrical conductivity	mS/cm	0.761	0.799	0.837	0.849	0.897
Ammonia nitrogen	mg/dm^3	18.83	21.15	16.80	14.02	16.36
Nitrate nitrogen	mg/dm^3	0.92	0.58	0.52	1.54	0.72
Orthophosphates	mg P/dm^3	0.50	0.47	0.26	0.32	0.36
COD	mg O_2 /dm^3	19	15	13	11	11

A statistical characteristic such as average, minimum and maximal value and standard deviation was calculated for the results of tests obtained in 5 measuring tests (Tab. 3). Standard deviation value shows a big convergence of test results (max. scatter of values equals 10%) and great dispersion of variable value COD was obtained both for treated sewage (20–38%) and for untreated sewage (40–55%). Calculated average values of tested variables were presented graphically on one coordinate system for untreated and treated sewage (Fig. 2).

Table 3. Statistical analysis of repeatability of test results in 1st series carried out with the software Statistica 6; S – sewage directed to filter bed, TS – treated sewage – filtrate

Parameter	Unit	Mean	Minimum	Maximum	St. dev.	N
pH_S	pH	7.83000	7.74000	7.88000	0.054772	5
pH_{TS}	pH	7.59600	7.48000	7.70000	0.084439	5
Elec. $cond_S$	mS/cm	0.86560	0.78800	0.92300	0.049682	5
Elec. $cond_{TS}$	mS/cm	0.82860	0.76100	0.89700	0.051505	5
$N-NH_4$ S	mg/dm^3	35.31400	33.74000	36.88000	1.287043	5
$N-NH_4$ TS	mg/dm^3	17.43200	14.02000	21.15000	2.691109	5
$N-NO_{3S}$	mg/dm^3	0.30400	0.11000	0.50000	0.152086	5
$N-NO_{3TS}$	mg/dm^3	0.86840	0.52000	1.54000	0.407962	5
PO_{4S}	mg P/dm^3	0.47200	0.37000	0.72000	0.144000	5
PO_{4TS}	mg P/dm^3	0.39200	0.26000	0.52000	0.113666	5
COD_S	mg O_2/dm^3	22.00000	13.00000	34.00000	9.000000	5
COD_{TS}	mg O_2/dm^3	13.80000	11.00000	19.00000	3.346640	5

The analysis of test results shows clear effect of additional sewage purification on modified filter bed. Electrical conductivity decreased by average 0.037 mS/cm and also, to some degree, the values of COD decreased by 8.2 mg O_2/dm^3 and concentration of orthophosphates increased by 0.08 mg P/dm^3. There was however a visible change in concentration of ammonia nitrogen which decreased from 35.31 to 17.43 mg/dm^3 (Fig. 2a), which means on average by 17.88 mg/dm^3. At the same time an increase of nitrate nitrogen concentration was noticed (Fig. 2b) to 0.87 mg/dm^3, which means an average of 0.57 mg/dm^3.

