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Removal of Humic Substances from Water with Granular Activated Carbons [†]

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Abstract: The article deals with the removal of humic substances from surface water in the High Tatras locality and with the reduction in the intensity of water coloration. Four different types of granular activated carbon (Norit 1240, WG12, Filtrasorb TL830, and Filtrasorb 300) were compared in the experiments. The quality of the water from the water source supplied to the filter columns, and the water at the outlet, the filtration speed, and the efficiency of sorption media were monitored. The results showed more than 70% efficiency in the removal of humic substances and COD_{Mn} from water, but only about 50% efficiency from the point of view of water color, as determined with the technology used.

Keywords: drinking water treatment; filtration; granulated activated carbon; humic substances; color of water; adsorption efficiency

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1. Introduction

Humic substances (HS) often constitute the main part of natural organic pollution (NOM) in natural waters. The structure of humic substances is not yet known exactly. These are complex high-molecular organic compounds of an aromatic–aliphatic nature that contain carbon, oxygen, hydrogen, and nitrogen. Humic substances have a relative molecular weight ranging from a few hundred to tens of thousands. The elementary composition of HS is listed in Table 1 [1,2].

Table 1. Elementary composition of humic substances presented in natural waters.

Humic Substances	Elementary Composition (%)			
	C	**O**	**H**	**N**
humic acids	52–62	30–39	2.5–5.8	2.6–5.1
fulvic acids	43–52	42–51	3.3–6.0	1.0–6.0

The highest representation expressed in % has organic carbon in humic substances, representing approximately 50%, and fulvic acids have almost the same content of oxygen as carbon does, while for humic acids, this value is lower by approximately 20%. Humic substances are characterized by the presence of carboxyl and hydroxyl functional groups (phenolic; alcoholic), as well as methoxyl and carbonyl groups. These groups are attached both to the aromatic cores and to the side aliphatic chains [3].

The properties of humic substances have a very close connection with their composition, which is influenced by humification processes, with the size of molecules (molecular weight) and particles, their degree of dispersion (true or colloidal solutions), their polarity, which is determined by the character of the skeleton (aromatic; aliphatic), and mainly, the type, number, and dissociation ability of functional groups. The function of the composition of humic substances is their solubility in water (fulvic acids have higher solubility than humic acids do, and therefore natural waters contain, on average, 87% of fulvic acids), ability to aggregate, ability to dissociate (mainly carboxyl functional groups), and related charge ratios (zeta potential of particles). Some properties of humic substances are also influenced by the composition of the water and its pH. Figure 1 shows basic properties of humic substances [3].

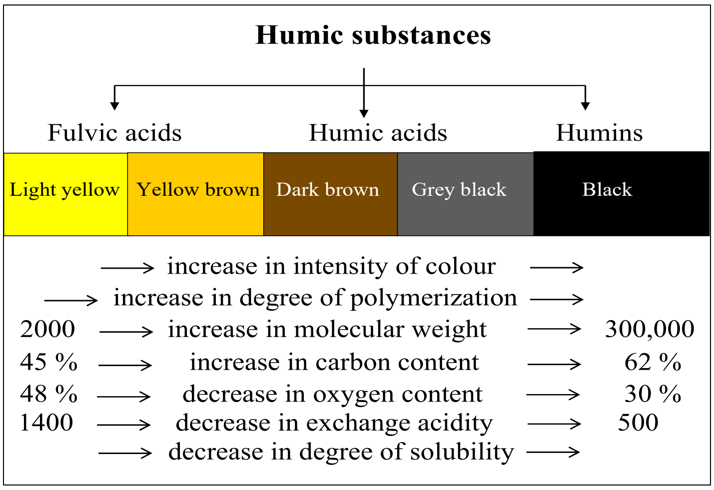

Figure 1. Basic properties of humic substances.

