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# Removal of Boron from Groundwater by Filtration Through Selected Filter Bed Materials

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## 1. Introduction

In recent years, there has been a significant increase in boron concentration in surface water and groundwater as a result of both natural and anthropogenic factors (Wolska, Bryjak 2013). Natural sources of boron emission are weathering rocks, volcanic eruptions, etc. In addition, anthropogenic factors are associated with human activities through emissions, effluents, or wastewater from industrial plants. Salts of boric acid and boron ($BO_2^-$, $B_4O_7^2$, $BO_3^{3-}$, $H_2BO_3^-$ and $H_4BO_4$) are widely applied in many industrial branches (Hilal et al. 2011; Turek, Dydo 2013). Boron is used in the manufacture of borosilicate glass, fiber glass, enamel, soaps and detergents, flame-retardants, glaze, porcelain, preservatives for the production of high-concentrated herbicides and artificial fertilizers in small doses. Scientists and technologists are still looking for such methods for boron removal from water, which, with low investment costs, would not lead to secondary environmental pollution. According to the WHO boron concentration should not reach 0.5 mg/l (WHO 1998). Therefore, the aim of the study was to analyze the efficiency of boron removal from groundwater using unit water treatment processes that are successfully applied for water purification at each industrial station.

## 2. Methods

Studies were carried out on different filter bed materials, different in structure, composition and properties. Four filter beds were selected for the experiment: activated carbon, particles – 0.6-1.5 mm; quartz sand, particles – 0.3-0.9 mm; mixed bed made of two different aluminosilicates – modified and natural zeolite, particles – 0,3-1,5 mm; pyrolusite, particles – 0.6-1.5 mm. The raw water samples were taken from deep wells of about 25 m depth.

**Analytical methodology:** color was measured by using UV-Visible spectrophotometry method in accordance with PN-EN ISO 7887, turbidity was determined by nephelometric method in accordance with PN-EN ISO 7027, pH was measured by potentiometric method based on PN-EN ISO 10523,chloride concentration was determined by using silver nitrate titration, in the presence of dichromate as an indicator (Mohr method) in accordance with PN-ISO 9297, electrolytic conductivity was measured by using conductometric method based on PN-EN 27888, iron concentration measured by using spectrophotometry method with ammonium thiocyanate, manganese concentration determined by using spectrophotometry method with ammonium persulfate (Hermanowicz et al. 1999), COD-Mn was made by using potassium permanganate (VII) in accordance with PN/C-04578.02, total hardness measured by using EDTA titrimetric method in accordance with PN-ISO 6059, total alkalinity and mineral alkalinity measured by using titrimetric method based on PN-EN ISO 9963-1, the boron concentration was measured by using the carmine method in concentrated sulfuric acid (VI) and azomethine-H method (HACH Co., Ltd., USA, method 8015 and method 10274). Table 1 presents the results of pollution level for raw water.

**Table 1.** Raw water parameters
**Tabela 1.** Parametry fizyko-chemiczne wody surowej

| Parameter | Concentration | Unit |
|---|---|---|
| **Color** | 20 | mg Pt/l |
| **Turbidity** | 1.7 | NTU |
| **pH** | 7.85 | - |
| **Conductivity** | 384.7 | μS/cm |
| **Chlorides $Cl^-$** | 0.5 | mg $Cl^-$ /l |
| **Concentration of Fe** | 1.1 | mg Fe/l |
| **COD-Mn** | 7 | mg $O_2$/l |
| **Concentration of B** | 2.1 | mg B/l |
| **Total alkalinity At** | 275.22 | mg $CaCO_3$/l |
| **Total hardness** | 200.16 | mg $CaCO_3$/l |
| **Concentration of Mn** | 0.5 | mg Mn/l |

The study was conducted in three test series. The test samples were collected once a day, every 24 hours. Statistical analysis based on a calculation of the Pearson correlation coefficient was performed using licensed software Statistica 12.5. The results of statistical analyzes were based on 95% confidence level. Pearson correlation coefficients were calculated between boron concentrations and all measured parameters in samples of water after filtration on each of the four filtration materials.