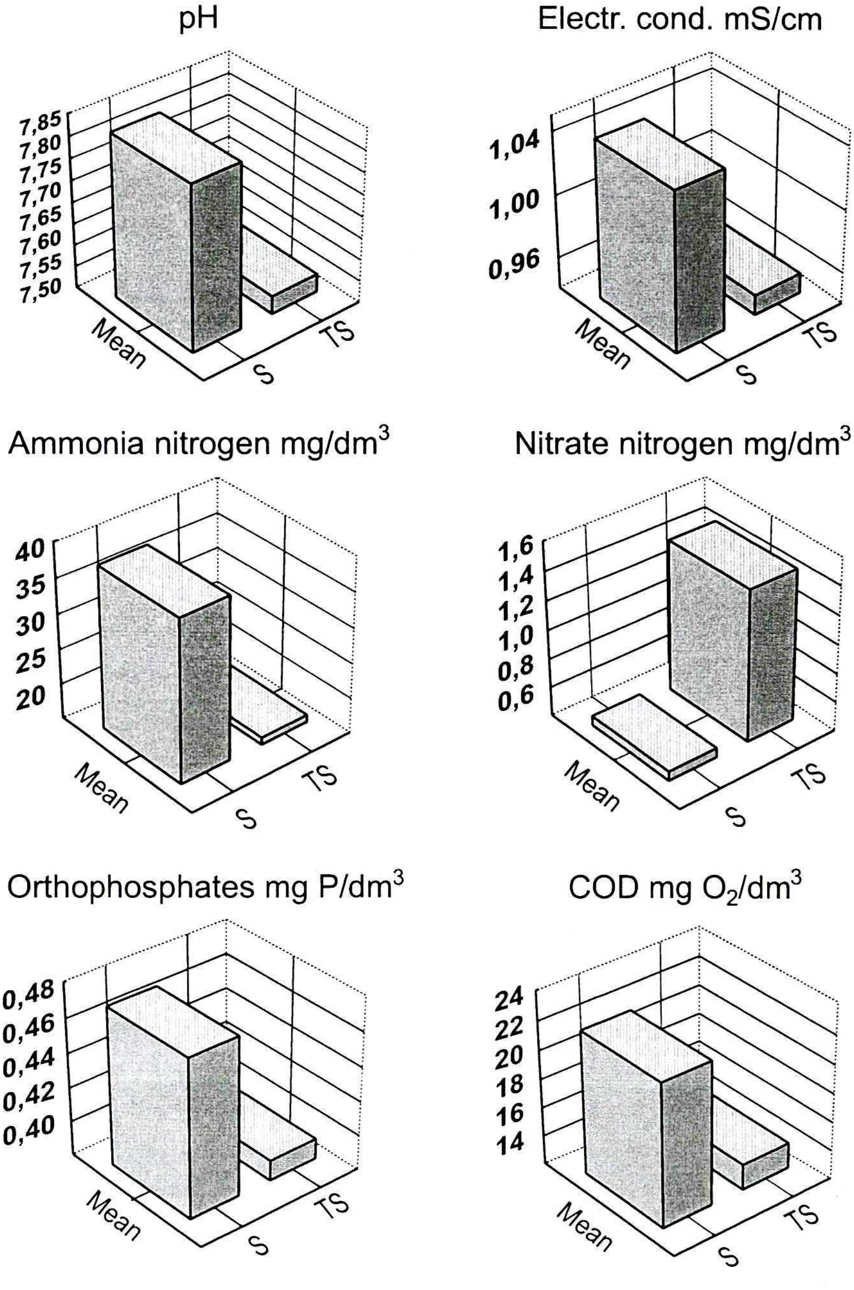


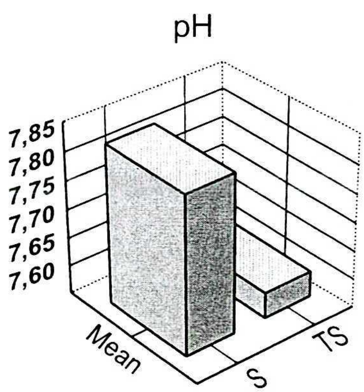
Fig. 2. Cumulative statement of results of sewage treatment in 1st series of tests on modified zeolite bed
S – sewage directed to filter bed, TS – treated sewage – filtrate

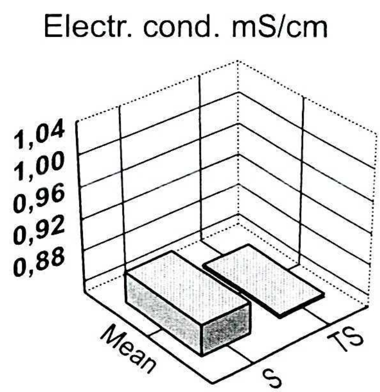
2nd series of tests

In the second series of tests the sewage disposed from SBR was characterized with phosphates concentration between 16.15 to 21.90 mg P/dm^3. Concentration of nitrate nitrogen was between 36.50 to 44.06 mg N/dm^3. Physical and chemical characteristic of filtered sewage was shown in Table 4 and characteristic of purified sewage in Table 5.

