

APPLICATION OF SELECTIVE AND POROUS MATERIALS FOR THE REMOVAL OF BIOGENIC COMPOUNDS AND INDICATOR BACTERIA FROM DOMESTIC WASTEWATER

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ABSTRACT

In the paper we present the evaluation of the effectiveness of the removal of biogenic compounds and indicator bacteria from domestic sewage in a vertical flow filter with a variety of fillings. The research was carried out on expanded clay, zeolite, calcined siliceous limestone, and crushed PET flakes. Based on the study of raw and treated wastewater, the effectiveness of removal of ammonium nitrogen, orthophosphates, *Escherichia coli*, and coliform bacteria was determined according to the selectivity, porosity and grain size of the fill. In the case of removing biogenic compounds, the most effective fillers were found to be of natural porous selective materials, such as zeolite and calcined siliceous limestone. They produced a very high removal rate of ammonium and orthophosphate, amounting to 75.34%, and over 79%, respectively. It has been demonstrated that the filter that was filled with natural porous material of fine particle size (the zeolite) was the most suitable for the removal of indicator bacteria. A very high degree of the *Escherichia coli* (99.98%) and coliform bacteria (99.94%) reduction was obtained.

Keywords: wastewater treatment, biogenic compounds, *Escherichia coli*, coliform bacteria, effectiveness, vertical flow filter

INTRODUCTION

The regulations remaining in force in Poland, specifying the permissible concentration of biogenic compounds (total nitrogen and total phosphorus) or the minimum percentage of their reduction for domestic or municipal sewage introduced into waters or into the ground, refer to the aforementioned values for treatment plants above 2000 PE [Rozporządzenie... 2014]. In the case of household treatment plants, included in the group of up to 2000 PE, the required level of biogenic indicators reduction has not been determined, and the highest allowable concentrations concern only the facilities which discharge sewage to lakes and their tributaries, and directly to artificial reservoirs located

on flowing waters [Rozporządzenie... 2014]. This results in an increased amount of eutrophic compounds, i.e. nitrogen and phosphorus, flowing into reservoirs and watercourses.

The effluent (treated sewage), which has not been subjected to disinfection, is also a source of microbiological contaminants, including pathogenic bacteria, which cause deterioration of sanitary condition when discharged into the environment. This problem concerns both the wastewater discharged from municipal wastewater treatment plants, and from household wastewater treatment plants [Michałkiewicz et al. 2011, Olańczuk-Neyman et al. 2012, Olańczuk-Neyman and Quant 2015]. The presence of pathogenic bacteria in the aquatic environment (*Escherichia*

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coli, *Klebsiella sp.*, *Enterobacter sp.*, *Citrobacter sp.* and *Proteus sp.*) is a sign of fresh pollution by secretions and faeces. At the same time, it may indicate the presence of more dangerous pathogenic bacteria, such as for instance *Salmonella sp.* or *Shigella sp.* [Olańczuk-Neyman et al. 2012, Olańczuk-Neyman and Quant 2015].

Literature reports indicate that the effectiveness of elimination of pathogenic bacteria in household systems is insufficient [Budzińska et al. 2007, Józwiakowski 2012]. The application of new types of filtration materials, often leading to increased efficiency in removing physical and chemical contaminants, may prove ineffective in the elimination of pathogenic bacteria. In view of their reduction in household wastewater treatment plants, the final purification step, responsible for safeguarding against bacteriological contamination, must be taken into account. Langenbach et al. [2009, 2010] demonstrated the suitability of a sand filter, used as the third stage of wastewater treatment for the removal of faecal bacteria.

Another solution may be in the septic tank system/vertical sand filters, exhibiting a high degree of removal of physicochemical [Chmielowski and Ślizowski 2008a, Chmielowski and Ślizowski 2008b] and bacteriological pollutants. As a result of using sand, it is possible to obtain purified sewage containing $1 \cdot 10^2 - 2 \cdot 10^4$ CFU per 100 ml of *Escherichia coli* bacteria and $5 \cdot 10^3 - 3 \cdot 10^5$ CFU per 100 ml of coliform bacteria [Chmielowski 2013, Wąsik and Chmielowski 2017a, Wąsik and Chmielowski 2017b]. Bellamy et al. [1995] found that the phenomenon leading to the removal of bacteria in the slow sand filter was their adsorption on the surface of the biofilm, adhering to the grains of the filling. According to Jenkins et al. [2011], grain size is the main factor affecting the performance of sand filters. Wąsik and Chmielowski [2017b] demonstrated that the degree of elimination of indicator bacteria was influenced most by the size of the filling particles, not the percentage content of the filling, or its type.

