



## Effectiveness of coagulation, sorption, and ion exchange processes in reduction of selected priority substances from surface waters

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### ABSTRACT

The aim of this work is to analyze micropollutants (MP) removal efficiency in coagulation, adsorption, and ion exchange processes. Research objects are two natural surface waters: Mała Panew River and Dobromierz Lake. Additionally spiked demineralized water is also being analyzed. Analyzed MPs are: anthracene, alachlor, heptachlor, and its epoxide and 4-nonylphenol. As coagulants aluminum sulfate (Alum), polyaluminum chloride (PAC), and polyaluminum chloride hydroxide (PACl) are used. Three flocculants are analyzed: Praestol™ 2540TR (anionic), Praestol™ 655BC (cationic), and Praestol™ 2500TR (nonionic). Sorption processes are carried out on GAC830 active carbon, as well as Lewatit S5428 and Lewatit TP207 resins. Both, coagulation and sorption processes are highly influenced by matrix organic load. Rising organic concentration positively affected MPs removal in coagulation process but decreases effectiveness of sorption processes. Coagulation process allowed to reach maximum 78% reduction as is in case of Heptachlor. Using organic and inorganic anionic sorbents allows to reach 100% MPs reduction.

*Keywords:* Coagulation; Sorption; Ion exchange; Micropollutants; Surface water

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### 1. Introduction

Priority substances are nothing else than organic and inorganic micropollutants, that are under special supervision due to The Water Framework Directive 2000/60/EC and its derivatives [1,2]. Actual priority substances list identifies 45 compounds and groups of compounds, of which 21 are described as hazardous. In major degree, these compounds have their origin in various areas of human activities. In particular, pesticides, pharmaceuticals, and industrial or household chemicals may be distinguished. Their occurrence in aquatic environment is the result of still insufficient awareness of threats and potential consequences resulting from the introduction of micropollutants into the ecosystem, and hence – the use of ineffective water and wastewater treatment techniques.

Micropollutants and its derivatives, due to its high stability, tend to accumulate in aquatic ecosystem, which in case of low-efficient water and wastewater treatment may negatively affect future water quality. The studies carried out so far in Poland, have shown the presence of compounds appearing on the priority substances list, mainly in surface waters. The results of monitoring carried out by Chief Inspectorate for Environmental Protection, as well as those published on behalf of The Regional Water Management Authorities, conducted from 2007 to 2017, show the presence of compounds from the priority substances list. In case of 23 out of 45 positions, a concentration that have exceeded permissible limit values included in the Regulation of the Minister of Environment of July 21, 2016 has been shown. What is important, the reported studies do not include a

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monitoring analysis of the concentration of 12 substances and group of substances introduced along with the amendment 2013/39/EU, that is, dicofol, PFOS, quinoxifen, dioxins, aclonifen, bifenox, cybutryn, cypermethrin, dichlorvos, hexabromocyclododecane, heptachlor, and its epoxide and terbutryn. Therefore, it can be concluded that exceedances were found in 23 out of 33 substances analyzed. These are listed in Table 1.

The nature of the substance itself does not facilitate the challenges associated with the reduction of micro-pollutants. Depending on the physico-chemical properties and other factors, these compounds are removed to varying degrees, with greater or lesser effectiveness. Hydrophilicity and hydrophobicity, acidity, biodegradability, volatility, chemical structure of the substance, as well as the characteristics of the medium being purified (pH, temperature, alkalinity, and natural organic matter (NOM) load) [6,7] determine the effectiveness of micro-pollutants removal. This variety makes it impossible to choose a universal technique to reduce these compounds. Research has been conducted for years to select the most effective technologies. The most popular are: sorption processes [8–10], membrane filtration as well as bio- and photo-degradation [6,11–16]. Studies already made mainly concern advanced oxidation processes (AOPs). Due to its ability to micropollutants degradation, not only phase-to-phase transfer, AOPs are considered as most effective and efficient. However, in practice, these techniques are mostly used in industrial sewage treatment plants. In the case of water treatment plants, especially those where the target recipient are households, most of the technologies listed above are still niche. In the vast majority, a technological process consists of the following: volume coagulation, flock separation on sand beds, sorption using activated carbon, and disinfection. Technology modernization is often impossible due to budgetary reasons or limited due to existing infrastructure, therefore it seems beneficial to invest in the existing system.