Table 4. Physical and chemical characteristics of sewage directed to filter column in 2nd series of tests

		Day				
Parameter	Unit	1	2	3	4	5
pH	–	8.04	7.86	7.78	7.8	7.84
Electrical conductivity	mS/cm	1.063	1.032	1.013	1.062	1.036
Ammonia nitrogen	mg/dm^3	44.06	34.62	36.50	40.38	35.09
Nitrate nitrogen	mg/dm^3	0.46	0.47	0.45	0.43	0.52
Orthophosphates	mg P/dm^3	16.15	16.39	19.21	20.72	21.90
TOC	mg/dm^3	9.10	10.10	11.96	10.80	8.87
IC	mg/dm^3	61.12	59.07	54.83	55.40	58.95
TC	mg/dm^3	70.23	69.17	66.79	66.20	67.82





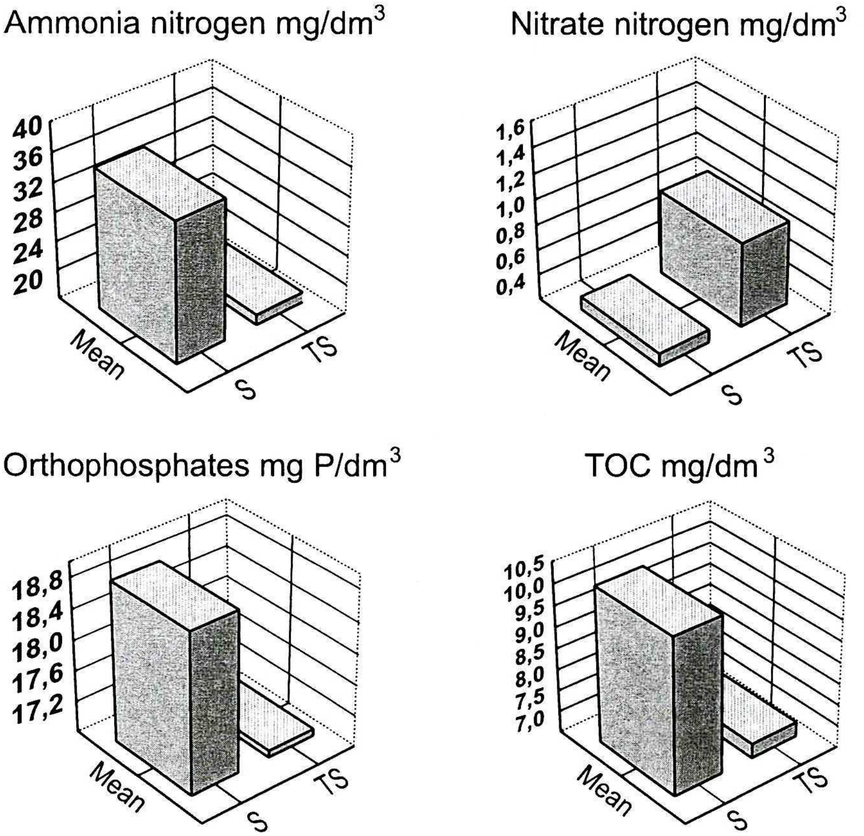


Fig. 3. Cumulative statement of results of sewage treatment in 2nd series of tests on modified zeolite bed
S – sewage directed to filter bed, TS – treated sewage – filtrate

Statistical analysis and analysis of values statistically calculated for measuring number 5 was shown in the Table 6. Statistical analysis shows the convergence of obtained results, standard deviation assumes the values within 0.7% to 14% of average values of tested variables. Average values of tested indexes for five measuring tests were shown on Figure 3. Results analysis show that in filtration process through the filter bed a decrease of practically all values from tested indexes occurred and only concentration of nitrate nitrogen increased from 0.47 to 1.45 mg/dm^3. The amount of nitrate nitrogen decreased from 38.13 to 16.05 mg/dm^3, on average 22.08 mg/dm^3, and so the effectiveness of zeolite bed in removal of this ion is significant and analogous to the values received in the first series of tests.