Elliott et al. [2015] report that the most likely mechanism for removing *Escherichia coli* is through the *schmutzdecke* or biofilm layer, and the intensity of this process increases with the depth of the deposit. Stevik [2004] and Langenbach [2010] confirmed that straining is responsible for the fixing of pathogenic

bacteria on the porous material, while the factor regulating the transport of bacteria through the filter is the size of grains of its filling. In porous media, where a pore diameter is larger than bacteria, adsorption is the main mechanism of bacterial retention [Stevik 2004, Langenbach 2010]. Selective materials possessing a positive charge can attract Gram-negative *Escherichia coli* bacteria [Lukasik et al. 1993, Truesdail et al. 1998], however, this phenomenon can be inhibited by organic substances dissolved in the sewage, which block the surface of the material [Foppen et al. 2008].

The aim of the present work is to evaluate the effectiveness of vertical flow filters in terms of removing selected forms of nitrogen and phosphorus as well as indicator bacteria from domestic wastewater. The assessment was carried out for different materials, varying in terms of their selective properties, porosity, and granulation.

RESEARCH MATERIAL AND METHODS

The study was carried out on a semi-technical scale, in vertical flow filtration columns, made of PVC, and filled with various materials. The flow through the filter bed was from top to bottom, and a detailed description of the model columns was described in previous works [Wąsik and Chmielowski 2017a, Wąsik and Chmielowski 2017b, Wąsik et al. 2017].

One artificial (S) material (crushed PET flakes), and three natural (N) materials were used in the study. Two of them possessed selective properties (zeolite, and calcined siliceous limestone), and one possessed non-selective properties (leca, or expanding clay). The materials were grouped according to their porosity (P – porous, NP – nonporous), selectivity (S – selective, NS – non-selective), and granulation: coarse (> 4.0 mm), medium (2.5–5.0 mm), and fine (1.0–2.5 mm) [Wąsik and Chmielowski 2017a, Wąsik and Chmielowski 2017b, Wąsik et al. 2017].

Each of the filter beds was dosed with the same amount of pre-treated household sewage, similar in terms of chemical and bacteriological composition, and metered in a periodic manner (24 times a day). Prior to the commencement of the study, the deposits were operated under changing hydraulic conditions for a period of several months for their development. The

tests were carried out over a period of several months, taking samples at a frequency of 2–4 weeks.

In order to determine the effectiveness of biogenic compounds removal, in raw sewage (past the initial settling tank) and in purified sewage, the content of ammonium nitrogen was determined by direct Nesslerisation (PN-ISO 5664: 2002), while the content of orthophosphates was determined using the ammonium molybdate method (PN-EN ISO 6878: 2006).

Bacteriological analyses included the determination of the number of indicator bacteria, i.e. *Escherichia coli* and coliform bacteria. The number of CFUs (colony forming units) in the effluents flowing into the filters was determined by surface culturing, after previous preparation of the appropriate dilutions. For quantitative analysis in treated wastewater, the membrane filtration method was additionally used, applying cellulose nitrate filters with 0.45 µm pore size (of the Sartorius brand). In both cases, the BioMérieux Endo media was used. The results were given in CFU per 100 ml.

The obtained results were subjected to statistical analysis with the aid of the STATISTICA 12 software. The characteristic values of biogenic and bacteriological pollutants, including the arithmetic mean, minimum and maximum values, and standard deviation, were determined for the tested materials.

Analysis of the removal of biogenic compounds and pathogenic bacteria – in respect to the selectivity, porosity, and grain size of the material – primarily included the grouping of source data using cluster analysis. A simple hierarchical method was applied for this purpose, consisting in determining agglomerates by linking the smaller clustering algorithm created in the previous steps (StatSoft 2017). The Ward method was adopted as the agglomeration method, and the Euclidean distance was adopted as the distance measure.

