



Laboratory elimination of ibuprofen from water by selected adsorbents

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ABSTRACT

The laboratory elimination of Ibuprofen from water by means of adsorption on selected adsorbents was part of specific academic research at the Department of Municipal Water Management, Faculty of Civil Engineering, Brno University of Technology. The purpose of the laboratory trial was to compare three selected sorbents, namely Filtrasorb F100, Bayoxide E33, and GEH, with regard to their effectiveness of Ibuprofen elimination from water. Filtrasorb F100 charcoal is generally used in practice for the elimination of medicinal product residues and other micropollutants. The sorbents Bayoxide E33 and GEH are used for the elimination of metal residues from water. The trial water was prepared in the laboratory by mixing the medicinal product with drinking water. An analysis of the samples taken after filtration from the individual columns in different time intervals proved that Bayoxide E33 and Filtrasorb F100 managed to successfully eliminate Ibuprofen from the water. The sorption material GEH became oversaturated in the course of filtration and did not continue to adsorb, with desorption instead appearing after some time.

Keywords: Water treatment; Adsorption; Ibuprofen; Filtrasorb F100; Bayoxide E33; GEH; Chromatography; Mass spectrometry; Effectiveness of sorption materials; Remove pharmaceutical

1. Introduction

Although concentrations of pharmaceuticals in the environment are in the range of ng/L to µg/L, the impact of such low concentrations on nontarget organisms is not fully understood. Prolonged exposure and the synergistic effect of several pharmaceuticals simultaneously occurring in low concentrations in the same ecosystem can pose serious threats. The concern with the detection of a compound in an environment truly arises when there is legitimate proof of their adverse impacts on aquatic and human life [1]. Methods for removing drugs from water have improved greatly in recent years. Membrane filtration, advanced oxidation processes, but also adsorption are among the reliable methods for removing pharmaceuticals from water. The adsorption process has gained momentum owing to its

simple design, low cost, flexibility, easy to operate nature, and insensitivity to toxic contaminants [1,2].

1.1. Adsorption

Adsorption is extremely important in hydrochemistry, as well as in water technology. A general definition describes adsorption as enrichment by chemical substances from the liquid form on the surface of a liquid or a solid substance. Adsorption is used for the removal of substances, for example, micropollutants, from liquid or solid states. Adsorption can be observed as a natural process in various elements of the environment. The adsorbed substance is called adsorbate and the substance on the surface of which adsorption takes place is called adsorbent. As adsorption is a surface process, the specific surface of the adsorbent is the key

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parameter of adsorbent quality. Most suitable adsorbents are represented by porous materials with a specific surface area in units of 10^2 and 10^3 m²/g. Adsorbents are categorized by polarity or acidity. Polar adsorbents include for example silica gels, hydrated oxides, and feldspars. Charcoal is an example of a non-polar adsorbent. Polar adsorbents better adsorb polar compounds and vice versa. Alkali adsorbents include hydrated oxides and acid adsorbents are represented by silica gels and silicates [3,4].

Adsorbent types currently used for drinking water treatment include granulated activated carbon (GAC), powder activated carbon (PAC), and ion exchanger-based adsorbents. The weight balance of the adsorbate consists of several phases, including the volume phase, aqueous phase, and adsorbent phase. The weight of the adsorbed pollutant per unit of weight of the adsorbent depends on the pollutant concentration in the aqueous phase. In the balanced state, there is the process of dynamic exchange between the molecules of the adsorbent phase and the molecules remaining in the adsorbate [5].

Adsorption included at the end of the technology line for drinking water treatment will positively affect drinking water quality in several basic ways. With the inclusion of adsorption on charcoal after the separation elements of the standard water treatment technology line (such as coagulation, flotation, and filtration), the effect of the thus extended water treatment line on drinking water quality is excellent. There are no by-products of point-blank oxidation (for example by ozone) and thus the sorption stage which follows after the previous separation of particles and organic substances with higher molecular weights is only reached by substances separable by adsorption. There is obviously a wide range of these and, from the hygienic point of view, the most important of them are micro pollutants, including medicinal product residues [6].