Research [17–19] showed that among the processes typical for water treatment plants, both sorption with the use of activated carbon and coagulation are the most effective in the context of micropollutants reduction. However, sorption on activated carbon is limited by the pore size, which means that some micropollutants adsorb with difficulty [20]. In addition, the efficiency of the process is negatively affected by the co-existence of high-molecular components of NOM that block pores and active sites, limiting the efficiency of the process [21,22]. An additional factor is the laborious process of coal bed regeneration, requiring the replacement of the entire bed volume. In the case of the coagulation process, the research [23–25] showed high process selectivity, which is why the evaluation of the process in a general context is impossible. Micropollutants removal mechanism during the coagulation process is based on adsorption, both on the colloids and on generated flocculates surfaces [26]. Substances sorption potential can be expressed using the  $\log P_{o/w}$  partition coefficient. Rogers [27] suggests a division into:

- |                                    |                             |
|------------------------------------|-----------------------------|
| • $\log P_{o/w} < 2.5$             | low sorption potential      |
| • $\log P_{o/w} > 2.5$ and $< 4.0$ | moderate sorption potential |
| • $\log P_{o/w} > 4.0$             | high sorption potential     |

Thuy et al. [28] confirm a possibility of micropollutants reduction effectiveness prediction in the coagulation process on the basis  $\log P_{o/w}$  value knowledge.

Design of this study was motivated by the current need to enhance conventional water treatment techniques effectiveness in micropollutants (MPs) removal. In the existing literature, there is a lack of studies focused on coagulation conditions, such as coagulants and flocculants type, and its impact on MPs reduction. Also, when analyzed, coagulation and sorption effectiveness are evaluated in separated processes, which does not reflect total efficiency of these two processes. Therefore, the aim of the conducted study was to evaluate the effectiveness of coagulation and sorption processes in the reduction of five selected priority MPs. Three types of aluminum coagulants were used in the coagulation process: aluminum sulfate (Alum), polyaluminum chloride (PAC), and polyaluminum chloride hydroxide (PACl). The effect of the coagulant dose and the addition of polyelectrolyte on the efficiency of the process was also investigated. In the case of sorption processes, the effectiveness of sorption on a granulated active carbon bed, as well as anionic and cationic ion exchange resin was compared. In order to determine the impact of the matrix, the study was carried out both on natural waters and water prepared on the basis of deionized water.

## 2. Materials and methods

### 2.1. Target compounds

The analyzed micro-contaminants were selected for the sake of the class of the compound, structural differences, and the partition coefficient value variances ( $\log P_{o/w}$ ). Five MPs, which are classified as priority substances were selected: anthracene (Ant), alachlor (Acl), heptachlor (Hcl), its epoxide (Hcl-e), and 4-nonylphenol (Npl). Structural formulas and  $\log P_{o/w}$  values are shown in Table 2.

### 2.2. Matrix

Study subjects were two natural surface water and one synthetic water. The quality of natural waters was characterized by the content of NOM. The following parameters (Table 3) were tested in these waters: pH, total organic carbon (TOC), dissolved organic carbon (DOC), particulate organic carbon (POC), absorbance in 254 nm ( $UV_{254}$ ) and specific ultraviolet absorbance ( $SUVA_{254}$ ). To emphasize the matrix impact on MPs removal efficiency, synthetic water was prepared. This one was simply deionized water spiked with MPs. One of the surface waters was taken from Mała Panew River in Czarnowąs water intake. This one characterized by a high load of NOM (Table 3) with 23% POC share in TOC. The second one had a much lower organic concentration (Table 3) and was collected from Dobromierz Lake in Świebodzice. In the water samples from Dobromierz Lake, the POC content was only 3% of TOC.

### 2.3. Coagulants and flocculants

Technical grade aluminum sulfate ( $Al_2(SO_4)_3 \cdot 16H_2O$ ) was used as Alum. To conduct zeta potential analysis and coagulation process 0.16 mol L<sup>-1</sup> solution was prepared.