The last step was to analyse the variance of the source data, that is, the degree of removal of ammonium nitrogen, orthophosphate, *Escherichia coli*, and coliform bacteria. The normality of the variable distribution was verified using the Shapiro-Wilk test. The homogeneity analysis of variance, applying the Levene test, was used to check the significant differences between the variances of the removed impurities. In order to demonstrate between which groups rank differences are the most significant, the non-parametric Kruskal-Wallis test was performed.

RESEARCH RESULTS AND ANALYSIS

The qualitative characteristics of pre-treated sewage flowing into filtration beds are presented in Table 1.

Table 1. Quality characteristics of pre-treated sewage inflowing to model columns

Parameter	Unit	Descriptive statistics			
		Mean value	Minimum value	Maximum value	Standard deviation
Ammonia nitrogen	mgN-NH ₄ ⁺ · dm ⁻³	105.12	42.825	197.95	32.90
Orthophosphates	mgPO ₄ ³⁻ · dm ⁻³	38.59	24.485	55.15	9.73
<i>Escherichia coli</i>	jtk (units) · 100 ml ⁻¹	1.51 · 10 ⁷	2.55 · 10 ⁵	1.51 · 10 ⁸	2.87 · 10 ⁷
/ Coliform bacteria	jtk (units) · 100 ml ⁻¹	9.36 · 10 ⁷	8.5 · 10 ⁵	5.74 · 10 ⁸	1.28 · 10 ⁸

On the basis of statistical analysis, it can be concluded that biogenic indicators in the sewage inflowing to individual filtration columns remained at a level similar to that reported in literature [Kaczor and Bugajski 2006, Kaczor 2009, Dębska et al. 2015, Obarska-Pempkowiak and others. 2015]. Also, the number of pathogenic bacteria in pre-treated sewage was similar to the values reported by other authors [Budzińska et al. 2007, Józwiakowski 2012].

Hierarchical cluster analysis made it possible to isolate the main clusters of the source data (see Figure 1). The first group of agglomerations included the degree of removal of biogenic compounds. The second group formed two smaller clusters, defining the type of filter material used, and the degree of removal of pathogenic bacteria. The binding distance was estimated at 1000.

The effectiveness of removing biogenic compounds and pathogenic bacteria in the tested filter

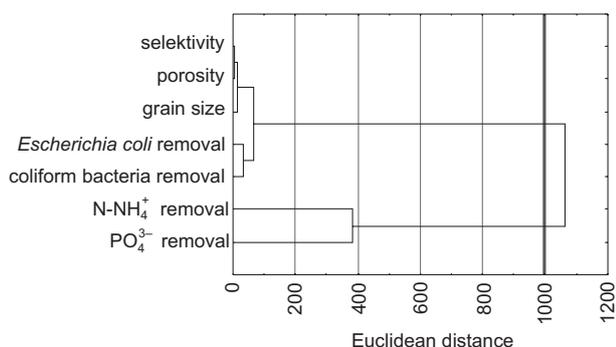


Fig. 1. Cluster dendrogram, Ward hierarchical clustering method

beds, depending on the type of material used, has been shown in box-plot charts (see Figures 2–7). The analysis thereof has shown that the higher efficiency of biogenic compounds removal was typical for the deposits, in which a porous material was used as filling (see Figure 2). High ammonia nitrogen removal (73.31% on average) and orthophosphates removal (on average 62.93%) were obtained. Kalenik and Wnacerz [2013] also observed higher efficiency of natural porous (chalcedonite) deposits compared to the non-porous (sand) deposits in terms of biogenic compounds removal (total nitrogen and total phosphorus) from synthetic sewage. According to the authors, the high porosity of chalcedonite translated into higher mass capacity, and it allowed the development of nitrifying bacteria and sorption of phosphorus compounds.

Kalenik [2014] noted the large internal dispersion and effective adsorption of phosphorus compounds in the case of another natural porous material, namely, clinoptilolite.