The undesirable effect of humic substances in terms of their influence on water quality and its treatment can be summarized as follows:

- An increase in the intensity of the color of the water;
- An increase the acidity of the water;
- An effect on the smell and taste of water;
- An effect on the formation of metal complexes (e.g., with Fe, Mn, Al, and Cu);
- An effect on the adsorption of organic compounds (e.g., pesticides, PCBs, and phthalates);
- An effect on the formation of trihalomethanes (THMs) during water chlorination [4–6].

The presence of humic substances in waters is manifested by their coloring, e.g., fulvic acids have a light yellow to yellow-brown color depending on the concentration, while humic acids are dark brown. Due to their origins, these are the natural colors of the waters. Usually, a concentration of humic substances of 2.5 $mg \cdot L^{-1}$ corresponds approximately to the water color of 20 $mg \cdot L^{-1}$ Pt. However, the color changes considerably with the pH value of the water (solutions with a higher pH are more colored). In addition, the color of water depends on the composition of humic substances, the size of dispersed particles, etc., which can be different in different surface waters. Therefore, a generally valid linear relationship between the concentration of humic substances and the color of water cannot be obtained [6].

Humic substances are characterized by complex-forming properties [7,8]. The formation of complexes is one of the reasons for the leachability of heavy metals from the soil by humic substances (their content in water increases) and the reason for the increased concentration of Fe and Mn in peat waters.

The content of humic substances in raw water causes a problem in the individual technological stages of water treatment [9,10]; higher doses of coagulant and disinfectants are applied, but mainly, they act as precursors for the formation of halogen compounds.

Important precursors of organohalogene compounds in water are primarily fulvic acids and humic acids. It has been experimentally confirmed that approximately 60% more organochlorine compounds are formed from fulvic acids compared to humic acids [11,12]. At the same time, fulvic acids have higher solubility in water than humic acids do, which is why natural waters contain an average of 87% fulvic acids. To limit the formation of chlorinated hydrocarbons, the content of humic substances in the water must be reduced as much as possible or the method of disinfection must be changed.

In Slovakia, the determination of humic substances in drinking water is not regulated by legislation [13]. It is based on the COD_{Mn} value, absorbance (A^{254}), or color of the water. Exceeding the limit value is the reason for the decision of whether or not to determine humic substances. The limit value for COD_{Mn} is 3.0 $mg \cdot L^{-1}$.

In the older standard [14], humic substances were among the physico-chemical indicators with a limit value of 2.5 $mg \cdot L^{-1}$. Exceeding the value of 2.5 $mg \cdot L^{-1}$ indicates the possible presence of THMs in water supplied with chlorine.

Requirements for the quality of raw water and limit values of water quality indicators for individual categories of standard methods for the treatment of raw water to drinking water are established by decree [15]. This decree contains recommended water treatment procedures for individual categories, A1, A2, and A3. The categories take into account the technological complexity and effectiveness of the treatment.

The requirements for the quality of surface water and the quality objectives of surface water intended for the abstraction of drinking water are determined by the regulation [16]. The assessment of surface water quality and its classification into individual categories is carried out on the basis of selected indicators, which include COD_{Mn} and TOC, turbidity, and water color and absorbance (Table 2).

Table 2. Classification of surface water into individual categories based on selected parameters.

Parameter	**Unit**	**Category A1**		**Category A2**		**Category A3**	
		OH	**MH**	**OH**	**MH**	**OH**	**MH**
COD_{Mn}	$mg \cdot L^{-1}$	2	3	5	7	8	10
TOC	$mg \cdot L^{-1}$	<5.0	5.0	5.0	7.0	8.0	10.0
Turbidity	FNU	2	5	-	30	-	50
Color	$mg \cdot L^{-1}$ Pt	10	20	50	100		
Absorbance (254 nm)		-	0.08	-	0.15	-	0.30

The lowest concentration of humic substances is found in groundwater (up to 0.1 $mg \cdot L^{-1}$). In surface waters, the concentration of them varies from 0.1 to 20 $mg \cdot L^{-1}$ and it is more than that in seawater since the freshwater environment possesses a greater fraction of organic material. In waters from peatlands, the concentration of humic substances fluctuates in a wide range, mostly in tens of $mg \cdot L^{-1}$. In some stagnant waters, it is possible to determine the concentration of humic substances in amounts up to 500 $mg \cdot L^{-1}$. Humic substances can be found in municipal wastewater with a concentration varying from 118 to 228 mg/g, and approximately 42% of these humic substances represent HS [6,17].

