Effectiveness of priority PAH removal in a water coagulation process
Anna Nowacka and Maria Włodarczyk-Makula

ABSTRACT
The paper presents results of research on the removal of priority hazardous polycyclic aromatic hydrocarbons (PAHs) (anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[g,h,i]perylene, indeno[1,2,3-cd]pyrene) from water in the coagulation and sedimentation process. Coagulants chosen for the analysis were aluminum(VI)sulfate and two pre-hydrolyzed coagulants – FLOKOR 1.2A and FLOKOR 1ASW/B. Water samples subjected to coagulation were collected from selected water treatment plants following the pre-ozonation process. Tested coagulants were dosed at optimal doses (1.2; and 1.8 mg Al³⁺/L). The process was carried out at optimum parameters: rapid mixing – 3 min at the rotational speed of 200 rpm; slow mixing – 10 min at 30 rpm; sedimentation – 60 min. Summary concentration of six priority hazardous PAHs in water after pre-ozonation amounted to 12.6 ng/L; after volumetric coagulation using selected coagulants it was in the range 6.8–8.3 ng/L. The highest efficiency in the removal of priority hazardous aromatic hydrocarbons was obtained after application of the preparation FLOKOR 1 ASW/B. Decrease in the summary concentration amounted to 46.4%. For the removal of individual priority PAHs, FLOKOR 1ASW/B was also more effective using lower coagulant dose than was aluminum(VI)sulfate (decrease in the range 23.6%–56.1%).

Key words | coagulation, high-performance liquid chromatography (HPLC), PAHs, pre-hydrolyzed coagulants, priority hazardous substances, water treatment

INTRODUCTION
The first list of priority substances that present significant risk to the aquatic environment was published in Decision No. 2455/2001/EC (Decision 2455/2001/EC). The list identified 33 substances or groups of substances where the major concern was for waters. Directive 2008/105/EC of the European Parliament and of the Council on environmental quality established environmental quality standards in the field of water policy (Directive 2008/105/EC) and replaced five older directives. In this Directive allowable concentrations (μg/L) of 33 priority substances and certain (eight) other pollutants are specified. The priority substances include eight compounds from the group of polycyclic aromatic hydrocarbons (PAHs): naphthalene, anthracene, fluoranthene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[g,h,i]perylene and indeno[1,2,3-cd]pyrene. In addition, some of these substances have been recognized as priority hazardous substances, including six PAHs (those mentioned above, with the exception of naphthalene and anthracene). Concentrations of benzo[a]pyrene in inland and other surface waters are not permitted to exceed 0.1 μg/L. Directive 98/83/EC of the Council introduces quality standards for water intended for human consumption (Directive 98/83/EC). Five compounds belonging to the group of PAHs were listed in this Directive, and the allowable concentration of benzo[a]pyrene, amounting to 0.01 μg/L, was specified. The total concentration of four PAHs, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[g,h,i]perylene and indeno[1,2,3-cd]pyrene, at the level of 0.10 μg/L. PAHs whose allowable concentrations are specified in Directive 98/83/EC are classified as
priority hazardous substances. For this reason, it is very important to monitor the concentration of these compounds in water treatment processes.