As PAC PAX-18 was used. As pre-hydrolyzed, high basic coagulant Flokor 1,2A (PACl) was used. PAX-18 and Flokor® (Technical Service Enterprise DEMPOL-ECO, Poland) coagulants are the trade name of ready-to-use aqueous solution of specific aluminum salts. Coagulants characteristics were determined using EN 1302:1999/AC:2002 standard [29]. The results are shown in Table 4.

Flocculants used in this study were organic polyelectrolytes based on polyacrylamide. Praestol™ 2540TR (A)

was used as anionic polyelectrolyte, Praestol™ 655BC (C) as cationic and Praestol™ 2500TR (N) as nonionic. Flocculants were used in form of 0.1% solution.

#### 2.4. Filter media

For sorption process, three filter media were chosen. The selected ones comprised two resins (anionic and cationic) and one granulated active carbon. Lewatit® S5428 is

Table 1

Priority and dangerous priority substances, for which exceedances of the permissible concentration limits have been found in years 2007–2017 [3–5]

Number	Priority substance	Identified as priority hazardous substance
1	Benzo(g,h,i)perylene	X
	Indeno(1,2,3-cd)pyrene	
	Benzo(a)pyrene	
	Benzo(k)fluoranthene	
	Benzo(b)fluoranthene	
2	Anthracene	X
3	Fluoranthene	–
4	Choroalkanes, C <sub>10</sub> –C <sub>13</sub>	X
5	Dichloromethane	–
6	Nonylphenols (p-nonylphenol)	X
7	Oktylphenol (4-(1,1',3,3'-tetramethylbutyl)-phenol)	–
8	Pentabromodiphenylether	X
9	Tributyltin compounds	X
10	Trichloromethane (chloroform)	X
11	Di(2-ethylhexyl)phthalate (DEHP)	X
12	Hexachlorocyclohexane	X
13	Pentachlorobenzene	X
14	Pentachlorophenol (PCP)	–
15	Atrazine	–
16	Chloropyrifos	–
17	Chlorfenvinphos	–
18	Diuron	–
19	Endosulfan	X
20	Mercury and its compounds	X
21	Cadmium and its compounds	X
22	Lead and its compounds	–
23	Nickel and its compounds	–

Table 2

Characteristics of substances analyzed in this paper

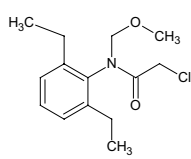
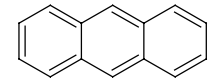
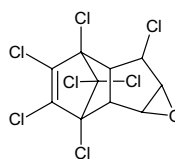
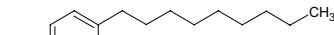
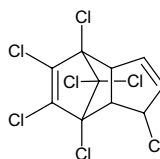
	Alachlor	Anthracene	Heptachlor epoxide	4-nonylphenol	Heptachlor
Structure					
logP <sub>ow</sub>	2.99	3.99	4.45	4.68	5.24

Table 3  
Raw waters characteristics

Water source	Mała Panew	Dobromierz
pH	7.86	7.12
TOC (mg L <sup>-1</sup> )	17.45	4.40
DOC (mg L <sup>-1</sup> )	13.52	4.26
POC (mg L <sup>-1</sup> )	3.93	0.14
UV254 (m <sup>-1</sup> )	35.8	5.98
SUVA (m <sup>2</sup> g <sup>-1</sup> )	2.65	1.40

Table 4  
Basic characteristics of used coagulants

Parameter	PACl	PAC	Alum (Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> ·16H <sub>2</sub> O)
Al <sup>3+</sup> , % mas.*	11.77	8.46	9.10
Cl <sup>-</sup> , % mas.*	6.52	22.6	–
pH	4.12	1.12	3.21
Basicity, %	87.12	32.32	0
OH/Al	2.53	0.97	–

\*mass concentration in %.

strongly basic anion exchange resin based on crosslinked polyacrylate. Lewatit<sup>®</sup>TP207 is weakly acidic cation exchange resin based on crosslinked polystyrene. As coal-based filter Norit<sup>®</sup> GAC830 was used.

### 2.5. Experimental procedures

Both surface waters and demi water were spiked with mixed standard to concentration 20 µg L<sup>-1</sup> for each MPs.