1.1. Removal of Humic Substances

Humic substances (HS) can be removed from drinking water via several treatment processes, but different treatment processes can have different efficiencies in terms of their removal and drinking water safety. Coagulation, flocculation, adsorption, oxidation, ion exchange, membrane filtration, and biological and electrochemical processes or advanced oxidation processes (AOPs) can be used to remove HS from water [10,18–21].

Coagulation is one of the most widely used processes for treating water from surface sources. The application of coagulation to remove humic substances from drinking water sources has received much attention from researchers worldwide, because it has been effective in helping to prevent the formation of disinfection byproducts (DBPs). Nevertheless, with the increased fluctuation of humic substances in water (in terms of concentration and composition), the efficiency of conventional coagulation has significantly decreased, and therefore it is necessary to develop improved coagulation processes by optimizing operating conditions, and to develop more effective inorganic or organic coagulants, as well as to combine coagulation with other water treatment technologies [21].

Only high-molecular humic acids are removed from water via coagulation, but in the case of low-molecular fulvic acids (with a relative molecular weight of up to 500), the efficiency is significantly lower, and some fractions cannot be removed at all. The best efficiency is achieved in the treatment of humic waters via clarification in the acidic region (at pH from 4 to 6), when large and easily separable aggregates are formed [22].

Different methods are used to remove humic substances from water; among them is adsorption, which is effective and also, compared to classic methods, simpler in terms of operation and the equipment used—filters with an adsorption bed. This technology is especially suitable for small water treatment plants, or where it is not possible to use conventional water treatment associated with coagulation.

The adsorbent affects the efficiency of the adsorption process. The porous structure and surface area of the adsorbent influences adsorption and the kinetics of adsorption itself. The larger the surface area of the adsorbent, the more efficient the adsorption process. For this reason, we are constantly looking for materials—adsorbents with a large surface area and thus faster kinetics of pollutant removal.

The adsorption of pollutants is also influenced by physico-chemical properties of treated water, such as pH value, the initial concentration of pollutants, the type and size distribution of molecules, and water temperature.

Activated carbon is a versatile adsorbent that can remove diverse types of pollutants such as metal ions, dyes, phenols, and a number of other organic and inorganic compounds and bio-organisms. However, its use is sometimes restricted due to its higher cost. Due to the higher cost of activated carbon, attempts are being made to regenerate spent activated carbon. Chemical as well as thermal regeneration methods are used for this purpose. However, these procedures are not very cheap and also produce additional effluents and result in a considerable loss of the adsorbent. Therefore, in situations where cost factors play a major role, scientists are looking for low-cost adsorbents for the removal of water pollution. A wide variety of materials have been investigated for this purpose and they can be classified into three categories: (i) natural materials, (ii) agricultural wastes, and (iii) industrial wastes. These materials are generally available free of cost or at a low cost as compared to that of activated carbons [23].

The removal of pollutants from waters utilizing biological materials is a relatively recent advancement. It was only in the 1990s that a new technology, biosorption, developed that could also help the removal of heavy metals and other pollutants from waters. Various biosorbents [23–25] have been tested for the removal of pollutants, especially metal ions, with very encouraging results.