Owing to their toxic, mutagenic and carcinogenic properties, PAHs are among the primary environmental pollutants. PAHs are easily adsorbable compounds, as evidenced by their high octanol/water partition coefficient (Wlodarczyk-Makula 2013). The occurrence of these compounds in groundwater and surface water has been confirmed in studies by many authors (Xia et al. 2006; Masih & Lal 2014). Despite the fact that they are classified as persistent and difficult degradable pollutants, PAHs are subject to constant physicochemical and biological changes. PAHs in water treatment processes are removed during filtration (adsorption by the filter material) and chemical disinfection (with chlorine or chlorine dioxide). Another type of separation of PAHs from water is sorption by solid particulates (in the case of coagulation – by flocs). This sorption does not in itself lead to the destruction of PAHs, however, it can support their removal (the compounds adsorbed by flocs can later be leached out of the filter material after the filtration process). Removal efficiency of PAHs from water during coagulation, filtration and chemical disinfection was investigated in a study by the current authors. In case of the concentration of four PAHs, reduction by 61.9% and 21.0% was obtained after volumetric coagulation and coagulation using pulsators, respectively. After filtration on sand filters, the concentration of the four PAHs was lower by 53.1%, while after chlorine disinfection it was lower by 38.3% (Nowacka & Wlodarczyk-Makula 2013). The advantages of coagulation, filtration and chemical disinfection may include lower costs than in advanced water treatment methods (for example membrane processes). The disadvantages of each of mentioned methods may include insufficient efficiency. In comparison, in case of sorption by activated carbon WG-12, the effectiveness of the removal of four PAHs from model aqueous solutions was 96.9% (Smol et al. 2014). Other authors have studied the removal efficiency of PAHs (naphthalene, acenaphthene, phenanthrene, fluoranthene, anthracene and pyrene) from water by adsorption on nano- and organo-modified nano-clays followed by coagulation-flocculation. It was found that the modified treatment process (using alum + PAC + ODAAPS-M-clay) was the most effective method with removal in the range of 97.7%–100.0% (Shabeer et al. 2014).

Properly conducted coagulation ensures the effective elimination of water turbidity and its accompanying color. Organic impurities are also removed. With appropriate selection of process parameters, heavy metals may also be removed during coagulation (Dałbowska & Rosińska 2013). The most frequently used coagulant in water treatment plants (WTPs) to date has been aluminum(VI)sulfate, which, however, is currently being replaced by pre-hydrolyzed coagulants (Duan & Gregory 2003; Pernitsky & Edzwald 2006; Nowacka et al. 2014). Changing coagulants is aimed at optimizing coagulation and minimizing adverse effects of the process. In accordance with the data in the literature (Sinha et al. 2004; Zhanmeng et al. 2013), and as confirmed by a study by the current authors (Nowacka et al. 2014), these reagents are more effective in the removal of substances affecting the turbidity of water and organic impurities. Moreover, they are more resistant to changes in temperature and pH (Gregory & Dupont 2001). Research on the exchange of coagulants is concentrated on increasing the effect of removing the pollutants mentioned above. However, their potential efficiency in the removal of PAHs has not been studied. Given that PAHs are present in surface waters and are toxic to humans, one important issue is to clarify the extent to which they are removed in the process of coagulation.

The aim of the present research was to determine the effectiveness of selected aluminum coagulants: aluminum-(VI)sulfate and two pre-hydrolyzed coagulants: FLOKOR 1.2A and FLOKOR 1ASW/B, in the removal of priority hazardous PAHs (anthracene – Ant, benzo[a]pyrene – BaP, benzo[b]fluoranthene – BbF, benzo[k]fluoranthene – BkF, benzo[g,h,i]perylene – BgP, indeno[1,2,3-cd]pyrene – IpC) from water in the coagulation process.

**MATERIALS AND METHODS**

**Water used for coagulation**

Water for the coagulation process was collected once (i.e. a single collection on one day) from a selected WTP in southern Poland following the pre-ozonation process. Instantaneous samples were taken in autumn. The analysed
samples were grab samples. Water samples intended for the studies were collected from waterworks taps prior to coagulation (after pre-ozonation). Samples were stored at +4 °C.

Selected indicators of water quality used for coagulation were as follows (average values):
- temperature: 18.2 °C,
- pH: 6.35,
- alkalinity: 1.60 mval/L,
- true color: 5 mg Pt/L,
- turbidity: 5.95 NTU,
- absorbance $UV_{254}$ (filtered samples): 5.9 m$^{-1}$,
- dissolved organic carbon (DOC): 5.58 mg C/L,
- total organic carbon (TOC): 6.44 mg C/L,
- specific UV absorbance (SUVA at 254 nm): 1.06 m$^3$/gC·m,
- aluminum: 0.085 mg Al/L.

**Coagulants**

Three coagulants were used in the study:
- $\text{Al}_2(\text{SO}_4)_3 \times 14\text{H}_2\text{O}$ – aluminum(VI)sulfate, in solid form,
- two pre-hydrolyzed coagulants:
  - dialuminium chloride pentahydroxide – FLOKOR 1.2A,
  - dialuminium chloride hydroxide sulfate – FLOKOR 1ASW/B.