Coagulants doses were chosen based on zeta potential isoelectric point ( $\zeta = 0$ ) (IP) analysis. For this purpose  $\zeta = f(\text{mg L}^{-1})$  and  $\zeta = f(\text{mg Al L}^{-1})$  dependencies were determined. Zeta potential measurement was conducted using Malvern Zetasizer Nanozeta potential meter spared with automatic titrator.

coagulation experiments were performed with standard Jar-test procedure on a Velp Scientifica flocculator, in 4-phased sequence for samples with flocculant added, and in 3-phased sequence for samples with only coagulant. In the first case, phases were, respectively: 100 rpm and 1 min (coagulant), 100 rpm and 1 min (flocculant), 40 rpm and 15 min, 60 min of sedimentation. Flocculant dose was 0.1 mg L<sup>-1</sup>. In the second case, procedure was similar, excluding flocculant mixing phase, which was not performed. In this part of experiment PACl in IP dose was used as coagulant. After sedimentation time supernatants were collected for further analysis.

Sorption processes were performed with raw and coagulated waters. Filtration columns were filled with 10 cm bed layer of sorption media. Activated carbon was preconditioned with deionized water for 24 h. Anionic and cationic resins were preconditioned with 1% NaOH and 10% NaCl mixed solution followed with deionized water rinsing. Feed water was pumped into the column with LeadFluid BT100F peristaltic pump for 12 r, maintaining contact time of 15 min. Collected samples were subjected to further analysis.

### 2.6. Analytical procedures

MPs concentrations were determined using gas chromatography (GC) (Agilent 7890B) coupled to a single quadrupole mass spectrometer detector (MSD) (Agilent 5977A). Columns used for solid phase extraction (SPE) were 6 mL C18 Bakerbond<sup>™</sup> spe with 500 mg octadecyl phase (J.T.Baker). Preconditioning columns were rinsed, respectively, with 5 mL of dichloromethane/ethyl acetate (DCE) (1:1, v/v) (Chempur/Merck), 5 mL of methanol (Sharlau), and 5 mL of deionized water. Pre-concentrated samples were extracted with DCE, acetone (Sharlau) and deionized water (respectively, 5 mL each). The eluted extract was then evaporated with Biotage<sup>®</sup> TurboVap<sup>®</sup> Classic II until 250 µL volume was reached. Solvent was then changed to Hexane (VWR Chemicals) and evaporated to 500 µL. As internal standard for selected ion mass analysis (SIM analysis) 1,2,3,4-tetrachloronaphthalene was added (100 µg L<sup>-1</sup> in hexane).

TOC and DOC concentration measurement was provided on AnalytikJena multi N/C 3100 apparatus. POC was calculated from the difference between TOC and DOC.

Absorbance was measured using Merck Spectroquant<sup>®</sup> Prove 300 spectrophotometer.

## 3. Results and discussion

### 3.1. Coagulants doses determination

Results of zeta potential based coagulants doses predictions are shown in Figs. 1a and b.

Coagulants doses for Mała Panew were 43 mg L<sup>-1</sup> for PACl, 85 mg L<sup>-1</sup> for PAC, and 87 mg L<sup>-1</sup> for Alum. In the case of Dobromierz, these were, respectively: 16, 34, and 40 mg L<sup>-1</sup>. As it is seen in both cases PACl dose demand is about half of PAC and alum doses. For the synthetic water coagulation process, the same doses as in the case of Dobromierz were chosen.

Coagulation efficiency is highly affected by coagulants Al species. High basic coagulants, as PACl, have proven much higher polymeric Al<sub>13</sub> content, showing superior coagulation effectiveness [30,31]. As seen in Figs. 2a and b, in both cases using PACl allows to reduce overall aluminum dose, which is especially noticeable for water with high organic load. For water in the Mała Panew River, where the percentage of insoluble organic suspension is higher and amounts to 23%, the Al dose for high basic coagulants PACl was lower by 29% and 35% in relation to PAC and aluminum sulfate (Alum), respectively. For Dobromierz water samples DOC concentration was relatively low (Table 3) and the percentage of DOC in TOC is high (97%), therefore differences in coagulants efficiency were less significant. This is also confirmed by the low value of SUVA at the level 1.3 m<sup>2</sup> g<sup>-1</sup>. In this case, the Al dose for PACl coagulant was 14% and 33% lower for PAC and Alum, respectively. Still, higher polymeric Al species share means better charge neutralization properties.