## 3. Results and Discussion

According to the Regulation of Minister of Health from 13th November 2015, the concentration of boron in drinking water should not exceed 1 mg B/l, and WHO sets 0.5 mg B/l. The raw water used for the research contained boron at the concentration of 2.1 mg B/l. The experiments were carried out in three research series. Each of them was completed with the deposit clogging and necessity of their backwash. It was realized by using water fed in counter-current with double intensity in relation to normal operation and for 30 minutes. The first series of tests made it possible to achieve the best results of boron removal from water on all tested filtration materials (Table 2). Boron concentration from 2.1 mg B/l decreased to 0.034 mg B/l on activated carbon, 0.45 mg B/l on pyrolusite, 0.45 mg B/l on quartz sand, and 0.05 mg B/l on mixed zeolite bed (Figure 1). The maximum sorption capacity of activated carbon relative to boron is 1 59 mg B/g (Pieńczak, Warchoł 2013). In comparison maximum sorption capacity to boron for fly ash reaches similar level (Osturk N., Kavak D. 2005) and over 200 mg/g for waste sepiolite (Osturk N., Kavak D. 2004). Both in the second and third series, boron concentration greatly increased, as compared to the one in raw water, amounting respectively to: 2.85 and 2.60 mg B/l on activated carbon, 3.45 and 3.116 mg B/l on pyrolusite, 2.316 and 2.211 on quartz sand, as well as 2.91 and 1.92 mg B/l on mixed bed. Molecules of boron compounds retained during the first series began to separate from deposit particles, resulting in the secondary deterioration of water flowing through the filters. The most effective filtration material for the removal of boron compounds proved to be the active carbon and mixed bed, on which the average efficiency amounted to 13% and 21%, respectively (Figures 1 and 2). In contrast, the efficiency of boron removal on quartz sand was about 8% on average.

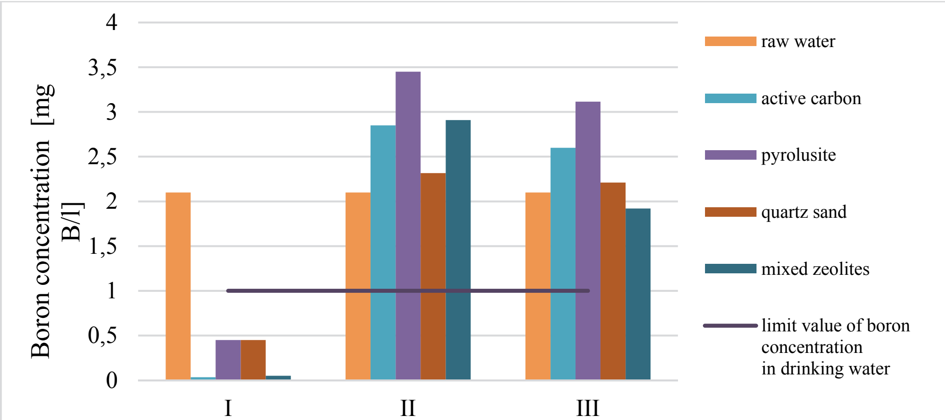


**Fig. 1.** Boron concentration in raw and filtrated water
**Rys. 1.** Stężenie boru w wodzie surowej i przefiltrowanej przez wybrane masy filtracyjne