The characteristics of the tested coagulants are presented in Table 1. These data come from the safety data sheets of the manufacturers (referring to commercial preparations).

To ensure easier application, 1% solutions of the tested coagulants were prepared.

### Experimental procedure for coagulation

On a laboratory scale, the study was carried out with the use of a six-beaker floculator. The coagulant was introduced to the water samples (volume: 2 L) as a 1% solution at the optimum doses. The optimum doses of the coagulants and the optimum process parameters were determined in the present authors’ previous studies, on the basis of indicators such as color, turbidity and $UV_{254}$ absorbance. Results of these studies (samples collected in autumn) have not been yet published. However, there are publications by the current authors concerning the determination of optimum doses of selected coagulants in winter (Nowacka et al. 2014). In autumn, the optimum doses of the coagulants used amounted to:
- aluminum(VI)sulfate: 1.8 mg Al$^{3+}$/L,
- pre-hydrolyzed aluminum coagulants: 1.2 mg Al$^{3+}$/L.

Appropriate doses of coagulants were added to a beaker and were quick-mixed with a mechanical stirrer for 3 min (at 200 rpm) followed by slow mixing for 10 min (at 30 rpm). Afterward, the samples were subjected to sedimentation for 60 min. Following sedimentation, 1.2 L of water was decanted for analysis of the content of selected priority hazardous PAHs.

All coagulation tests were performed at ambient temperature, without pH correction (results of previous studies have shown that correction of the pH to 5.50 and 7.50 does not improve the efficiency of the process).

### Analytical methods

Determination of pH was accomplished using the potentiometric method. A titrimetric method was used for determination of the total alkalinity of water samples. Their true color was identified in accordance with ISO 7887 – Method C (ISO 7887 2011), using a CamSpec Ltd. M501 spectrophotometer. A quartz cuvette with an optical path length of 50 mm was used, as determined after filtration of the water sample through a membrane filter with a pore-size 0.45 μm. Turbidity was measured with a Lovibond turbidity meter using the nephelometric method.

Determination of $UV_{254}$ absorbance was performed in accordance with the method used by the US EPA (Poter
using a Camspec Ltd. M501 spectrophotometer. A quartz cuvette with an optical path length of 10 mm was used. For DOC analysis, the sample was filtered through a membrane filter. DOC and TOC concentrations were determined using an Analytik Jena TN/TC model multi N/C carbon analyzer. DOC and UV\textsubscript{254} absorbance were used in the calculation of SUVA at 254 nm (Poter 2005). Aluminum concentrations in water samples were determined using the method of inductively coupled plasma-atomic emission spectrometry with a Perkin–Elmer Optima 8000 spectrometer. Determinations of all indicators selected for analysis were performed in three replications.

**Analysis of PAHs**

In this study, the content of the six PAHs known as priority hazardous substances was investigated. The concentrations of selected PAHs in water samples before and after coagulation were determined through high-performance liquid chromatography with a fluorescence detector (HPLC-FTD). For the research, 500 mL of a water sample before and after the coagulation process (using various coagulants) was collected. A quantity of 87.5 mL of 2-propanol (17.5 mL per 100 mL of the sample) was added to the sample. To isolate extracted analytes from other organic substances extracted at the same time, a C18 extraction column was used (solid phase extraction or SPE). To prepare it for use, 6 mL of hexane was slowly passed through the column, then the column was dried under vacuum for 2 min. Prior to introduction of the extracts, the column was conditioned with methanol (6 mL) and HPLC water (6 mL). The test sample was passed through the column, maintaining the vacuum and aspiration rate of 10 mL/min. The sample was then dried under vacuum for 30 min and the PAHs were eluted with hexane (3×1 mL). The resulting eluate was gently evaporated until dry in a stream of nitrogen. An acetonitrile in the amount of 1 mL was then added. The prepared sample was analyzed by HPLC. Indications were performed on a liquid chromatograph (Waters Alliance 2,695 with a Supelcosil LC-PAH column, 15 cm × 4.6 mm × 5 mm). The identification of PAHs was performed in duplicate. The successive stages of the preparation of samples for the identification of PAHs are presented in the diagram in Figure 1.