II stage of tests

Revision of results shown in Figs. 2 and 3 was carried out on real sewage (Tab. 7). Filtered sewage had neutral reaction, between pH 6.87 and 7.46. Average conductivity amounted to 0.973 mS/cm. Concentration of ammonia nitrogen was from 6.12 to 8.76 mgN/dm^3, but concentration of nitrate nitrogen between 5.28 and 14.80 mg N/dm^3. General organic carbon was on the level of 8 mg/dm^3. First stage of tests showed significant efficiency of filter bed

Table 5. Physical and chemical characteristics of filtrate received in 2[nd] series of tests

		Day				
Parameter	Unit	1	2	3	4	5
pH	–	7.35	7.47	7.54	7.50	7.60
Electrical conductivity	mS/cm	0.949	0.944	0.932	0.953	0.986
Ammonia nitrogen	mg/dm^3	17.42	12.52	18.23	16.79	15.31
Nitrate nitrogen	mg/dm^3	1.45	1.55	1.29	1.62	1.35
Orthophosphates	$mg\ P/dm^3$	14 .86	14.68	16.91	18.21	19.80
TOC	mg/dm^3	6.38	6.394	6.60	7.15	7.70
IC	mg/dm^3	63.46	63.01	59.90	59.67	64.47
TC	mg/dm^3	57.07	56.62	53.30	52.52	56.77

Table 6. Statistical analysis of results repeatability in 2[sd] series of tests, carried out with software Statistica 6; S – sewage directed to filter bed, TS – treated sewage – filtrate

	Unit	Mean	Minimum	Maximum	St. dev.	N
pH_S	pH	7.83000	7.74000	7.88000	0.054772	5
pH_{TS}	pH	7.49200	7.35000	7.60000	0.093113	5
Electr. $cond_S$	mS/cm	1.04120	1.01300	1.06300	0.021300	5
Electr. $cond_{TS}$	mS/cm	0.95280	0.93200	0.98600	0.020167	5
$N\text{-}NH_{4\,S}$	mg/dm^3	38.13000	34.62000	44.06000	4.013913	5
$N\text{-}NH_{4\,TS}$	mg/dm^3	16.05400	12.52000	18.23000	2.246404	5
$N\text{-}NO_{3\,S}$	mg/dm^3	0.46600	0. 43000	0.52000	0.033615	5
$N\text{-}NO_{3\,TS}$	mg/dm^3	1.44800	1.29000	1.62000	0.136821	5
$PO_{4\,S}$	$mg\ P/dm^3$	18.87400	16.15000	21.90000	2.562602	5
$PO_{4\,TS}$	$mg\ P/dm^3$	16.89200	14.68000	19.80000	2.191796	5
TC_S	mg/dm^3	10.16600	8.87000	11.96000	1.268929	5
TC_{TS}	mg/dm^3	6.84400	6.38000	7.70000	0.571690	5
IC_S	mg/dm^3	57.87400	54.83000	61.12000	2.669800	5
IC_{TS}	mg/dm^3	62.10200	59.67000	64.47000	2.181713	5

in removal of ammonia ions, which is why revision of tests on real sewage related to the change of values for only the one unit.

Table 7. Physical and chemical characteristics of sewage additionally treated on filter bed

Parameter	Unit	minimum	maximum	average
pH	–	6.87	7.46	7.23
Electrical conductivity	μS/cm	835	1091	973.90
Ammonia nitrogen	mg/dm^3	6.12	8.76	7.38
Nitrate nitrogen	mg/dm^3	5.28	14.80	9.46
Nitrite nitrogen	mg/dm^3	0.036	0.78	0.30
Orthophosphates	mg P/dm^3	0.62	7.28	2.95
TOC	mg/dm^3	7.25	10.05	8.34

For the received empirical dependencies percentage calculation for removal of ammonia nitrogen and effectiveness of technological system (h) were measured. Tests and calculation results were presented in Figures 4–6. During tests, in order to remove the organic substance totally, filter bed was regenerated with two oxidizing solutions. Solution H_2O_2 was used in two concentrations (3 and 6%).