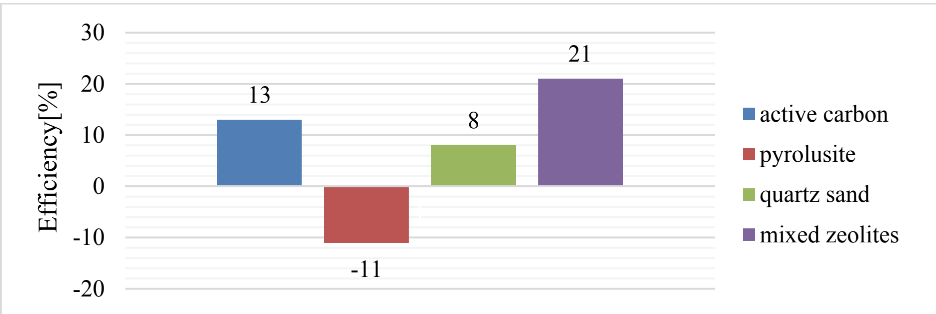


**Fig. 2.** Final efficiency of boron removal from the water through different filter bed materials
**Rys. 2.** Porównanie średniej efektywności usuwania boru metodą filtracji na wybranych złożach filtracyjnych

In order to assess the relationship between boron concentration and other water quality parameters after filtration, the Pearson correlation coefficients were calculated (Table 3).

**Table 2**. Filtrated water parameters through selected filtration materials
**Tabela 2**. Parametry fizyko-chemiczne wody po procesie filtracji na wybranych złożach

| Parameter | unit | Raw water | I series | | | | II series | | | | III series | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | *1 | *2 | *3 | *4 | *1 | *2 | *3 | *4 | *1 | *2 | *3 | *4 |
| **Color** | mg Pt/l | 25 | 1 | 8 | 7 | 0 | 7 | 22 | 16 | 10 | 6 | 3 | 23 | 15 |
| **Turbidity** | NTU | 2,0 | 0.98 | 0.66 | 1.5 | 1.2 | 0.64 | 0.76 | 1.37 | 1.11 | 2.2 | 1.25 | 3.03 | 2.55 |
| **pH** | - | 7.98 | 7.51 | 8 | 7.8 | 8.18 | 8.5 | 8.5 | 8.3 | 8.67 | 8.06 | 7.95 | 8.37 | 8.07 |
| **Cond.** | μS/cm | 415 | 337 | 347.5 | 367.5 | 490 | 355.7 | 366 | 378.5 | 514 | 326.3 | 324.5 | 450.2 | 335.8 |
| **$Cl^-$** | mg $Cl^-$/l | 10 | 10 | 10 | 10 | 10 | 3 | 3 | 2 | 1 | 15 | 5 | 6 | 5 |
| **Fe** | mg Fe/l | 0.9 | 0.011 | 0.016 | 0.037 | 0.018 | 0.031 | 0.022 | 0.05 | 0.02 | 0.044 | 0.034 | 0.088 | 0.058 |
| **COD-Mn** | mg $O_2$/l | 5.5 | 0.5 | 1 | 1.8 | 1 | 0.3 | 0.5 | 1 | 0.3 | 0.3 | 0.5 | 1 | 0.2 |
| **B** | mg B/l | **2.1** | **0.034** | **0.45** | **0.45** | **0.05** | **2.85** | **3.45** | **2.316** | **2.91** | **2.6** | **3.116** | **2.211** | **1.92** |
| **$Z_p$** | - | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| **$Z_m$** | mg $CaCO_3$/l | 200.2 | 25.02 | 100.08 | 150.12 | 275.22 | 120.1 | 220.18 | 220.18 | 270.2 | 125.1 | 200.2 | 200.2 | 250.2 |
| **Hard.** | mg $CaCO_3$/l | 1000 | 700.6 | 820.66 | 780.66 | 1301.1 | 240.19 | 160.13 | 180.14 | 280.2 | 900.7 | 920.7 | 200.2 | 1481 |
| **Mn** | mg Mn/l | 0.3 | 0 | 0 | 0.002 | 0 | 0 | 0 | 0.001 | 0 | 0.001 | 0 | 0 | 0.005 |