1.2. Ibuprofen

For the purpose of the experimental elimination of a medicinal product residue from water, Ibuprofen was selected for the reason of its proven presence in drinking water sources and in circulated drinking water itself. Ibuprofen is a well-known analgesic (pain-reducing) and antipyretic (body temperature reducing) drug. As it is also anti-inflammatory, it is classified as one of the non-steroidal anti-inflammatory drugs (NSAIDs). Experiments have showed that Ibuprofen is up to 30 times stronger than Aspirin and 20 times stronger than antipyretics. It is administered against mild and moderate pain of various origins, such as joint, muscle, tooth, and other pain. The drug is an

over-the-counter medicinal product that can be obtained without a medical prescription but only in certain limited doses. A medical prescription is needed for the administration of higher doses of Ibuprofen [7,8]. The chemical properties of Ibuprofen are shown in Table 1.

1.3. Presence of Ibuprofen in aquatic environmental matrices

According to data collected from 134 articles published between 1997 and 2009, the main therapeutic classes found in the environment are NSAIDs [9]. According to some studies, Ibuprofen has been found to be present at higher concentrations in sewage treatment plants and subsequently in surface water, ranging from 230 ng/L to 4,500 mg/L [1]. Furthermore, the presence of Ibuprofen and its metabolites in the biota of the environment has been demonstrated. Residual Ibuprofen concentrations included fish plasma, gills, kidneys, liver, and muscles [2]. The fate of Ibuprofen in the environment is dependent on consumption, metabolic capacity in treatment processes, degradation, sorption properties of water and soil components, and other factors such as pH and climatic conditions [10].

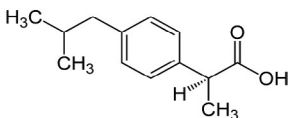
The monitoring of drug residue presence in waters has been performed in the Czech Republic due to media interest. Concentrations of Ibuprofen at the outflow from wastewater treatment plants reached up to 11.2 µg/L. As wastewater treatment plants were unable to completely eliminate the drug from wastewater, the residues were introduced to surface water where the maximum measured level of Ibuprofen was 4.4 µg/L. As surface water may be a source of drinking water, drug presence analysis in drinking water was also performed. Ibuprofen levels in drinking water reached a maximum of 0.12 µg/L. Laboratory trials have proved that medicinal products present in drinking water may display toxic effects on microorganisms and human cells. Drug levels in drinking water are not yet problematic for people so much as they are problematic for the environment [11].

2. Materials and methods

2.1. Adsorbent characteristics

Three adsorbents were used for the laboratory experiment of Ibuprofen elimination from water. The charcoal adsorbent Filtrasorb F100 was selected as a standard adsorbent used for the removal of micro-pollutants from water. The adsorbing materials Bayoxide E33 and GEH were selected on the basis of the favorable results of the laboratory removal of metals from water. The characteristics

Table 1
Ibuprofen properties [8]

Designation	Structural formula	Summary formula	Molar weight	Melting point
Ibuprofen		C ₁₃ H ₁₈ O ₂	206.28 g/mol	76°C

and properties of the individual adsorbents are described below.

2.1.1. Filtrasorb F100

The granulated active carbon Filtrasorb F100 (Fig. 1) is used for the elimination of dissolved organic compounds from water. GAC has been used in water management for over four decades in applications such as drinking and process water treatment, wastewater treatment, food, pharmaceutical, and industrial water treatment. The granules of F100 are made from selected bitumen coals using the process known as re-agglomeration (flocculation). Active carbon resists wear connected with repeated washing, hydraulic transport, and reactivation for reuse. The raw coal is extracted in the United States and GAC is subsequently produced from it to assure the maximum quality and consistency of the finished product. The activation is carefully monitored with the aim of producing a significant number of both low and high-energy pores for effective adsorption of a broad range of organic contaminants [12]. The technical and physical parameters of the Filtrasorb F100 adsorption material are summarized in Table 2.

2.1.2. Bayoxide E33

The adsorption crystalline iron-oxide-based medium Bayoxide E33 (Fig. 2) is made by the British Company Severn Trend Services mainly for the purpose of the elimination of arsenic and other metals from water. This adsorption material is able to remove arsenic by the reduction of its levels in water below 4 µg/L. The sorbent is used in granulated form as Bayoxide E33 or in tablets as Bayoxide E33P. The advantages of this material include long life in continuous operation, low investment and operation costs, and the long life of the dry medium [13]. The technical and physical parameters of the adsorbent are summarized in Table 2.