1.2. WTP Nový Smokovec

The water treatment plant (WTP) was put into operation in 1972. The capacity of the WTP is 20 $L \cdot s^{-1}$. The water treatment plant treats water from the Štiavnik stream in Nový Smokovec. The stream is also called Červený potok due to the fact that, in certain periods (snow melting, spring periods, long-term rains, and torrential rains), the water is colored red due to the humic substances from the peat soils, and shows an above-limit COD_{Mn} value, low pH (around 6.0), increased water color (up to 30 $mg \cdot L^{-1}$), and also turbidity (max. 5.8 FNU).

Figure 2 shows the COD_{Mn} values in the period from 2012 to 2022.

The water is slightly mineralized water; the total amount of dissolved substances at 105 °C is between 37 and 88 $mg \cdot L^{-1}$, the conductivity is 5.0 $mS.m^{-1}$, the average concentration of calcium is 4.3 $mg \cdot L^{-1}$, the average concentration of magnesium is 1.2 $mg \cdot L^{-1}$, the sum of Ca + Mg is 0.14 $mmol \cdot L^{-1}$, the absorbance is 0.057, $ANC_{4.5}$ is in a concentration of

0.38 mmol·L^{-1}, aggressive CO_2 according to Heyer is in a concentration of 9.5 mg·L^{-1}, the average manganese concentration is 0.055 mg·L^{-1}, the iron concentration is 0.04 mg·L^{-1}, the turbidity is 1.2 FNU, the water color is 10 mg·L^{-1} Pt, the water temperature is between 1.8 and 13.1 °C, and the pH is from 5.9 to 8.7. The indicated values are at the entrance to the water treatment plant.

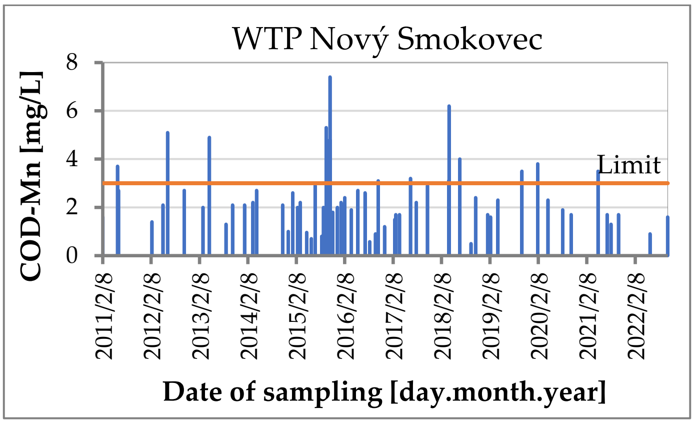

Figure 2. Values of COD_{Mn} in raw water from Štiavnik stream.

The treatment technology consists of a sampling object, sedimentation via flow through a stilling and sedimentation tank, filtration on three open rapid sand filters and deacidification on filters with a PVD filling. Treated water is accumulated in a reservoir. Currently, the water treatment plant is used as an additional water source.

2. Materials and Methods

The experiments were carried out at the Nový Smokovec plant. Two filter columns (with an inner diameter of 5.0 cm, height of 150 cm, and height of filter filling of 90–100 cm) filled with granular activated carbon Norit 1240, WG12, Filtrasorb 300, and Filtrasorb TL830 were used to verify the efficiency of humic substance elimination from water. Their basic characteristics are listed in Table 3.

Table 3. Properties of utilized granulated activated carbon [26–29].

Parameter	Norit 1240	WG12	F300	F830
Iodine number (mg·g^{-1})	min. 1020	min. 1000	min. 1000	min. 900
Methylene blue (mg·g^{-1})	min. 230	min. 30	min. 245	min. 260
Specific surface (BET) (m^2·g^{-1})	1150	min 1000	1050	1100
Particle size (mm)	0.85–2.0	1.0–1.5	0.85–2.0	0.6–2.36
Median diameter of particles (mm)	1.4	1.2	1.4	1.6
Operational density (g·gcm^{-3})	0.480	0.450 ± 30	0.450	0.460
Abrasion (-)	75	85	96	95
Hardness (-)	97	95	75	78
Coefficient of uniformity	1.6	1.3	1.4	2.1
Humidity (wt.%)	max. 2	max. 2	max. 3	max. 2

During the experiments, the quality of raw and treated water at the outlet from the filter columns and the water flow were monitored. The filtration conditions are summarized in Table 4.