*1 – activated carbon, 2 – pyrolusite, 3 – sand, 4 – mixed zeolites

**Table 3.** Pearson coefficients of correlations between boron and the rest of water pollutants
**Tabela 3.** Współczynniki korelacji Pearsona między stężeniem boru a innymi parametrami wody po filtracji na złożach filtracyjnych

| R | Active carbon | Pyrolusite | Sand | Mixed zeolite |
|---|---|---|---|---|
| | **Conc.B** | **Conc. B** | **Conc.B** | **Conc. B** |
| **Color** | **0.9971*** | 0.3604 | **0.8768*** | 0.7768 |
| **Turbidity** | 0.2324 | 0.5488 | 0.3922 | 0.1197 |
| **pH** | **0.9289*** | 0.5168 | **0.9868*** | 0.6429 |
| **Conductivity** | 0.2339 | 0.0392 | 0.5059 | -0.0514 |
| **$Cl^-$** | -0.1753 | **-0.9840*** | **-0.8900*** | **-0.9938*** |
| **Fe** | **0.8861*** | 0.6855 | 0.6604 | 0.4783 |
| **COD-Mn** | **-0.9968*** | **-0.9948*** | **-0.9987*** | **-0.8948*** |
| **At** | **0.9922*** | **0.9985*** | **0.9736*** | -0.3584 |
| **Hardness** | -0.2993 | -0.4829 | **-0.9998*** | -0.6684 |
| **Mn** | 0.4289 | 0.0000 | **-0.8399*** | 0.1749 |

*strong correlation

It was noticed that a strong positive correlation occurs between boron concentration vs. color when filtration takes place on activated carbon, quartz sand, and mixed bed. The highest Pearson correlation coefficient was recorded for filtration on activated carbon r = 0.9971, while the lowest – on pyrolusite r = 0.3604, where correlation is virtually absent. Efficiency of water color purification applying filtration on different materials was documented earlier in literature (Skoczko et al. 2015; Kaleta et al. 2009). The color concentration decreased after each filtration procedure. Permissible value in drinking water was exceeded during filtration on pyrolusite in the second series and on quartz sand in the second and third series of tests. Such strong correlation between boron concentration and color may result from the fact that boron molecules can form color compounds with other contaminants present in water. The Pearson coefficient between boron concentration and turbidity was below 0.5 indicating that there is no correlation between these traits. Efficiency of turbidity removal from water on different filtration materials was earlier described in literature (Skoczko et al. 2015, Kaleta et al. 2009). Analysis of the dependence between water pH and boron concentration in water after filtration on various beds reveals that almost complete positive correlation occurs on activated carbon and quartz sand. Correlation coefficient is: r = 0.9289 and r = 0.9868, respectively. Whereas dependence of boron concentration on pH after filtration on pyrolusite and mixed bed is quite weak for respective. Dependence of boron concentration on water pH can be explained by a conversion of boron form in water. At pH value increased to about 8, boric acid in molecular form prevails (Bodzek, Konieczny 2011). For majority of sorption materials, the maximum sorption capacity can be obtained for pH = 9 or above, where bivalent anion $H_{10}(BO_3)_4^{2-}$ dominates. Minerals that better absorb boron in acidic environment are an exception. Lemarchand et al. (2005) reported that boron sorption by humic acids reaches the maximum at $5 < pH < 9$ and suddenly decreases at $pH > 9$. This was confirmed by tests performed within frames of the present study.

Pearson coefficient between boron concentration and electrolytic conductivity amounted to less than 0.5, indicating that no dependence between these two traits was observed. Therefore, insoluble or neutral boron forms predominated in treated water, which was confirmed by pH value about 8.

Correlation between boron and iron concentration is at the moderate level or does not occur at all. The lowest Pearson coefficient was achieved in filtration on mixed bed r = 0.4783. In the case of quartz sand and pyrolusite, average r coefficient was about 0.68, whereas on activated carbon r = 0.8861. In every case, it was the positive correlation. According to Table 2, iron concentration in water treated on particular filtration materials oscillated from 0.01 mg Fe/l to 0.06 mg Fe/l.