2.1.3. GEH

GEH (Fig. 3) is a highly efficient iron-hydroxide-based adsorbent. It is made by a specialized patented process and used for selective adsorption of arsenic in a specific process. This medium is suitable for drinking water treatment for it does not release any chemical compounds into the treated water and does not change its pH. The technology of treatment is based on the adsorption of the contaminant on granulated iron hydroxide (GEH sorbent), placed in a reactor through which the treated water flows. The adsorption capacity of the material depends on operation conditions. GEH material should be stored in plastic barrels, big bags, or silos. The material is stable and its shelf life is up to 1 y. Product drying must be prevented, though, which may be caused by strong sunshine [14,15]. The properties of the adsorbent are summarized in Table 2.

2.2. Filtration kit

The filtration kit (Fig. 4) consisted of a barrel with the model water, a pump, three filtration columns, a pipeline



Fig. 1. Sorption material Filtrasorb F100.



Fig. 2. Sorption material Bayoxide E33.

with stop valves and containers for the filtered water. The inner diameter of the columns was 4.4 cm and their bottom was filled with a draining layer preventing the leakage of the adsorption material. The draining layer consisted of a layer of fine stones in the size of 1–2 cm, glass beads in the size of 4 mm and glass beads in the size of 2 mm. Over the drainage layer, there was a layer of the filtration fill selected according to the manufacturer recommendations with a thickness between 0.75 and 0.8 m. The material filtration layer needs to be washed before filtration. The wash was performed by the opposite water flow to the filtration itself, that is, from the bottom to the top, until the water outflow was absolutely clear and the material was completely washed clean. After that, filtering was performed for all the materials.

Table 2
Technical and physical parameters of adsorption materials [12–15]

Parameter	Unit	Filtrisorb F100	Bayoxide E33	GEH
Surface area	m ² /g	900	120–200	250–300
Particle size	mm	0.8–1.0	0.5–2.0	0.3–2.0
Density	kg/cm ³	0.58	0.4–0.6	1.25
Working pH content	–	Slightly alkaline	5.5–8.5	5.5–6.5
Minimum pour height	m	0.75	0.8	0.8
Price	CZK/kg	115	480	490
Porosity	%	*	85	72–77
Color	–	Black	Amber	Dark brown-red to black

*Unspecified by the manufacturer.



Fig. 3. Sorption material GEH.

2.3. Model water

The model water for testing was made by mixing drinking water and Ibuprofen standard in a ratio for the resulting drug level in the water to approximate the real concentration in surface waters. Drinking water was taken from the Brno city water supply network, which is operated by Brněnské vodárny a kanalizace, a.s. Water is supplied to the distribution network for the locality from the Palackého vrch water reservoir, from which half of the city is supplied. The quality of drinking water complies with the Decree of the Ministry of Health of the Czech Republic 252/2004 Coll., laying down hygiene requirements for drinking and hot water and the frequency and scope of drinking water control, as amended. The Ibuprofen standard was prepared by the Department of Environmental Chemistry and Technology, Faculty of Chemistry, Brno University of Technology (BUT). A 30l barrel was used for the model water storage. The drug needed to be profoundly mixed in the model water for the concentration to be even across the barrel. Water was pumped from the barrel through a suction hose which was capped off with a suction basket.

2.4. Contact time

The adsorption efficiency depends on various filtration conditions, such as filtration rate, grain size, but also on the adsorbent empty bed contact time (EBCT). The EBCT was calculated according to the formula [4]: The adsorption efficiency depends on various filtration conditions, such as filtration rate, grain size, but also on the adsorbent EBCT. The EBCT was calculated according to the formula [4]:

$$EBCT = \frac{h}{v_f} = \frac{h \times A_r}{V} = \frac{V_r}{V} [\text{min}] \quad (1)$$

where h is the bed height (m), v_f is the flow rate (L/min), A_r is the bed area (m²), V is the volume flow rate (m³/min), and V_r is the bed volume (m³).