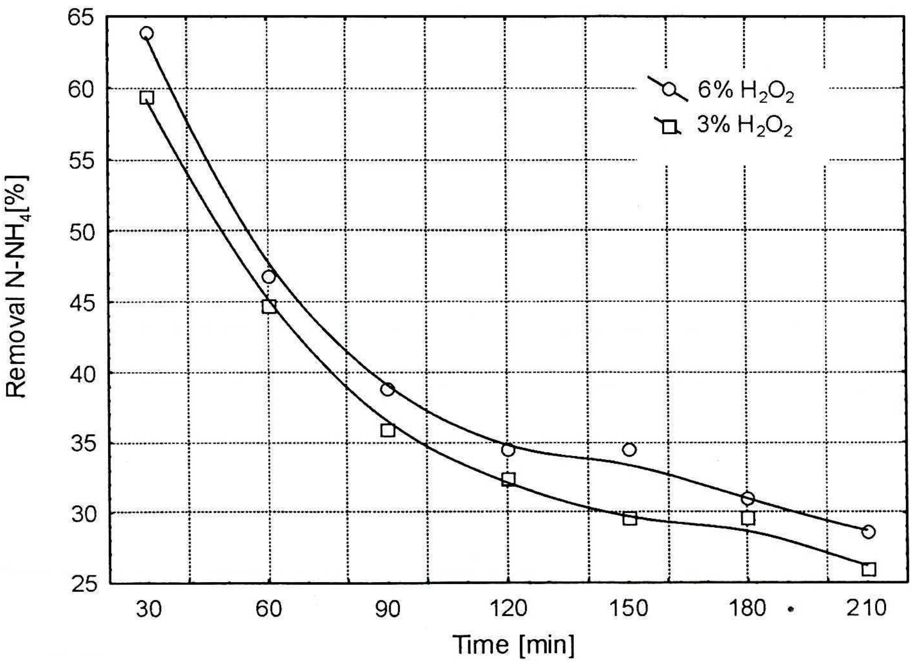


Fig. 4. Kinetics of removal of ammonia ions on filter bed; removal of nitrate nitrogen on zeolite column, depending on concentration of regeneration solution

Regeneration of bed was made after finishing every series of tests. Received results show high effectiveness of the filter bed in municipal wastes treatment. During early minutes the time of the process of removal of ammonia ions amounted to 60–65%. Over time, absorptive and catalytic capacity of filtration bed decreased and effectiveness of sewage treatment also decreased. After 180 minutes of operation, effectiveness of system decreased to 30%.

Effectiveness of filtration system changed similarly (Fig. 5 and Tab. 8) calculated from the dependency:

$$\eta_t = \frac{(C_o - C_k)V_r}{tV_z} = 28 - 13.82\log(t), [\mathrm{g/h\cdot m^3}] \tag{1}$$

from which the concentration of ammonia ions in filtrate received in time operation of column operation and treated sewage volume can be measured:

$$C_k = C_o - \frac{(28 - 13.82\log t)tV_z}{V_r}, [\mathrm{g/m^3}] \tag{2}$$

with the assumption that filtration is carried out at the speed of 4 m/h and the concentration of ammonia ions in filtered sewage is about 6–8.5 mg/dm^3. For the equation (1) in order to define its convergence with empirical data, standard deviations were measured:

$$\pm L = t_\alpha \sqrt{\frac{nS^2}{n(n-1)}} = t_\alpha \sqrt{\frac{665.894}{306}} = t_\alpha 0.084 = 0.177 \tag{3}$$

Regeneration of the filter bed allows for its repeated use in the process of sewage treatment. The tests show that 3% concentration of solution H_2O_2 is sufficient for bed regeneration. 6% concentration of solution in little measurements increases the sorptive capacity (Fig. 4–5).