The degree of stoppage of pathogenic bacteria also depended on the porosity of the filling, while the mechanism of the process was mainly based on adsorption. On the porous deposit, the retention efficiency of *Escherichia coli* was on average 99.65%, and for the coliform bacteria group, the corresponding figure amounted to 99.81% (see Figure 3).

When analysing the removal of biogenic compounds in reservoirs filled with materials of varying selectivity, it has been observed that selective beds displayed a markedly higher efficiency (see Figure 4). The average efficiency of ammonium nitrogen removal in this case amounted to 74.58%, and of orthophosphate removal, 68.92%. Both for the non-selective natural (ZNSN) and the artificial (ZNSS) deposits, a similar, over 60% $N-NH_4^+$ removal efficiency was found, and in the case of PO_4^{3-} , the corresponding figure was approx. 30%. The type of material had a greater impact on the removal of bacteriological contaminants (see Figure 5). Compared to the artificial material (ZNSS), the natural fillings (ZNSN and ZSN) arrested the *Escherichia coli* bacteria to a greater extent (99.04% and 99.98% on average); it was also the case for the coliform bacteria (in both cases, 99.85% on average). Selective media, due to their positive charge, were able to retain Gram-negative bacteria based on the principle of

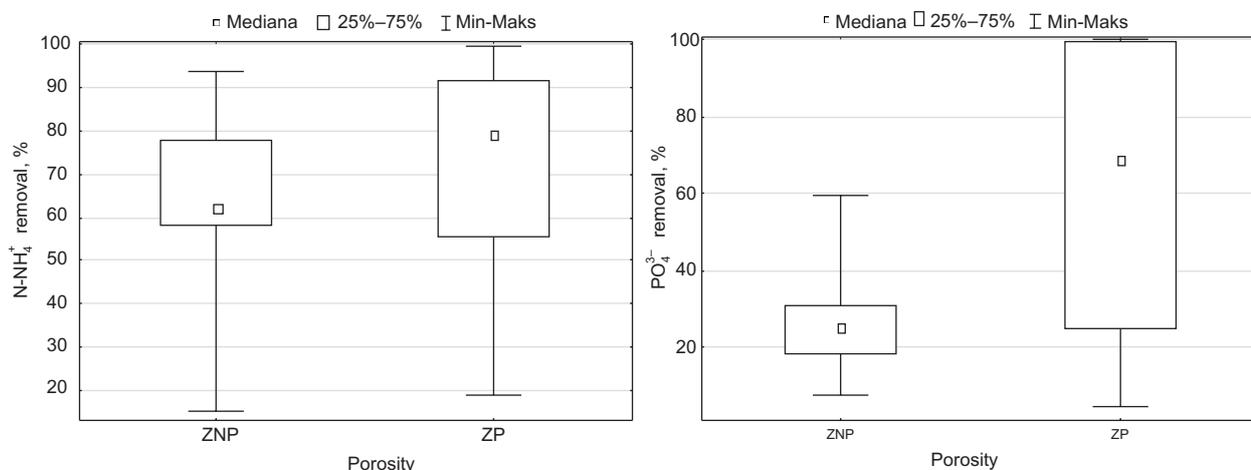


Fig. 2. Box-plot graphs of biogenic compounds removal on beds filled with porous (ZP) and non-porous material (ZNP)