A constant flow rate of 35 L/h was chosen to calculate the contact time. Because the adsorbent bulk height in each column was different according to the manufacturer's recommendations, different contact times were calculated (Table 3). EBCT results are shown in the table below. According to the theoretical calculation of EBCT, sampling at 1, 2, 4, and 6 min was evenly distributed.

2.5. Measurement methodology

After preparation of the columns with the adsorption materials and the barrel with the model water to be tested, the laboratory experiment could commence. As a single pump was used for the trial, the model water filtration was performed in the filtration columns one by one. The model water flowed through the pump and a flow meter to the columns for filtration. The pump maintained a stable flow rate of 35 L/h. Bottom outlets were used for the filtered water sampling at the time intervals of 1, 2, 4, and 6 min. The total number of samples taken was four samples of filtered model water per column and a sample of model water from the barrel used for specification of the initial level of the drug. The entire laboratory experiment was performed only once. The laboratory of the Department of Municipal Water Management specified the values of turbidity, pH, and the temperature of each sample.

Turbidity was measured by a HACH 2100Q IS turbidimeter. Turbidity is the rate of summary energy dispersing

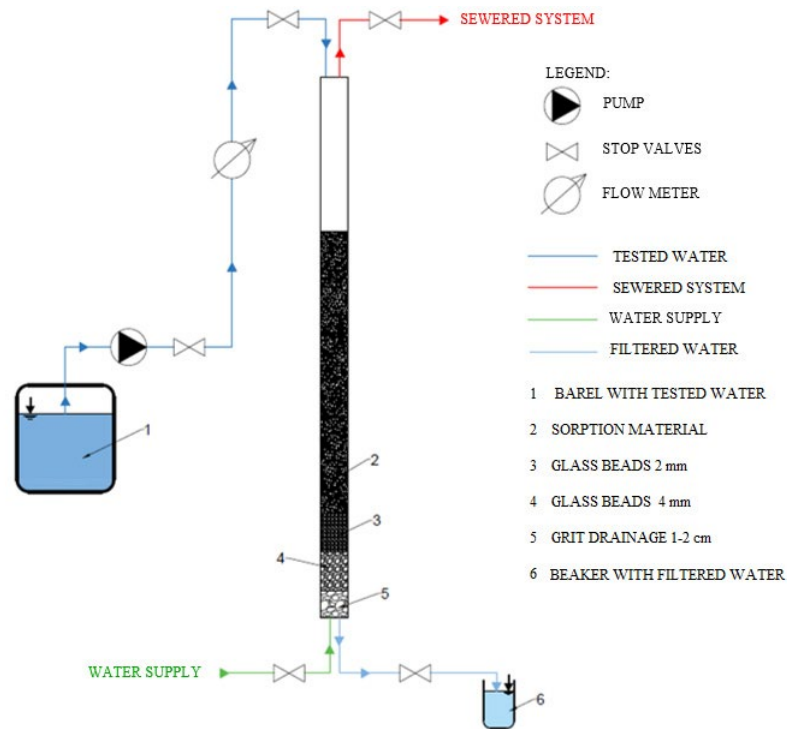


Fig. 4. Diagram of the filter device.

Table 3
Bed height and EBCT for individual sorbent materials

	E33	GEH	F100
h	0.8	0.8	0.75
EBCT	5.79	5.79	5.43

in the course of the pass of a ray of light through a dispersion layer with the unit thickness to all sides from the light beam. Turbidity values are measured by the instrument in FNU (Formazine Nephelometric Units), internationally recognized units for turbidity measurement. The relation to the better known ZF units is as follows: $1 \text{ ZF} = 1 \text{ FNU}$ [16].

In the described laboratory experiment, the water pH was measured with a pH meter with an Adwa AD14 thermometer, a high-standard microprocessor controlled portable pH meter with inbuilt temperature measurement with an automatic temperature compensation feature. The two-line display of the tester shows pH and temperature at the same time. The Adwa meter is watertight and humidity resistant. Its functions include two-cycle automatic calibration, the HOLD function, keeping the measured values on the display, a measurement stability indicator and a battery charge indicator. The meter is equipped with an exchangeable probe [17].