To verify the adopted procedure, values of recovery for priority PAHs were designated. For this purpose, a standard mixture of 16 PAHs (Restek PAHs Mix) was introduced into the sample of distilled water. Then, quantitative and qualitative determinations of the PAHs were carried out in accordance with the procedure described above. The recovery rate ranged from 48.3% (indeno[1,2,3-cd]pyrene) to 73.1% (benzo[b]fluoranthene). Recovery rates for other individual PAHs amounted to 67.3% for anthracene, 58.9% for benzo[a]pyrene, 67.4% for benzo[k]fluoranthene and 50.3% for benzo[g,h,i]perylene. Obtained recovery values are similar to rates achieved in studies by other authors (Kabziński et al. 2002). Calculations of concentration take these recovery rates into account.

**Statistical analysis**

Results concerning changes in the concentration of individual PAHs in water after coagulation were statistically analyzed. Concentrations of PAHs were compared on the basis of the identification of these compounds in water following coagulation with optimal doses of various coagulants and optimal process parameters. The significance of the coagulants used was evaluated based on Student’s t-test for the difference of two independent trials. The calculations were performed using the STATISTICA program (StatSoft). The number of results in trials was the same. The critical values for four degrees of freedom (n = 3) and confidence level (95%) were adopted using tables. The value from tables of Student’s t-distribution is \(t_\text{i} = 2.776\).

**RESULTS AND DISCUSSION**

**Changes in the value of selected indicators of water quality after the coagulation process**

The values of analyzed indicators after the coagulation process using optimal doses of the selected coagulants (aluminum(VI)sulfate, FLOKOR 1,2A and FLOKOR 1ASW/B) along with optimal process parameters are presented in Table 2. The use of pre-hydrolyzed coagulants resulted in lower consumption of water alkalinity than in the case of ALK. These results were similar to those
obtained in earlier studies (in the winter) by the present authors (Nowacka et al. 2014) and by other authors (Krupińska 2014). In all analysed cases, the same results were obtained in terms of the removal of colored compounds (a reduction of 80.0%). The highest efficiency in the removal of compounds affecting turbidity was obtained for FLOKOR 1ASW/B (67.2%). This coagulant was proven to be effective in removing turbidity in winter as well. The least effective was FLOKOR 1.2A (reduction of 55.5%).

The highest efficiency in the removal of organic impurities, expressed using the UV$_{254}$ absorbance indicator (filtered samples), was obtained with the application of FLOKOR 1ASW/B (27.1%). The lowest was observed with application of the preparation FLOKOR 1.2A (15.3%). The best results in reducing the concentration of DOC (24.9%)
were obtained following coagulation using FLOKOR 1ASW/B. For the other two coagulants, the removal efficiency of organic substances, expressed using a DOC indicator, amounted to 17.2%–20.9%. The lowest value of SUVA was recorded for the FLOKOR 1ASW/B optimal dose. The results obtained for ALK, FLOKOR 1.2A and FLOKOR 1ASW/B have been confirmed in the literature. Molczan et al. (2006) demonstrate that the removal of DOC in the coagulation process does not exceed 25% if the value of SUVA at 254 nm is less than or equal to 2.0 m³/g·Cm. The lowest concentration of residual aluminum in the water samples was achieved as a result of the coagulation process using FLOKOR 1ASW/B. This concentration was about 10.8% and 23.4% lower than in the cases of ALK and FLOKOR 1.2A, respectively. However, for all coagulants, the concentration of aluminum after the coagulation process increased significantly in comparison with raw water. Aluminum was identified in unfiltered samples. Concentrations after filtration were much lower. Analysis of the results leads to the conclusion that the highest effectiveness of the coagulation process in autumn was obtained after the application of FLOKOR 1ASW/B.