Table 3 shows that there is a very large negative correlation between the concentration of boron and COD-Mn. In any case, the Pearson coefficient is greater than 0.8. Negative values indicate that parameters were changed in the opposite direction. In each test series, the concentration of boron increases, which is probably due to the exhaustion of the adsorption properties towards boron compounds and the concentration of organic and certain inorganic compounds expressed as COD-Mn is reduced in each of the three series. The highest correlation was obtained on activated carbon r = -0.9987, and the lowest during filtration on a mixed bed r = -0.8948. Permissible COD-Mn concentration in drinking water was exceeded only in raw water, while after filtration, this value substantially decreased. The highest COD-Mn concentration was achieved on quartz sand and the lowest on activated carbon. Mean value of COD-Mn ranged from 0.37 mg $O_2$/l to 1.27 mg $O_2$/l. This water parameter decreased due to filtration on all analyzed beds as compared to boron concentration, because of bio-sorption processes. Microorganisms colonizing the activated carbon or quartz sand decompose organic compounds, thus COD-Mn gets decreased in the treated water. Taking into account the correlation coefficients due to bio-sorption, boron-containing compounds were not removed, because boron concentration increased as compared to the COD-Mn value (Kaleta 2005, Prus et al. 2009).

The relationship between the boron concentration and alkalinity in three cases – filtration on activated carbon, pyrolusite and sand – is characterized by a positive value of the Pearson coefficient, while on mixed bed, a negative correlation is shown. In each of the correlations, Pearson coefficient is close to 1, which indicates a very strong dependence between boron and alkalinity. This fact is related to the change in the form of boron present in water, depending on the nature of the aqueous medium. In natural waters, boron occurs most often in the form of not dissociated boric acid in molecular form, which depends largely on the pH of water (Bodzek, Konieczny 2011).

According to the correlation coefficients between the boron and manganese concentrations, a positive relationship can be observed in two cases: on activated carbon and mixed bed, while a strong negative correlation was obtained on quartz sand. Pearson correlation coefficient $r = 0$ was recorded after filtration process on pyrolusite. The highest manganese concentration was observed in the third series on mixed bed – 0.005 mg Mn/l and in the first series on activated carbon – 0.003 mg Mn/l. Other test samples revealed the absence of manganese or its concentration below 0.002 mg Mn/l. The high negative correlation on quartz sand is probably due to the high affinity of the absorbent material to adsorbed manganese, which reduced the concentration of this parameter, and resulted in an increase in boron concentration as a result of blocking the active coating of filter bed particles.

## 4. Summary

Therefore, the trial to assess the effectiveness of boron removal during the process of filtration on selected filtration beds, was undertaken within frames of the present study. It was confirmed that the final effect of boron compounds removal from water flowing through a deposit depends on many factors, including: granulation of bed particles, porosity, filtration rate, type and size of contaminants particles, etc. The experiments prove that not every bed that is able to remove boron from water, and is sensitive to elevated values of this parameter. Bed composed of two aluminosilicates, as well as activated carbon appeared to be the most effective (Figure 2). Mean efficiencies amounted to 21% and 13%, respectively, taking into account all test series. Both beds are materials with improved sorption capacity. Boron was adsorbed within their pores and not only on their surface like in the case of other tested beds. Due to large dimensions of boron complex particles, it was difficult to elute it from bed pores, which resulted in lower efficiency in subsequent series. Another tested bed that allowed for partial retaining the boron compounds was quartz sand (mean efficiency 8%). Pyrolusite appeared to be completely useless for boron removal. The average boron concentration resulting from three series of filtration increased by 11% in treated water. When analyzing only the first series of tests, activated carbon appeared to be the most effective with the efficiency of boron removal reached up to 98.4%, while quartz sand removed 79% of boron. In subsequent series, there was a secondary release of retained boron into the water. Boron concentration increased in the second series by 36%, and in third by 23%, as compared to the raw water. Zeolite mixed bed used in the first series allowed for achieving 97.6% efficiency, while in the second series, boron concentration increased respectively by 38%, and in the third series, slight efficiency at the level of only 8.6% was recorded. According to some researchers (Pieńczak, Warchoł 2013), boron removal efficiency on various filtration materials depends on their structure (porosity, specific surface) as well as type of functional groups responsible for complexation of boron ions present in filtered water. The latter phenomenon determined the worsening of the filtrate quality in subsequent test series realized in the present study. The first series allowed for an efficient retaining of boron, while in the subsequent ones a secondary release of boron into the water occurred. It indicates that rinsing the beds should not be performed using only the tap water. When it is known that water subject to treatment contains elevated concentrations of boron, the washing agent should at first alter the pH of water and form of boron retained on a bed. Dogan (2007) observed that pH value determines the protonation and de-protonation of functional groups of a sorbent, as well as the type and concentration of boron forms in a solution, which remarkably affects the removal efficiency. According to Wang et al. (2013), for most of sorption materials, maximum sorption effectiveness can be obtained for pH = 9 or above, for which where bivalent anion $H_{10}(BO_3)_4^{2-}$ dominates.