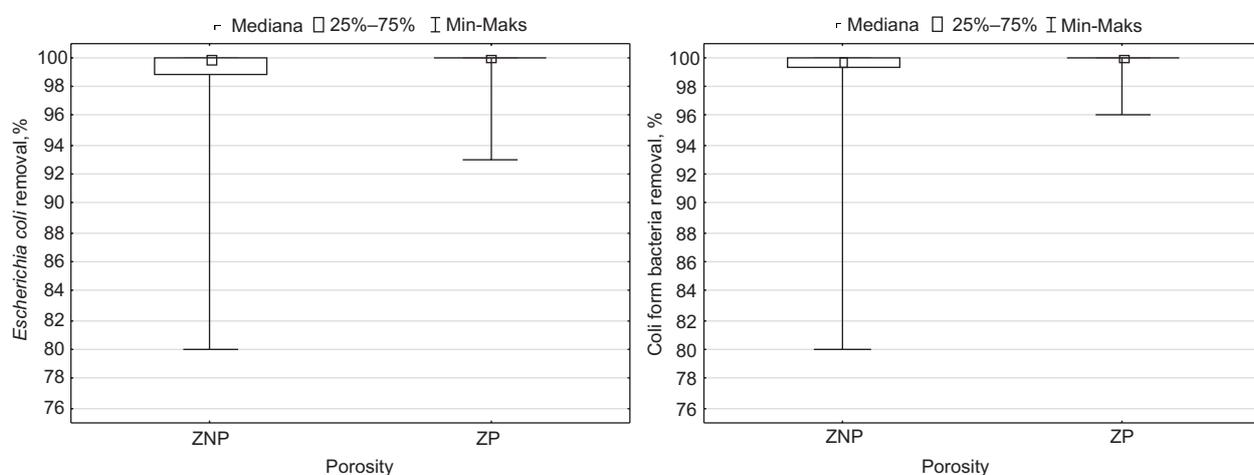


Fig. 3. Box-plot graphs of pathogenic bacteria removal on beds filled with porous (ZP) and non-porous material (ZNP)

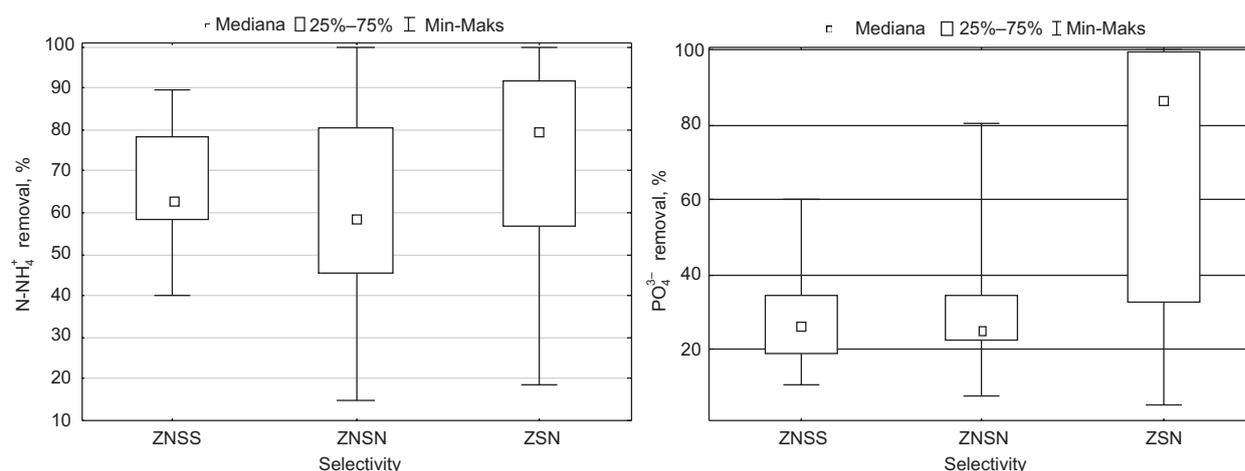


Fig. 4. Box-plot graphs of biogenic compounds removal on beds filled with non-selective plastic (ZNSS) and natural (ZNSN) material or selective natural material (ZSN)

electrostatic attraction, which influenced the highest reduction in pathogenic bacteria among the examined materials.

Considering the granulation of the tested filtration materials, the highest level of N-NH₄⁺ removal and pathogenic bacteria was found on the filling with fine grain size, namely between 1–2.5 mm (see Figures 6, 7). In the case of ammonium ions, the corresponding figure was 75.34% on average; for *Escherichia coli* bacteria, it was 99.96%; and for coliform bacteria, 99.94%. The process of removing ammonium nitrogen was based mainly on the production of biofilm on

the grains of the filter material, while bacterial contamination was reduced based on strain and adsorption mechanisms.