Changes in concentrations of priority hazardous PAHs after the coagulation process

Figure 2(a) presents the results of qualitative–quantitative determination of particular priority hazardous PAHs in water following pre-ozonation. The combined concentration of six PAHs amounted to 26.5 ng/L. Quantitative investigations showed that benzo[b]fluoranthene was dominant in water intended for the coagulation process. The lowest concentrations were observed in the cases of anthracene and benzo[k]fluoranthene. In the case of benzo[a]pyrene (BaP), a very important compound in the PAH group, the concentration before the coagulation process amounted to 2.12 ng/L. BaP was also identified in water intended for human consumption.

Concentrations of PAHs in water samples measured after the coagulation process using the optimal dose of aluminum(VI)sulfate, FLOKOR 1.2A, FLOKOR 1ASW/B and the optimum processing conditions are shown in Figure 2(b)–2(d). Following the process using ALK, a decrease was noted in the concentrations of all analyzed PAHs. Three-ring anthracene concentration in the water samples was below the detection limit. The contents of five-ring hydrocarbons (20.9%–31.3%) and six-ring PAHs (33.0%–38.6%) also decreased following the use of ALK. The dominant hydrocarbon in the water samples after the coagulation process using the formulation FLOKOR 1.2A was benzo[b]fluoranthene. In this case as well, no anthracene was present in the water after coagulation. For five- and six-ring hydrocarbons, removal efficiency within respective ranges of 19.9%–31.9% and 24.9%–26.0% was obtained. As in the case of other coagulants, following application of the FLOKOR 1 ASW/B formulation, no anthracene content was observed. For five-ring PAHs, decreases in concentration in the range of 23.6%–38.1% were recorded. Following the application of this coagulant, the highest effectiveness among all the tested formulations in the removal of six-ring hydrocarbons was obtained, in the range of 43.9%–56.1%.

In all examined cases, the coagulant FLOKOR 1ASW/B was the most effective in the removal of priority hazardous PAHs from water following coagulation. Use of the preparation FLOKOR 1.2A did not produce the expected results in the removal of priority PAHs. Possibly the optimal dose, determined based on the values of true color, turbidity and UV254 absorbance, was not adequate to remove the analyzed PAHs. It will be necessary to conduct additional studies with different doses to determine the suitability of this coagulant for removing priority hazardous aromatic hydrocarbons. The lowest summary concentration of six PAHs was observed following the application of FLOKOR 1ASW/B (a decrease of 46.4%). For the other two coagulants, this value was in the range of 34.5%–39.5%.

The most effective coagulant in the removal of selected PAHs from the water in autumn was FLOKOR 1ASW/B. The greater efficiency of this aluminum pre-hydrolyzed formulation might be influenced by its increased (but not too high) alkalinity and appropriate content of aluminum and chlorine (the lowest among the coagulants in this group and the smallest unit mass of Al/Cl). The high effectiveness of FLOKOR 1ASW/B in the removal of selected priority PAHs may also be related to the structure and properties of the resulting flocs following coagulation with this coagulant. The highest efficiency in the case of FLOKOR 1ASW/B was obtained for six-ring PAHs. This may be related to the
Figure 2 | Average concentration of priority hazardous PAHs in water samples (a) before coagulation, and after coagulation using (b) aluminum(III)sulfate, (c) FLOKOR 1.2A and (d) FLOKOR 1ASW/B.
high value of the octanol/water coefficient, which indicates a strong capacity to sorb these hydrocarbons on the surface of the flocs, and to their molecular structure. Changes in concentrations of the PAHs may result from physicochemical transformations of these compounds in water, especially sorption by solid particles.