### 3.2. Coagulation and flocculation processes effectiveness

In coagulation process, two main removal mechanisms may occur. First, is due to sweeping coagulation in which pollutants are trapped inside agglomerates formed

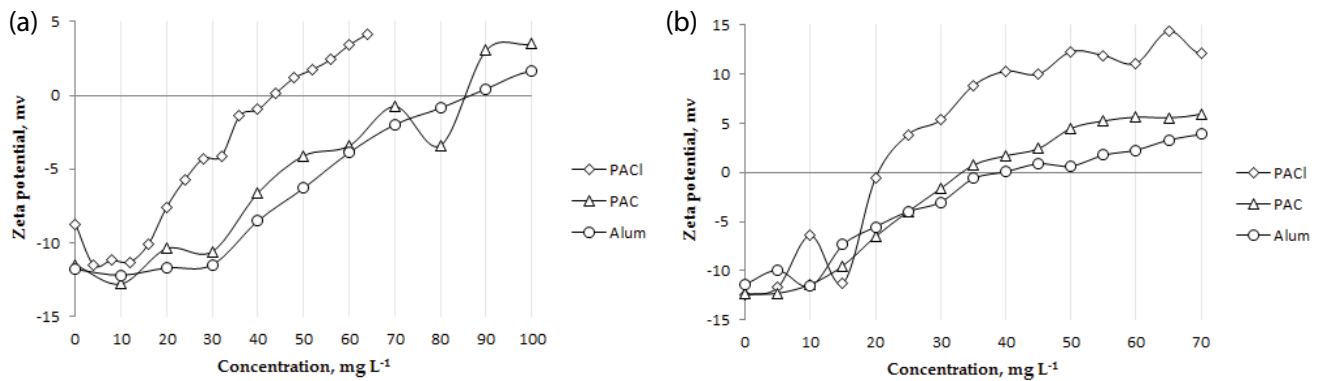


Fig. 1. (a and b) Zeta potential vs. coagulant dose for Mała Panew River (left) and Dobromierz Lake (right).

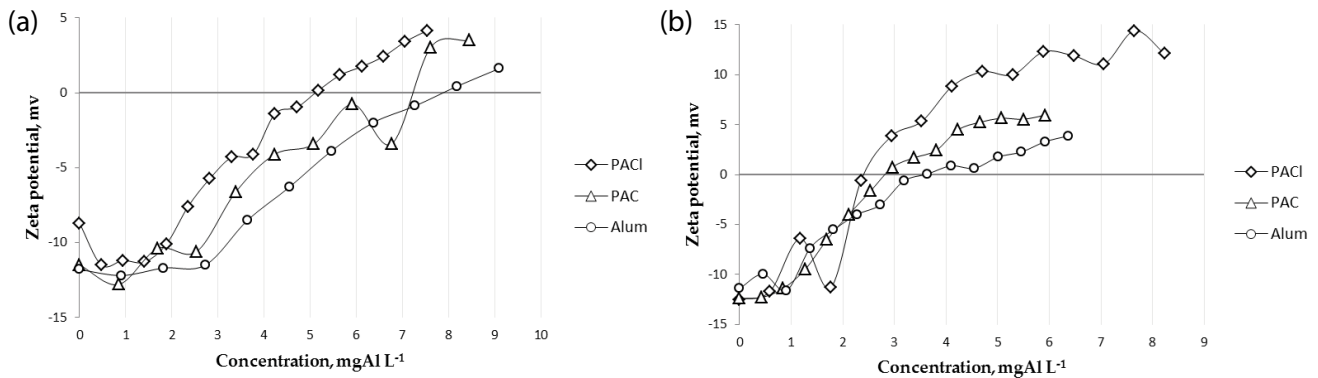


Fig. 2. (a and b) Zeta potential vs. Al dose for Mała Panew River (left) and Dobromierz Lake (right).