For the determination of humic acids (HA), a spectrophotometric method in the visible region (at a wavelength of 420 nm) was employed, which used the extraction of humic substances at a low pH value into pentanol and subsequent re-extraction from pentanol with a NaOH solution [30,31]. To convert absorbance into concentration, we used an empirical coefficient (valid for peat waters in Slovakia).

$$\mathrm{HL}\left(\mathrm{mg}\cdot\mathrm{L}^{-1}\right) = \frac{\mathrm{A} \times 68 \times 250}{250 \times 2.8} \quad (1)$$

where HL is the determined concentration of humic substances, A is absorbance at 420 nm, 250 mL is the volume of water used for extraction, 68 is the empirical coefficient and 2.8 is the width of the cuvette used for analysis (in cm).

Table 4. Filtration conditions.

Parameter	**Norit 1240**	**WG12**	**F300**	**F830**
Height of filtration bed (cm)	92	92	100	100
Weight of the bed (g)	1127.4	907.5	1418.3	1350.8
Avg. flow through the column (mL·min^{-1})	203.14	208.86	205.94	207.94
Avg. filtration velocity (m·h^{-1})	6.207	6.382	6.293	6.354
Bed contact time (min)	8.89	8.65	9.53	9.44

A Hach DR2800 spectrophotometer (Hach Lange GmbH, Düsseldorf, Germany) was used for the analysis of humic substances, water color, and other water quality parameters, while turbidity was determined using the Hach 2100Q instrument (Hach Company, Loveland, CO, USA), and the pH values were determined using Hach SensION+ ph3 Benchtop Meter (Hach Lange GmbH). Chemical oxygen demand was determined using the method of Kübel–Tieman (COD-Mn) in accordance with STN EN ISO 8467:2001 [32].

3. Results and Discussion

Based on the obtained data, we compared the effectiveness of granulated activated carbon (Norit 1240, WG12, Filtrasorb TL830, and Filtrasorb 300) in removing humic substances and COD$_{\mathrm{Mn}}$ from water (Figures 3–5), and at the same time, we also monitored other physico-chemical parameters defined in Decree Ministry of Health SR no. 91/2023 Coll. for drinking water.