As the laboratory of the Department of Municipal Water Management is unable to determine residual Ibuprofen concentrations in water, the relevant assessment was performed by the Department of Environmental Chemistry and Technology of the Faculty of Chemistry, BUT in Brno.

The samples were evaluated with the help of liquid chromatography with weight spectrometry detection.

2.6. Determination of Ibuprofen concentration

2.6.1. Sample preparation

Ibuprofen was extracted from water samples by solid-phase extraction (SPE) by Supel™-Select HLB, 200 mg, 6 mL (Supelco, Sigma-Aldrich) using a Baker vacuum system (J.T. Baker, Deventer, The Netherlands). Briefly, SPE cartridges were conditioned with 5 mL of high performance liquid chromatography (HPLC) grade methanol followed by 5 mL of Milli-Q water. Two hundred and fifty milliliters of water samples was spiked by 100 ng of a deuterated internal standard of Ibuprofen (IBU-d3) and then processed through the cartridge, dried under a vacuum for 20 min, and eluted with $2 \times 5 \text{ mL}$ of HPLC-grade methanol, dried under a gentle stream of nitrogen and afterwards dissolved in 0.5 mL of HPLC-grade methanol-Milli-Q water (50:50, v/v).

2.6.2. Instrumental analysis

Final analysis, identification and quantification, were performed by HPLC with a diode array detector (DAD) and a mass spectrometry (MS) detector with ion trap analyzer and electrospray ionization (HPLC-DAD-MS; HPLC Agilent 1100 Series; Mass spectrometer Agilent 6320 Series, ion trap liquid chromatography–mass spectrometry (LC/MS)). Chromatographic separation was achieved with the Kinetex C18 ($150 \times 3.0 \text{ mm}$, $2.6 \mu\text{m}$) column. The optimum column temperature was adjusted to 40°C . For the analysis, eluent A was methanol and eluent B was 10 mM formic acid at a

flow rate of 0.3 mL/min. The sample injection volume was set at 20 μ L. The gradient program of eluent A (%): $t_0 = 40$, $t_6 = 90$, $t_{14.5} = 90$, and $t_{17} = 40$ with a post-time of 7.2 min. The total time of analysis was 34.2 min. The retention time of Ibuprofen was 11.6 min.

3. Results

3.1. Resulting Ibuprofen concentrations after adsorption

The initial concentration of Ibuprofen in the tested model water was 1.02 μ g/L. The adsorption material Filtrasorb F100 reduced Ibuprofen concentration already after a minute to 0.12 μ g/L, but as further minutes passed the concentration increased again. The pH value rose in the course of adsorption through activated carbon after the first minute and then rested on about 7.5. The water temperature also rose after the first minute of adsorption and then dropped to 19°C after minute 6. The low temperature of the tested model water was caused by temperature measurement immediately after the taking of the water from the tap and mixing it with the drug standard. Turbidity increased after the first minute, probably due to the elevation of some particles by filtration to the sample taken but stabilized from minute 2 at 1.14 ZF. The results of water analysis after adsorption on Filtrasorb F100 charcoal are summarized in Table 4.

Bayoxide E33 material eliminated Ibuprofen in a manner similar to charcoal. After the first minute, the Ibuprofen level went down to 0.08 μ g/L and then increased slightly. Residual concentration ranged around 0.13 μ g/L on average. Through the effect of Bayoxide E33, the pH of the filtered water increased to the mean value of 7.46. The temperature rose in the beginning, but dropped back down in the last two samples of the series. Turbidity decreased since the beginning of the trial during filtration with Bayoxide E33, only rising in minute 6 of the trial. The results of water analyses after adsorption on Bayoxide E33 are summarized in Table 5.

The Ibuprofen level dropped after the first 2 min of adsorption on GEH but higher concentrations were measured after minutes 4 and 6 in comparison to the initial condition of the model water. This sudden increase was probably caused by the desorption of GEH, when the material was oversaturated after the first 2 min and stopped eliminating the drug concentration. The pH value slightly increased in the course of the first minute of adsorption and then dropped steadily down to 7.26. The measured temperature

values were similar like in the case of Bayoxide E33, where the temperature increased continuously and decreased in the last two samples. Turbidity dropped in the course of filtration through GEH to 1.11 ZF after the first minute, and increased to 1.58 ZF after the second minute, then was decreasing steadily to the final 1.05 ZF. The results of water analysis after adsorption on GEH are summarized in Table 6.