during flocculation phase. This one is especially promoted by organic and inorganic flock occurrence. In Mała Panew River 23% of TOC was in form of insoluble organic suspension, which compared to 3% in Dobromierz Lake is relatively high. Also, high molecular organic compounds presence which is already considered as soluble, may positively affect this coagulation mechanism efficiency. In low-contaminated medium swiwe, coagulation becomes less significant in favor of the adsorption process, which is second considered coagulation mechanism. This one is because of lack of aggregation flocks. Adsorption process occurs in both coagulation and flocculation phases, first on emerging aluminum hydroxide particles followed by on-flocs adsorption. In study case, the removal efficiency in natural waters, in relation to prepared water, was noticeable higher. In general, the best efficiency was achieved in Mała Panew River, where initial organic load was the highest. The lowest removal was reported in case of synthetic water, what is probably due to process limitation for adsorption only. In the case of natural waters PACI presented as the most effective coagulant and the least effective was Alum. Situation changed in synthetic water purification process, where Alum turned out to be the most effective and PACI the least effective. In this case, Alum dose was the highest, which probably resulted in the largest amount of aluminum hydroxide, hence most efficient adsorption. Coagulation process resulted in 77.9% reduction at most, in case of Hcl using PACI coagulant (Fig. 3a).

When using flocculants, in majority of cases, cationic polyelectrolyte turned out to be the most efficient. PACI coagulant coupled with cationic flocculant resulted in 85.4% reduction of Hcl in water from Mała Panew river (Fig. 3a), which was the highest result obtained in this study. Hcl removal in water from Dobromierz Lake, had lower effectiveness with reduction level of only 75.9%. In this case, the best result for cationic flocculant was achieved for the removal of Ant, where the efficiency of the process reached 21.1% higher level than coagulation with only PACI.

As predicted, the highest removal susceptibility was for substance with the highest  $\log P_{o/w}$  – Hcl. With the exception of Npl in Mała Panew sample, lower  $\log P_{o/w}$  resulted in lower removal efficiency. It emphasizes the importance of adsorption process in MPs reduction during coagulation process. Figs. 3 and 4 show the overall MPs removal in coagulation and flocculation processes.

### 3.3. Sorption processes effectiveness

Effectiveness of sorption process is presented in Figs. 5–7. In case of raw waters treatment, process efficiency highly depends on matrix load. For GAC high concentration of organic carbon significantly decreases MPs reduction, thus, the best efficiency was reached for synthetic water (Fig. 7), where only for the Acl the reduction level was 90% and for all the other MPs a complete removal was noted. In case

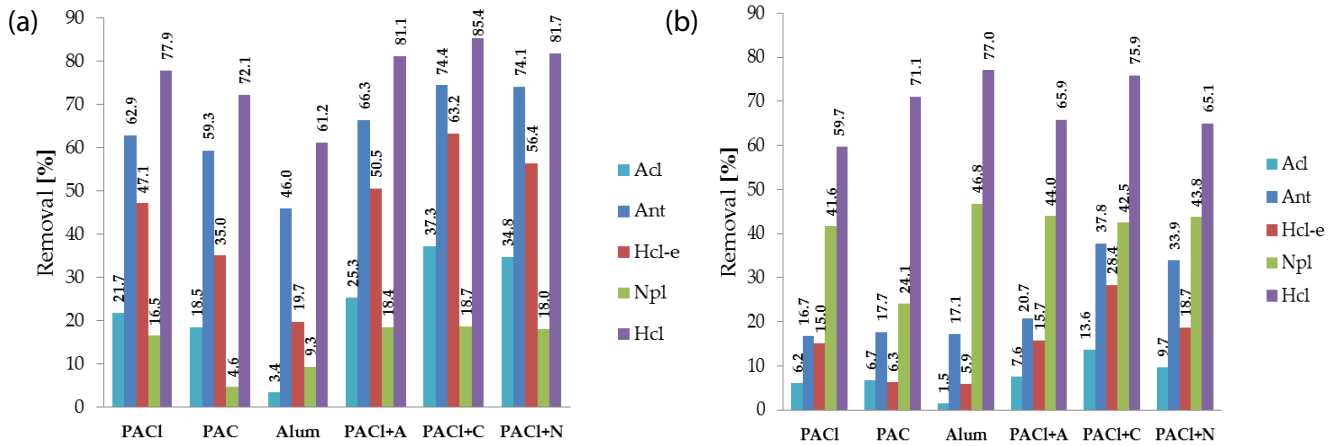


Fig. 3. (a and b) MPs removal in coagulation and flocculation processes in Mała Panew (left) and Dobromierz (right) samples.

of Mała Panew river and Dobromierz Lake samples, average reductions were 61% (changes from 41.1% to 72.7%) and 88% (changes from 69.3% to 100%) respectively. In the

Dobromierz Lake water reduction reached 100% (under calibration range) for Hcl.