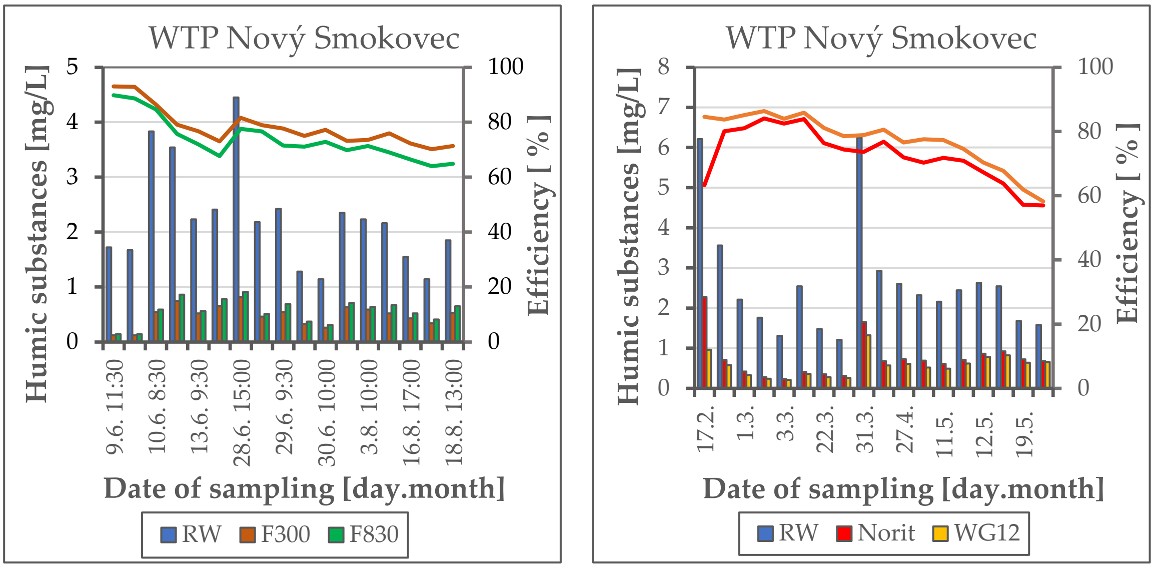

Figure 3. Concentration of humic substances in raw and treated water and the efficiency of sorption materials.

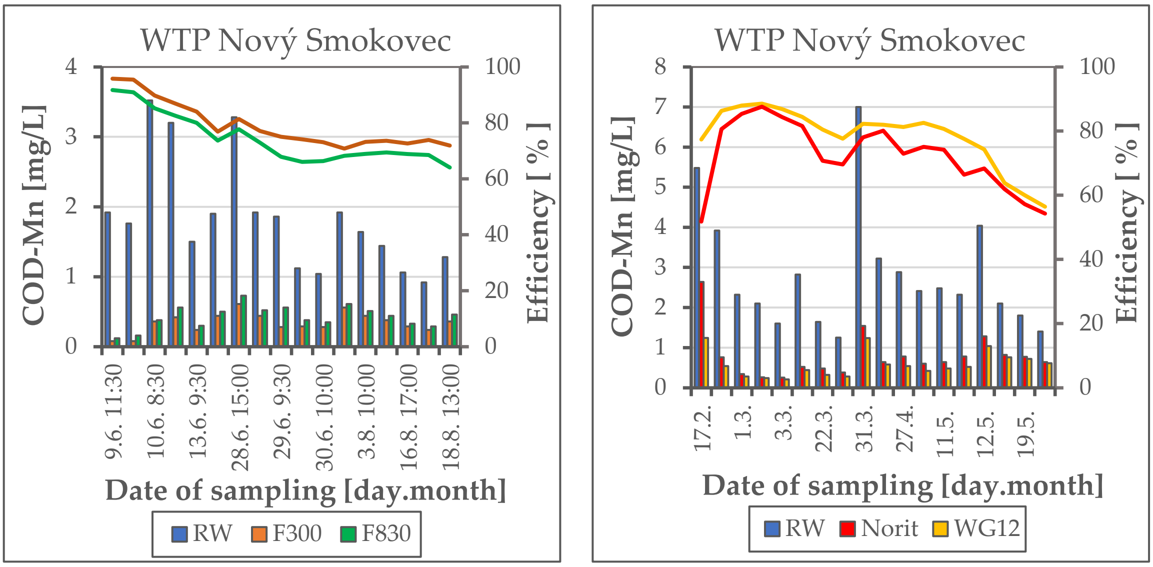

Figure 4. Concentration of COD_{Mn} in raw and treated water, and the efficiency of sorption materials.