Desorption is a reverse process to adsorption. It is in fact, the release of the adsorbed substance back into the environment, water in this case. The substance may be released either from the surface or from the volume of the adsorbent. The pH value is especially important for desorption, for it determines adsorption power, for example, of weak acids and alkali on the activated carbon particles. Table 5 shows that in the course of minute 2, the test water temperature, that is, the temperature of the surrounding environment of the adsorbent, increased to its maximum and for the adsorbed substance, that is, Ibuprofen, the concentration decreased to a minimum of 0.15 μ g/L. That was the turning point when desorption started. Desorption may take place into a gaseous or liquid phase. In this case it was the liquid phase [4,18].

Fig. 5 is a graphic representation of the progress of Ibuprofen elimination on the individual adsorption materials. The diagram reveals at first sight that in the case of GEH, desorption started after 2 min, while Filtrasorb F100 and Bayoxide E33 removed Ibuprofen from water after 1 min.

3.2. Effectiveness of sorption materials

The resulting effectiveness of the individual adsorbents used for the elimination of Ibuprofen from water is shown in Table 7. The following formula was used for specification of sorbent effectiveness in the drug removal from the test water [19]:

$$\eta = \frac{C_{RW} - C_F}{C_F} \quad (2)$$

where η is the contamination removal efficiency (%), C_{RW} is the concentration of contamination in raw water (mg/L), and C_F is the concentration of contamination after filtering (mg/L).

Bayoxide E33 was found to be most effective in Ibuprofen elimination from water by the laboratory experiment

Table 4
Analysis after filtration through the sorption material Filtrasorb F100

Filtrasorb F100				
Time (min)	pH (–)	Temperature (°C)	Turbidity (ZF)	Ibuprofen concentration (μ g/L)
0	7.34	16.50	2.26	1.02
1	7.54	22.30	2.71	0.12
2	7.52	21.80	1.14	0.26
4	7.52	20.00	1.14	0.32
6	7.49	19.00	1.14	0.29

Table 5
Analysis after filtration through the sorption material Bayoxide E33

Bayoxide E33				
Time (min)	pH (–)	Temperature (°C)	Turbidity (ZF)	Ibuprofen concentration (μ g/L)
0	7.34	16.50	2.26	1.02
1	7.51	21.60	0.89	0.08
2	7.47	21.60	0.52	0.11
4	7.41	21.30	0.49	0.13
6	7.46	20.60	0.67	0.15

Table 6
Analysis after filtration through the sorption material GEH

GEH				
Time (min)	pH (-)	Temperature (°C)	Turbidity (ZF)	Ibuprofen concentration (µg/L)
0	7.34	16.50	2.26	1.02
1	7.38	21.40	1.11	0.19
2	7.37	21.60	1.58	0.15
4	7.36	21.50	1.16	1.18
6	7.26	20.50	1.05	2.11

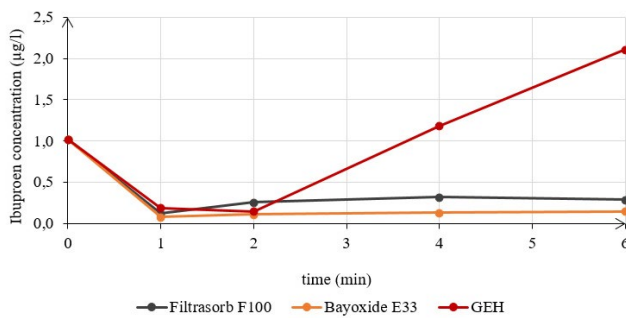


Fig. 5. Removal of Ibuprofen from water by sorption materials.