In the studies by Chmielowski and Ślizowski [2008a, 2008b], conducted on the natural non-porous bed, the increase in the replacement grain diameter of the filter bed (above 1.65 mm) was the reason for the decrease in the efficiency of wastewater treatment. At the same time, the bed filled with siliceous limestone material provided efficient elimination of PO₄³⁻ ions, on average at the level of 79.01% (see Figure 6). This means that the removal of orthophosphates was due to

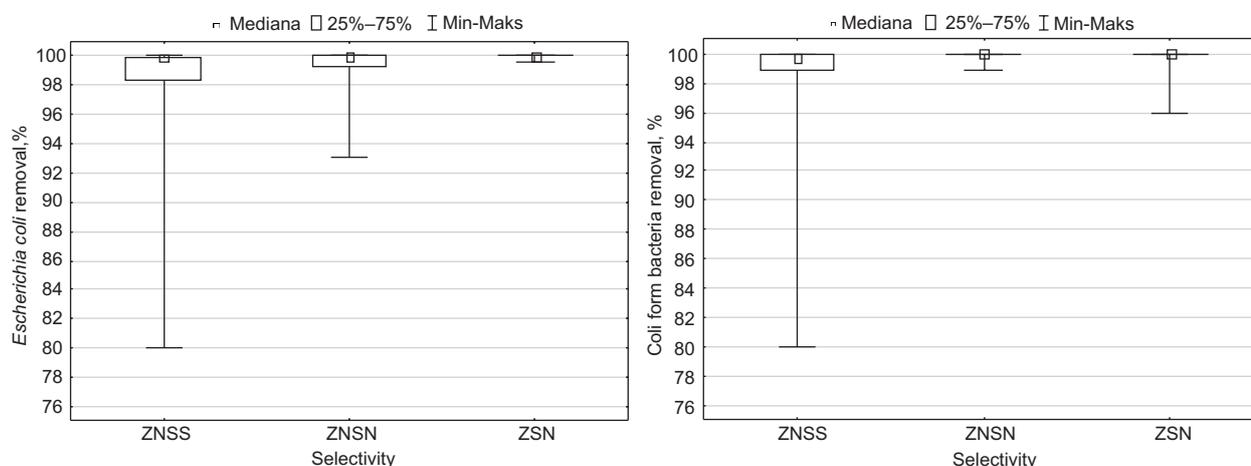


Fig. 5. Box-plot graphs of pathogenic bacteria removal on beds filled with non-selective plastic (ZNSS) and natural (ZNSN) material or selective natural material (ZSN)

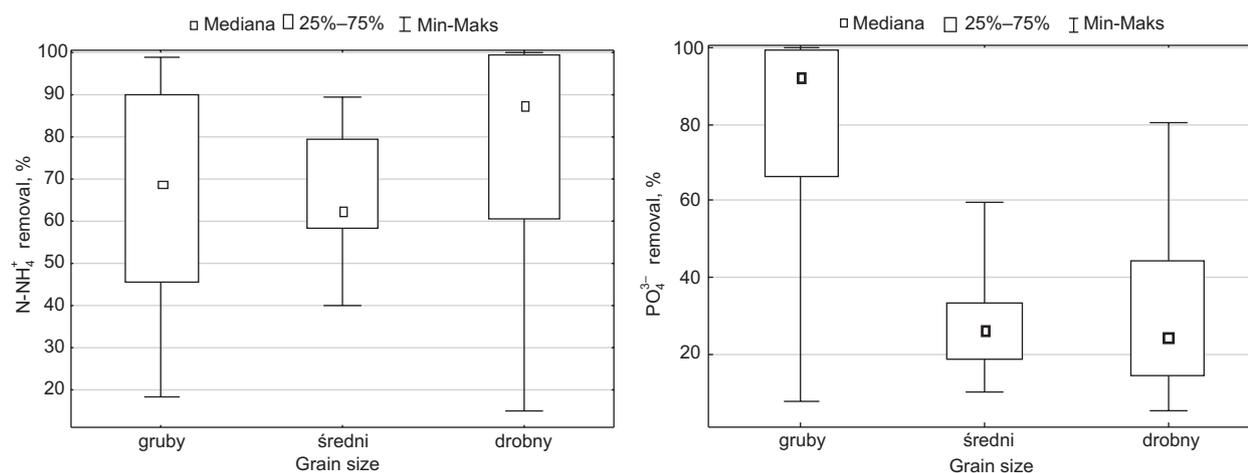


Fig. 6. Box-plot graphs of biogenic compounds removal on beds filled with coarse, medium and fine material

a mechanism other than the one that is typical for bio-filters, such as chemical reactions [Wąsik et al. 2017].