Raw waters treated on S5428 resin were characterized by high removal potential, independently of matrix load. The average effectiveness was 93.3%, 93.8%, and 97% for Mała Panew River, Dobromierz Lake, and synthetic water, respectively. This fact confirms reports [21,22], that occurring in natural waters, heavy molecular weight compounds, like humic acids, may block pores of sorption grains decreasing process effectiveness.

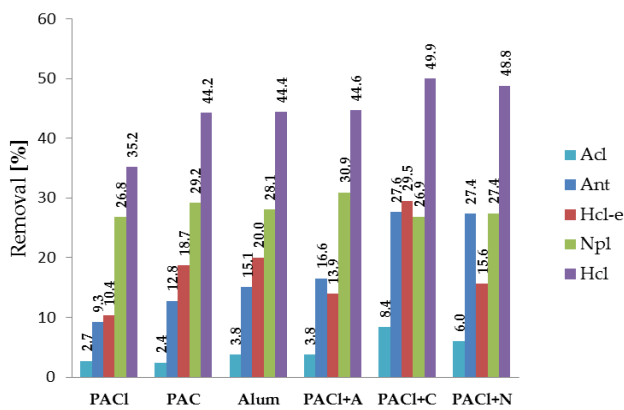


Fig. 4. MPs removal in coagulation and flocculation processes in synthetic water samples.

For pre-coagulated samples similar phenomena was observed. For samples from the Mała Panew River, the GAC sorption increased the average process efficiency for all MPs by 25.8%, with Npl achieving the highest increase of 39.6% and Hcl-e the lowest of 19.3%. The use of coagulation and GAC for water samples from Mała Panew River (Fig. 5) was characterized by the lowest reduction rate for Npl (80.4%) and the largest for Hcl (94.5%). On the other hand, coagulation and GAC allowed 100% reduction of MPs for water from Dobromierz Lake and synthetic water. Pre-treated waters sorption on S5428 caused 100% removal for every MPs in all three cases. TP 207 cation exchange resin

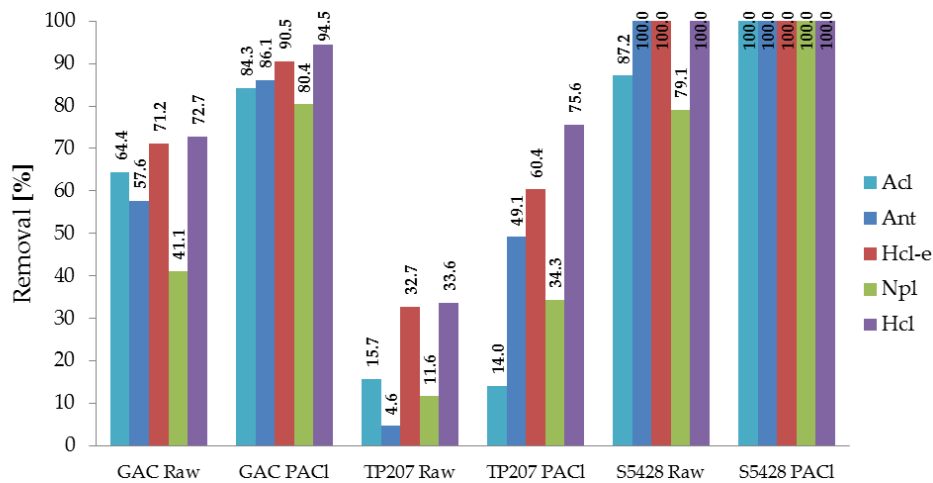


Fig. 5. Sorption process effectiveness of Mała Panew River water.