Table 7
Efficiency of sorption materials on the removal of Ibuprofen

Time (min)	Adsorbents efficiency (%)		
	F100	E33	GEH
0	0.00	0.00	0.00
1	88.24	92.16	81.37
2	74.51	89.22	85.29
4	68.63	87.25	-15.69
6	71.57	85.29	-106.86

performed. Its effect was the strongest of the three tested materials in all test time intervals, always ranging around 90%. Values a little lower than Bayoxide E33 were shown by the activated carbon Filtrasorb F100, reaching a maximum efficiency at 88.24% after the first minute of adsorption. GEH developed desorption after 2 min of the experiment, with negative values of efficiency. A graphic representation of individual adsorbent efficiencies is shown by Fig. 6.

4. Discussion and summary

Due to the considerable consumption of drugs all over the world, their presence in drinking water sources is a current theme. Although the drug concentrations in water sources are low, they can damage aqueous organisms and the environment. That is why we try to eliminate these adverse admixtures from water to drink harmless and healthy drinking water. At present there are numerous technological processes for the removal of micro contaminants

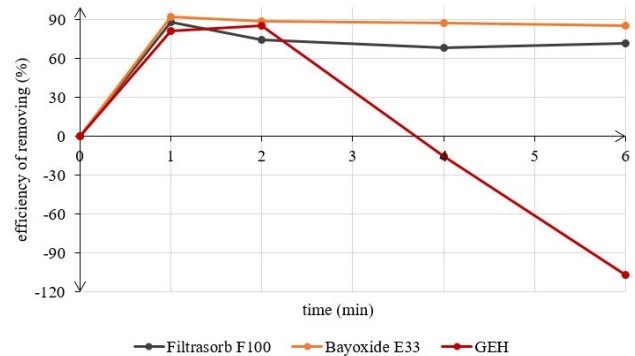


Fig. 6. Efficiency of the sorption materials.

from water, including adsorption. This water treatment process was chosen for the laboratory removal of Ibuprofen from water. Ibuprofen was chosen for the reason of its extensive consumption as a frequent anti-inflammatory drug.

The purpose of the experiment was to compare the efficiency of three adsorbents, the traditional Filtrasorb F100 and another two (Bayoxide E33 and GEH), selected on the basis of the positive results in metal elimination from water.

The best sorption effect in the elimination of Ibuprofen from water in the laboratory setting was shown by Bayoxide E33, whose efficiency in drug elimination reached up to 92.16%. The second best material was Filtrasorb F100, with an Ibuprofen removal efficiency of 88.24%. As adsorption on the GEH material turned to desorption after 2 min of filtration, this material is assessed as inappropriate for drug elimination from water. The pH value ranged in the case of all three materials between 7.26 and 7.54. The progress of pH values was similar in the case of Filtrasorb F100 and GEH sorbents, with a pH increase in the course of the first minute of the experiment followed by a steady decrease. The pH value for treatment with Bayoxide E33 slightly increased in minute 6, unlike the other two sorbents. This experiment showed that the optimal pH for removing Ibuprofen from water was 7.37–7.51. The raw water temperature was lower than the measured temperature values in the course of the adsorption process. The temperature progress was similar in the case of Filtrasorb F100 and Bayoxide E33, with a decreasing trend since the first minute. In the case of GEH, the highest temperature was measured in the course of minute 2 and only then did the temperature began to decrease. This was the material with a turn towards desorption from minute 2, as described above. All the sorbents decreased the initial turbidity value of the model water, with the best result shown by Bayoxide E33, reducing the turbidity value below 1 ZF.

Thus, the laboratory experiment has shown that out of the tested sorption materials Bayoxide E33, the material commonly used for metals removal, especially arsenic, from water, is the best for the elimination of Ibuprofen along with turbidity from treated water. The reduction in turbidity in water also depends on the grain size of the material. When comparing the materials used, Bayoxide E33 and GEH have larger grains than Filtrasorb F100. Bayoxide E33 reduced turbidity to a very low level, but GEH did not reduce turbidity so well. It is possible that desorption of

Ibuprofen from the material also affected turbidity values. According to the measured values, Filtrasorb F100 is also suitable for the elimination of micro pollutants from water. GEH, although suitable for metal removal from water, was ineffective in the elimination of Ibuprofen in our experimental setting. The results of the performed laboratory experiment inspire the consideration of the further investigation of the removal of micro-pollutants from water with the help of various other sorption materials.

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