On the bed filled with material with a coarse grain size, i.e. a calcined siliceous limestone, a higher efficiency in the removal of pathogenic bacteria was also observed in comparison with the medium-sized grain. On average, it amounted to over 99.7% (see Figure 7), and it was associated with the method of the bacteria reduction due to the formation of struvite [Wąsik et al. 2017].

At the last stage, the analysis of variance of the source data was carried out, concerning the efficiency of ammonium nitrogen, orthophosphate, *Escherichia*

coli bacteria, and coliform bacteria removal. The Shapiro-Wilk test in all cases presented test probabilities at a lower (than assumed) level of significance ($p < 0.05$). For this reason, the hypothesis about the normality of the distribution of the analysed data had to be rejected. The Levene test's result confirmed the heterogeneity of the variance of the efficiency of ammonium nitrogen, orthophosphate, *Escherichia coli*, and coliform bacteria removal at the significance level of 0.05.

The results of the Kruskal-Wallis nonparametric test, performed for variables characterized by the lack of normal distribution and the heterogeneity of variance, are presented in Tables 2–4. For all cases of the

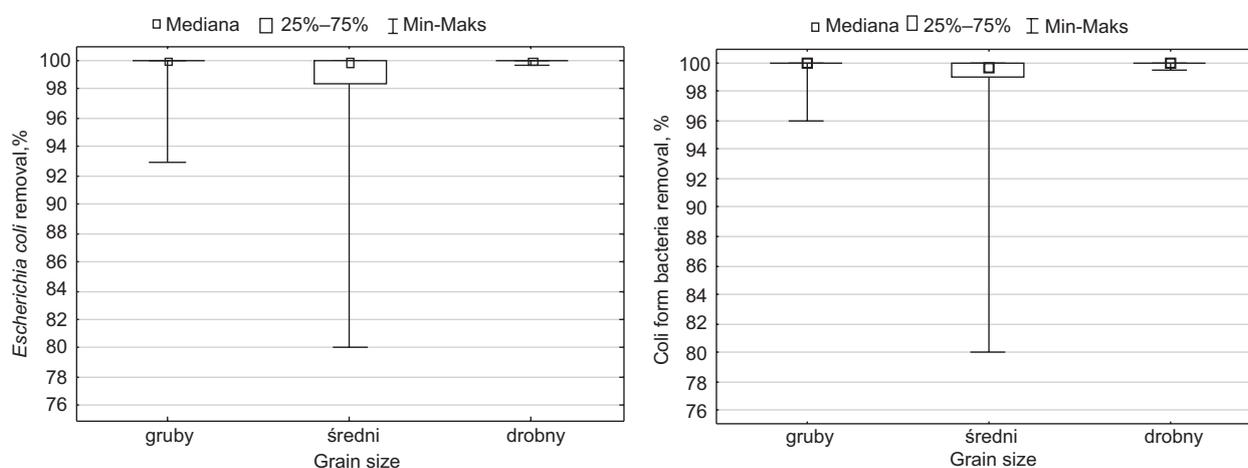


Fig. 7. Box-plot graphs of pathogenic bacteria removal on beds filled with coarse, medium and fine material

grouping variable, the test probability p was obtained at a level lower than 0.05, which indicates a significant difference in the degree of removal of the contaminants in the compared groups of materials due to their selective properties, porosity, and granulation.