Comparison of the results obtained with data in the literature is difficult, because other studies on the removal of priority PAHs from water were not carried out during the process of coagulation. The available studies on the removal of these compounds concern other processes such as membrane processes (Kabsch-Korbutowicz 2010) or advanced methods of oxidation of organic contaminants. However, these are mainly related to wastewater and sludge, not treated water. The effectiveness of activated carbon in the removal of PAHs from water according to reports by other authors ranges from 62.0% to 100%. The process, however, must be preceded by the removal of the PAHs’ carriers, which can be achieved during coagulation, filtration and sedimentation. Removal of PAHs in the course of individual water treatment processes has been investigated in other studies by the present authors (Nowacka & Wlodarczyk-Makuła 2013). Water samples for analysis were collected in winter following subsequent stages of treatment (from the WTP described above). In these studies, the concentrations of 16 PAHs were determined. Higher efficiency in the removal of PAHs was obtained after pre-ozonation and classical coagulation (four PAHs: 60%, 16 PAHs: 47%) than after pre-ozonation and contact coagulation with the use of pulsators (four PAHs: 21%, 16 PAHs: 26%). The highest degree of removal of benzo[a]pyrene was obtained after pre-ozonation and the classical coagulation process (59%). In the process of coagulation using pulsators, a decrease of 38% was achieved. In individual water treatment processes, fluctuations in the concentrations of 16 PAHs (39–204 ng/L) were observed. Reduced values were recorded after pre-ozonation and coagulation, and sedimentation processes, among other factors (Nowacka & Wlodarczyk-Makuła 2013).

Statistical analysis

Changes in concentrations of selected PAHs such as anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[g,h,i]perylene and indeno[1,2,3-cd]pyrene were compared by analysing their concentrations following the coagulation process using optimal doses of two different coagulants. Aluminum(VI)sulfate with FLOKOR 1.2A, aluminum(VI)sulfate with FLOKOR 1ASW/B, and FLOKOR 1.2A with FLOKOR 1ASW/B were compared. Table 3 shows the values of Student’s t-test which determined the significance of different coagulants in the same process. The resulting statistical analysis showed that differences in tested coagulants were not statistically significant in the cases of:

- ALK vs. FLOKOR 1.2A: benzo[b]fluoranthene, benzo[k]fluoranthene,
- ALK vs. FLOKOR 1ASW/B: benzo[b]fluoranthene, benzo[k]fluoranthene,
- FLOKOR 1.2A vs. FLOKOR 1ASW/B: benzo[b]fluoranthene.

In these cases, the calculated values were lower than the theoretical values ($t_d < t_t$). In other cases it was found that the use of different types of coagulants is statistically significant for the achieved effects of coagulation (removing priority hazardous PAHs). Calculated values were higher than theoretical values ($t_d > t_t$).

**CONCLUSION**

The research performed on the effectiveness of non-hydrolyzed aluminum(VI)sulfate and the pre-hydrolyzed aluminum

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<thead>
<tr>
<th>PAH</th>
<th>Value of Student’s t-distribution ($t_d$)</th>
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<tr>
<td>Anthracene</td>
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<tr>
<td>Benzo[a]pyrene</td>
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<tr>
<td>Benzo[b]fluoranthene</td>
<td>1.796</td>
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<td>Benzo[k]fluoranthene</td>
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<td>Benzo[g,h,i]perylene</td>
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<tr>
<td>Indeno[1,2,3-cd]pyrene</td>
<td>4.950</td>
</tr>
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Table 3 | The values of Student’s t-distribution ($t_d$) with $t_t = 2.776$.
coagulants FLOKOR 1.2A and FLOKOR 1ASW/B led to the following conclusions:

(1) The highest efficiency in the removal of priority hazardous aromatic hydrocarbons was obtained after application of the preparation FLOKOR 1 ASW/B. The decrease in the combined concentration amounted to 46.4%. From the point of view of the removal of PAHs from water, it would be advantageous to exchange the aluminum(VI)-sulfate currently used in favor of FLOKOR 1ASW/B.

(2) The greatest effectiveness in the removal of individual priority PAHs was obtained with the application of coagulant FLOKOR 1ASW/B (using a lower coagulant dose than in the case of ALK), as evidenced by the following reductions in concentrations:
- benzo[a]pyrene: 35.4%,
- benzo[b]fluoranthene: 23.6%,
- benzo[k]fluoranthene: 58.1%,
- benzo[g,h,i]perylene: 56.1%,
- indeno[1,2,3-cd]pyrene: 43.9%.

(3) The analysis of the general indicators of water quality indicates the highest efficiency for the coagulation process following the application of the coagulant FLOKOR 1ASW/B (the lowest amount of alkalinity, turbidity reduction of 67.2%, and the lowest content of organic compounds as expressed by the indicators UV$_{254}$ absorbance, DOC and TOC).

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