Minerals that better absorb boron in acidic environment are an exception. On the other hand, Lemarchand et al. (2005) reported that boron sorption by humic acids reaches the maximum at $5 < pH < 9$ and suddenly decreases at $pH > 9$. It indicates that efficient rinsing should be made in two stages: firstly, using strongly alkaline means that convert molecular boron onto ionic form (reaction of rinsing solution should be greater than 9), and then using tap water.

Based on the study, a conclusion was drawn that better efficiency of boron removal can be achieved in the process of filtration on analyzed beds using a multi-stage filtration. The described experiments indicate that the single-stage filtration is ineffective. Similarly, Kluczka et al. (2007) found that achievement of a high degree of water purification from boron compounds can be realized by means of applying a series of columns filled with activated carbon. This theory is confirmed by studies performed by Piecuch (Piecuch et al. 2000), who proved higher efficiency of multi-stage filtration when studying the efficiency of industrial sewage treatment on carbon deposit. A single-stage filtration should not be applied at higher boron concentrations in water. The use of multistage filtering enables the elimination of an ion exchange process, in which the post-regeneration washings generate large costs associated with disposal of these solutions (Kluczka et al. 2007). The authors of the present study tested sand bed, pyrolusite, mixed zeolite, and carbon filter. Typical water treatment station includes these combined in a series of technological system, which means that they are usually used as multistage filtration. Considering the conducted experiments from this point of view, overlapping boron removal efficiency can be achieved, which will reduce its concentration to the normative values.

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PN-EN 27888 – Water quality – Determination of the electrical conductivity
PN/C-04578.02 – Determination of the chemical oxygen demand (COD) by permanganate
PN-ISO 6059 – Water quality – Determination of total calcium content and magnesium - EDTA titration method
PN-EN ISO 9963-1 – Water quality – Determination of alkalinity – Part 1: Determination of alkalinity and total alkalinity to phenolphthalein

# Usuwanie z wody związków boru metodą filtracji na wybranych złożach

## Streszczenie

Obecnie na skutek intensywnej działalności antropogenicznej wody powierzchniowe i podziemne zagrożone są poważnym skażeniem związkami boru. Większość stacji uzdatniania nie projektuje specjalnych technologii tylko do jego usuwania. W związku z tym celem prowadzonych prac badawczych była analiza efektywności usuwania boru z wód podziemnych za pomocą takich metod uzdatniania, które są wykorzystywane na typowych stacjach. W pracy porównano skuteczność filtracji na wybranych masach różniących się składem i właściwościami takich jak: węgiel aktywny, piasek kwarcowy, złoże mieszane zeolitowe oraz piroluzyt. Sprawdzono też korelacje pomiędzy innymi zanieczyszczeniami wody a borem. Przeprowadzone w trzech seriach badania pokazały, iż można skutecznie usuwać bor z wody prawidłowo wykonanych płukaniach związkami o charakterze alkalicznym a następnie wodą. W przeciwnym przypadku w kolejnych seriach filtracyjnych następuje wtórne uwalnianie boru oraz znaczny spadek efektu oczyszczania.

**Słowa kluczowe:**
bor, złoża filtracyjne, woda podziemna, filtracja

**Keywords:**
boron, filter bed, underground water, filtration