Table 2. Kruskal-Wallis test results (independent variable: selectivity)

Filter	ANOVA; Mean rank value Kruskal-Wallis test: $p < 0.05$			
	<i>E. coli</i> removal %	Coliform bacteria removal %	N-NH ₄ ⁺ removal %	PO ₄ ³⁻ removal %
ZNSS	32.87778	41.96875	59.88136	41.06667
ZNSN	48.80952	77.93478	52.78261	43.60526
ZSN	83.11957	97.94792	76.28261	71.29762

Table 3. Kruskal-Wallis test results (independent variable: porosity)

Filter	ANOVA; Mean rank value Kruskal-Wallis test: $p < 0.05$			
	<i>E. coli</i> removal %	Coliform bacteria removal %	N-NH ₄ ⁺ removal %	PO ₄ ³⁻ removal %
ZNP	38.63889	49.27333	56.91429	39.36792
ZP	73.12931	91.40833	73.65517	67.63208

Table 4. Kruskal-Wallis test results (independent variable: grain size)

Filter	ANOVA; Średnia ranga / Mean rank value Test Kruskala-Wallis / Kruskal-Wallis test: $p < 0.05$			
	<i>E. coli</i> removal %	Coliform bacteria removal %	N-NH ₄ ⁺ removal %	PO ₄ ³⁻ removal %
Coarse	83.94872	99.17949	61.16216	81.64286
Medium	33.72115	44.29577	59.84848	40.15686
Fine	61.92857	86.68000	81.72000	38.27500

CONCLUSIONS

As the analysis of variance has shown, the effectiveness of filtration media in terms of ammonium nitrogen, orthophosphate and indicator bacteria removal was influenced by the selectivity, porosity, and grain size of the fill.

Taking into account the effectiveness of biogenic compounds removal, it was found that natural selective porous materials, such as zeolite and calcined siliceous limestone, facilitated the removal of ammonium nitrogen and orthophosphate in the highest degree – over 73%, and 69–79% respectively.

The filter filled with a natural porous material with fine graining exhibited a very high degree of stoppage

of pathogenic bacteria. The use of zeolite as a filter material made it possible to reduce the numbers of *Escherichia coli* bacteria in the range of 99.65–99.98%, and coliform bacteria, in the range of 99.81–99.94%.

Natural materials, such as for instance zeolite, have the highest average ammonium nitrogen removal rate compared to sand or expanded clay [Dacewicz and Chmielowski 2018]. Sand filters are the most effective in reducing indicator bacteria [Wąsik and Chmielowski 2017b], but their propensity to colmatization is a disadvantage. It is recommended that research be undertaken in the field of developing a solution that would protect the sand filter against clogging. It is proposed that further research be conducted, using other plastic materials that are an alternative filling of vertical filters [Dacewicz et al. 2018].

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ZASTOSOWANIE MATERIAŁÓW SELEKTYWNYCH I POROWATYCH DO USUWANIA ZWIĄZKÓW BIOGENNYCH I BAKTERII WSKAŹNIKOWYCH ZE ŚCIEKÓW BYTOWYCH

ABSTRAKT

W artykule przedstawiono ocenę skuteczności usuwania związków biogenych oraz bakterii wskaźnikowych ze ścieków bytowych w filtrach pionowych o zróżnicowanym wypełnieniu. Analizie poddano keramzyt, zeolit, wyprażaną opokę oraz rozdrobnione płatki PET. W oparciu o badania ścieków surowych i oczyszczonych określono efektywność usuwania azotu amonowego, ortofosforanów oraz bakterii *Escherichia coli* i bakterii grupy *coli* w zależności od selektywności, porowatości oraz wielkości uziarnienia wypełnienia. W przypadku usuwania związków biogenych najskuteczniejszym wypełnieniem okazały się naturalne materiały selektywne porowate, tj. zeolit i wyprażana opoka. Stwierdzono na nich bardzo wysoki stopień usunięcia odpowiednio jonów amonowych na poziomie 75,34% oraz ortofosforanowych powyżej 79%. Wykazano, że filtr zasypany naturalnym materiałem porowatym o drobnym uziarnieniu okazał się najbardziej odpowiedni do usunięcia bakterii wskaźnikowych. Stopień eliminacji bakterii grupy *coli* wyniósł 99,94%, a bakterii *Escherichia coli* 99,98%.

Słowa kluczowe: oczyszczanie ścieków, związki biogenne, *Escherichia coli*, bakterie grupy *coli*, skuteczność, filtr o przepływie pionowym