Test results show that in the filtration process through modified filtration bed removal of ammonia ions on high level is achieved and effectiveness of the process with assumed permanent initial concentration of ammonia ions depends on the volume of filtered sewage. With the increasing volume of sewage the sorptive and kinetic capacities of the bed as well as of the whole process decrease:

$$C_0 - C_k = 2.5 + 6\exp(-0.25Vr / Vz), [\mathrm{g/m^3}] \tag{4}$$

Over time the effectiveness of bed operation is decreases from 31.26 to 5.0 g/(h·m^3). On the basis of graphic dependency effective work time equals 24 hours. It may be assumed that with the volume of bed 1 m^3 and speed of filtration 4 m/h it is possible to purify thoroughly 142 m^3 of sewage. At this point the bed should be regenerated. The amount of ions removed in the function of filtrate volume (with fixed initial concentration of ammonia ions C_o = 6–8 mg/dm^3) is compatible to the volume specified by functional curve and

Table 8. Effectiveness of filter bed from measurement and equalization; V_r–volume solution, V_z – volume zeolite

Filtration time, h	C_0 - C_K [g/m³]	V_r/V_z	η_t from measurement [g/h·m³]	η_t from equation [g/h·m³]	$\Delta\eta_t$ [g/h m³]	$\Delta\eta_t^2$
3% H_2O_2 regeneration						
0.5	5.20	2.97	30.89	32.16	1.27	1.613
1.0	3.92	5.94	23.29	28.00	4.71	22.184
1.5	3.12	8.91	18.53	25.57	7.03	49.420
2.0	2.84	11.88	16.87	23.84	6.97	48.580
2.5	2.6	14.85	15.45	22.50	7.06	49.844
3.0	2.60	17.82	15.45	21.4 1	5.96	35.522
7.0	2.28	20.79	13.54	20.48	6.94	48.164
24.0	2.00	142.57	11.88	8.93	2.95	8.703
48	1.12	285.15	6.65	4.77	1.88	3.534
6% H_2O_2 regeneration						
0.5	5.26	2.97	31.25	32.16	0.91	0.828
1.0	3.86	5.94	22.93	28.00	5.07	25.705
1.5	3.20	8.91	19.01	25.57	6.56	43.034
2.0	2.84	11.88	16.87	23.84	6.97	48.581
2.5	2.84	14.85	16.87	22.50	5.63	31.697
3.0	2.56	17.82	15.21	21.41	6.20	38.44
7.0	2.36	20.79	14.02	20.48	6.46	41.731
24	1.80	142.57	10.69	23.84	13.15	172.923
48	0.95	285.15	5.64	4.77	0.87	0.828

volume of solution and so after and by function integral dependency equation shall be received from which we can calculate efficiency of the configuration with the given amount of sewage to be treated.

$$\sum_{0}^{Vr/Vz} C_0 - C_k = \int_0^{Vr/Vz} [2.5 + 6\exp(-0.25x)]dx = 2.5\int_0^{Vr/Vz} dx + 6\int_0^{Vr/Vz} \exp(-0.25x)dx =$$

$$2.5x - 24\exp(-0.25x)\Big|_0^{Vr/Vz} = 2.5Vr/Vz - 24e^{-0.25Vr/Vz} + 24 \quad (5)$$