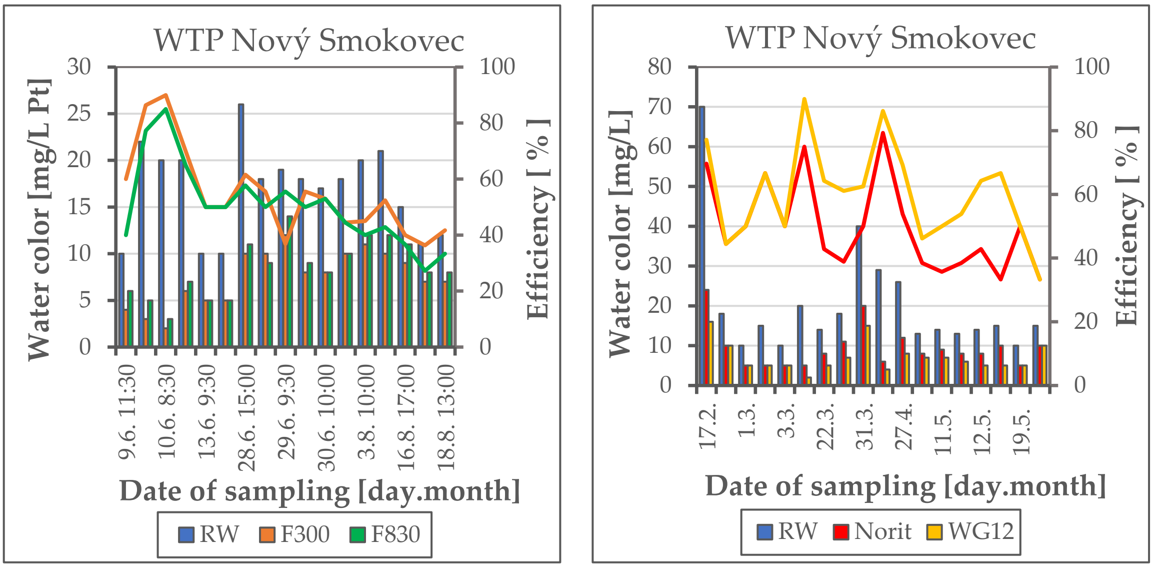

Figure 5. Color of water in raw and treated water, and the efficiency of sorption materials.

4. Conclusions

Humic substances (humic acids; fulvic acids) are created via the decomposition of organic matter and are a common part of natural waters. If they are not removed in the water treatment process and come into contact with disinfectants, the reaction with them leads to the formation of carcinogens and toxic substances. It is necessary to remove HS from the water to prevent the formation of these harmful substances.

Adsorption is a very effective method of removing humic acids from water. Activated carbon, zeolites, modified clays, iron-based sorption materials, materials obtained from various types of waste, and biological adsorbents are used as adsorption materials. Adsorption efficiency is affected by several factors, such as pH, initial HS concentration, type of adsorbent, and contact time.

This article presents results from pilot plant experiments of HS removal via adsorption. Different types of activated carbon (Norit 1240, WG 12, F300, and F830) were used as sorption materials, and the efficiency of the materials used was monitored and compared. The experiments were carried out at the Nový Smokovec plant. The average efficiency of removing humic substances from water was 78.10% in the case of Filtrasorb 300, 74.13% in the case of Filtrasorb TL830, 72.15% in the case of Norit 1240, and 77.37% in the case of WG12. The average water color reduction efficiency using granular activated carbon was 60.32% for WG12, 49.61% for Norit 1240, 54.62% for Filtrasorb 300, and 50.47% for Filtrasorb 830TL. The adsorption capacity of activated carbon was not exhausted (Figure 3).

The experiments were carried out with breaks from February 17 to August 18. The effort was to capture the deteriorated water quality with a pronounced red color and a high content of humic substances, which did not occur during the given period. Therefore, the experiments will continue in 2023.

On the basis of the pilot tests, granular activated carbon will be proposed for the overall modernization of the WTP in Nový Smokovec.

Author Contributions: D.B. and J.S. worked out a concept and plan of experiments. J.I. and D.B. ensured the installation of all equipment, and the assembly and verification of used technologies. D.B., J.I. and J.S. performed all experiments and water sampling. J.I. analyzed water samples (part of the water samples were analyzed in a laboratory of the water company in Poprad). D.B. and A.M. evaluated the obtained results from the experiments. A.M. processed the literature research on the given issue. D.B. and J.I. wrote the paper with the contribution of all co-authors. All authors have read and agreed to the published version of the manuscript.

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