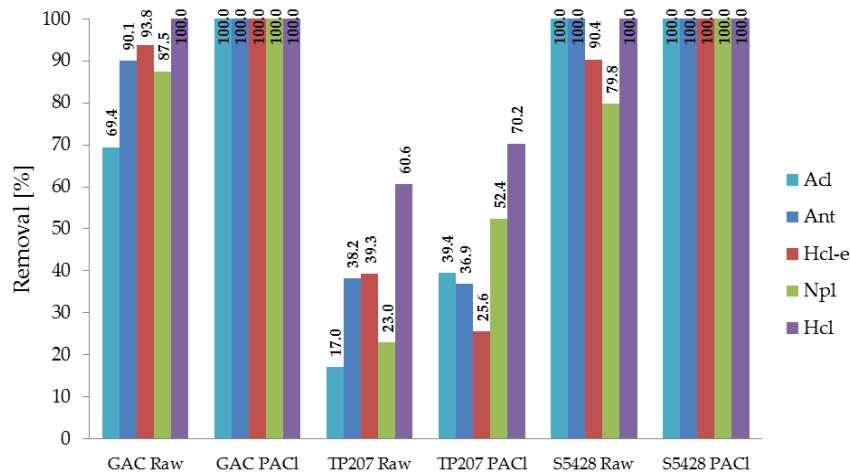


Fig. 6. Sorption process effectiveness of Dobromierz Lake water.

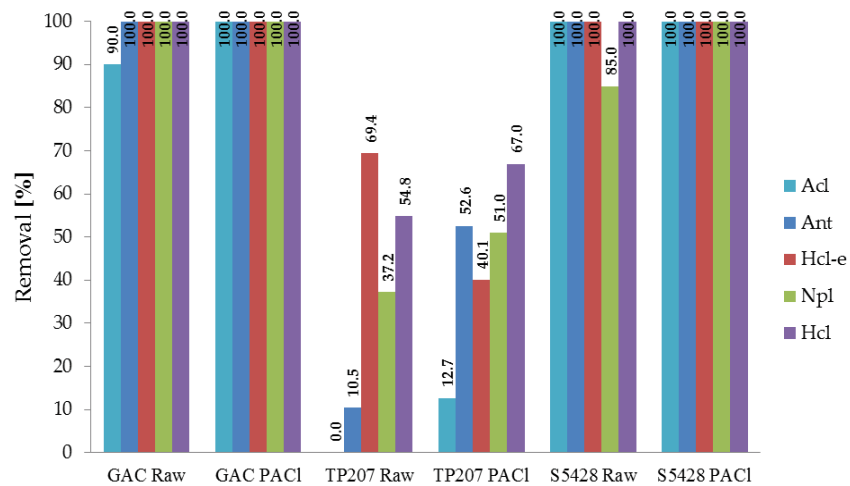


Fig. 7. Sorption process effectiveness of synthetic water.

characterized with low efficiency and high unpredictability for each case.

#### 4. Conclusions

- The efficiency of two processes, coagulation, and sorption, is highly affected by matrix load. High concentrations of organic contamination may increase a coagulation processes efficiency due to higher impact of sweeping coagulation mechanism. On the other hand, greater concentration of high molecular compounds may decrease sorption effectiveness, especially in the case of activated carbon, where pores may be blocked.
- Overall, the coagulation process efficiency was the highest when using PACl. For both natural waters, using this coagulant allowed to reach zeta potential isoelectric point most effectively. This phenomenon was especially visible in the more contaminated, Mała Panew River.
- Micropollutants removal in coagulation process reached the highest efficiency in the case of Mała Panew River, using PACl. Maximum level for Hcl removal was noted for both Mała Panew and Dobromierz Lake. The

coagulation efficiency can achieve the level of 77.9% and 59.7% for water sample from Mała Panew River and Dobromierz Lake, respectively.

- The effectiveness of flocculant usage was the highest for cationic polyelectrolyte. The use of a catalytic flocculant in the water treatment process allowed to increase the efficiency of MPs removal by a maximum of 21.1% for Ant in the water from Dobromierz Lake.
- Sorption process on anionic resin, as well as on activated carbon is highly efficient in MPs removal. In both cases, reduction reached 100%. However, inorganic resins tend to be more resistant to high content of NOM.
- Conducted study has shown that coupling coagulation and sorption processes gave superior results in MPs removal for all tested waters.

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