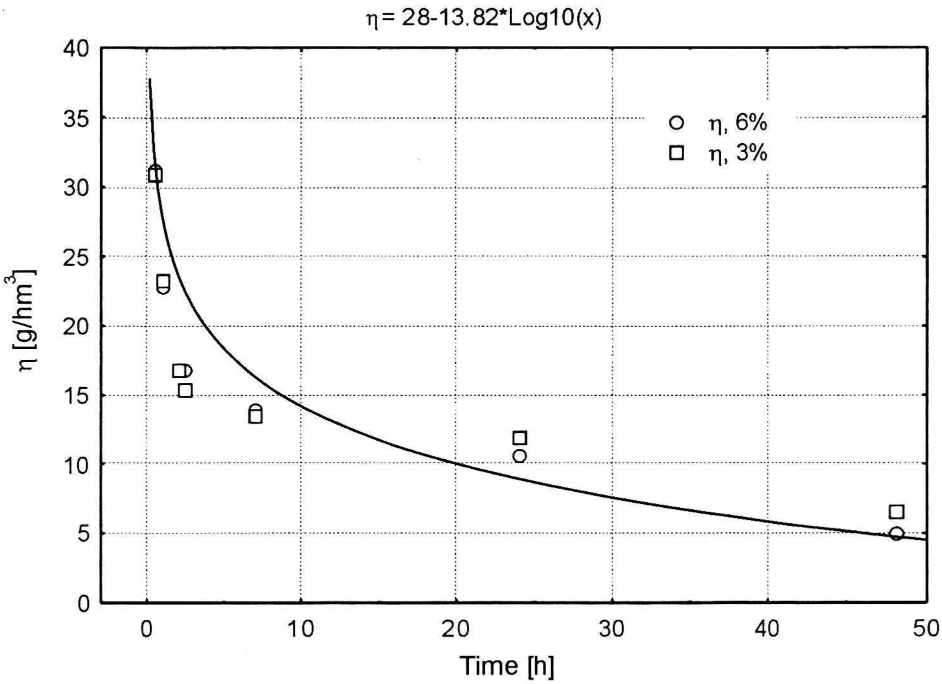


Fig. 5. Time influence of filter bed on its effectiveness in removal of ammonia ions from sewage calculating into volume of bed unit

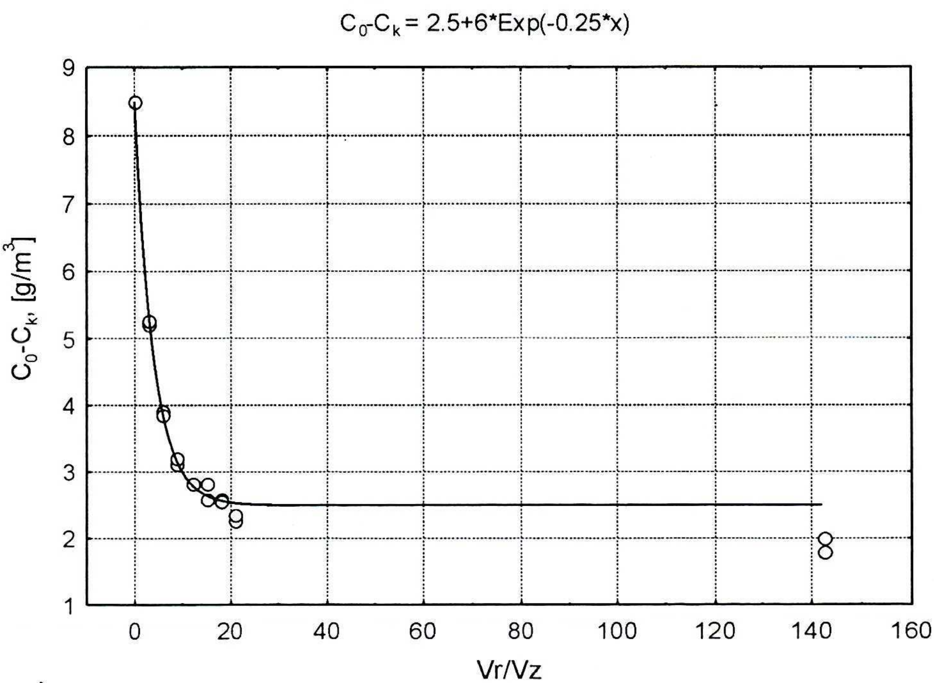


Fig. 6. Number of ammonia ions removed in function of volume solution with specified fixed initial concentration C_0 = 8.5 g/m³; V_r – volume solution, V_z – volume zeolit

For example, filtering 140 m^3 of sewage with initial concentration of 8 mg/dm^3, 374 g N-NH_4 is removed on zeolite bed with the volume of 1 m^3 and during filtration of 30 m^3 removal of ammonia ions amounts to 99 g N-NH_4. While calculating the amount of removed ammonia ions in the equation (5) decrease of adsorption capacity with the time of bed operation is taken into consideration.

CONCLUSIONS

The proposed method for additional sewage in the filtration process on a bed of modified zeolite is effective. The bed may be periodically regenerated with 3 or 6% of H_2O_2 solution and 0.3% $KMnO_4$ solution. By applying higher solution concentration more exact purification of filter bed is obtained but then exploitation costs are higher. The process may be described with simple mathematical dependencies.

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