

REMOVAL OF ORGANIC MATTER FROM SURFACE WATER USING COAGULANTS WITH VARIOUS BASICITY

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ABSTRACT

Humic substances are a natural admixture of surface water and determine the level of organic pollution of water and colour intensity. Application of coagulation process in surface water treatment allows for decrease turbidity and colour of water, as well as organic matter content. In Poland most drinking water treatment plants use aluminium sulphate as a coagulant. Research works on pre-hydrolyzed coagulants, e.g. polyaluminium chlorides (general formula $Al_n(OH)_mCl_{3n-m}$) are also carried out. The aim of this study was to evaluate the effectiveness of the coagulation process using polyaluminium chlorides with different basicity, in reducing the level of pollution of surface water with organic substances. Apart from the typical indicators used to evaluate the content of organic compounds, the potential for trihalomethanes formation THM-FP was also determined. The influence of the type of coagulant (low, medium, highly alkaline) on the efficiency of organic compound removal, determined as total organic carbon TOC, oxidisability OXI, absorbance UV_{254} was stated. Under the conditions of the coagulation (pH 7.2–7.4, temperature of 19–21°C), the best results were obtained using highly alkaline polyaluminium chlorides PAX-XL19F, PAX-XL1905 and PAX-XL1910S, decrease in TOC and OXI by 43–46%, slightly worse – 40–41% using low alkaline PAX18. Using the medium alkaline coagulants PAX-XL61 and PAX-XL69, 30–35% removal of organic matter was obtained. Despite various effects of dissolved organic carbon removal, depending on the used coagulant, THM-FP in purified water did not differ significantly and ranged from 10.0 to 10.9 mgCHCl₃ m⁻³. It was by 37–42% lower than in surface water.

Keywords: coagulation, pre-hydrolyzed aluminium salts, organic matter, water.

INTRODUCTION

Natural organic matter (NOM) is defined as a complex matrix of organic materials present in all natural water. The hydrophilic fractions of NOM are composed mostly of aliphatic carbon and nitrogenous compounds, as carboxylic acids, carbohydrates and proteins. Hydrophobic NOM primarily consists of humic and fulvic acids (humic substances) [Matilainen et al. 2010]. Particular increase of interest in the composition of humic substances in water is noted since the publication of research regarding the generation of trihalomethanes (THMs) during the chlorination of water. Four derivatives of methane belong

to THMs group: chloroform CHCl₃, bromoform CHBr₃, bromodichloromethane CHCl₂Br, dibromochloromethane CHClBr₂. The permissible concentration of the sum of their content in drinking water according to [Regulation 2015] equals 100 mg m⁻³. Analyses of water collected in different water treatment plants in the world [Abdullah et al. 2003; Kim 2009; Chang et al. 2010; Pardakhti et al. 2011], and also in pool water [Lee et al. 2009], confirm the possibility of THMs presence in water subjected to chlorination. In order to reduce THMs concentration in drinking water, various technological approaches are applied: removal of produced THMs, removal of THMs precursors, use of disinfectants other than chlorine.

THMs precursors are mainly humic acids, metabolites of aquatic organisms, mono-, di- and tri-carboxylic acids, and aromatic carboxylic acids [Dojlido 2002]. For the reduction of organic matter in water, coagulation can be used [Świdarska-Bróz et al. 2008; Matilainen et al. 2010; Alexander et al. 2012; Jeong et al. 2014].

Efficiency of the coagulation process depends on physico-chemical composition of water, pH value, type and dosage of coagulants. The most often used aluminium coagulants are aluminium sulphate $Al_2(SO_4)_3$ and aluminium chloride $AlCl_3$. In recent years, the usage of pre-hydrolyzed coagulants is more and more popular, e.g. alkaline polyaluminium chlorides with general formula $Al_n(OH)_mCl_{3n-m}$. In solutions of polyaluminium chlorides, besides monomers Al^{3+} , $Al(OH)^{2+}$, $Al(OH)_2^+$, which are also present in solutions of non-hydrolyzed aluminum coagulants, presence of many polymerized aluminium hydroxyl complexes with general formula $Al_p(OH)_q^{(3p-q)+}$, e.g. $Al_2(OH)_2^{4+}$, $Al_3(OH)_4^{5+}$, $Al_{13}O_4(OH)_{24}^{7+}$, was identified [Yan et al. 2008]. Polycationic products of preliminary hydrolysis of aluminium, after addition of polyaluminium chlorides to the treated water, undergo the hydrolysis to $Al(OH)_3$ far more slowly than aluminium present in the solution of aluminium sulphate. This results in the fact that the usage of polyaluminium chlorides is more effective in destabilisation of negatively charged colloids, which cause mainly water colour and turbidity, in comparison with the results obtained with the usage of aluminium sulphate. $Al_{13}O_4(OH)_{24}^{7+}$ is called polymer A_{13} and is considered as the most stable and effective in destabilisation of negatively charged colloids especially the colloidal particles which causes high turbidity of water [Lin et al. 2008]. Higher effectiveness of hydrolysed coagulants compared to aluminium sulphate results not only from the presence of Al_{13} polycations but also from the difference in a structure of the aluminium hydroxides which are generated during hydrolysis of these coagulant agents [Lin et al. 2009; 2014].

During water purification by means of the coagulation process, often over 90 and 80% reduction is obtained respectively for turbidity and colour [Dąbrowska and Rosińska 2013]. Research on treatment of water with different compositions also confirm the effectiveness of the coagulation in the removal of organic matter [Hussain et al. 2013; Wang et al. 2013].

The aim of this study was to evaluate the effectiveness of the coagulation process using pre-hydrolyzed salts, polyaluminium chlorides with different basicity, in reducing the level of pollution of surface water with organic substances. Apart from the typical indicators used to evaluate the content of organic compounds (total and dissolved carbon, ultraviolet absorbance at 254 nm), the potential for trihalomethanes formation THM-FP was also determined. The THM-FP potential reflects the amount of precursors present in water, capable of generating THMs.

MATERIAL AND METODS

Surface water from the Poraj dam reservoir was used for the study. This reservoir is located on 764 km of the Warta river, in the area of Poraj and Kozięłowy municipalities. The area of the reservoir is 497 ha, length – 4.9 km, the average depth – 3.9 m. The water was collected in November 2015.

Hydrolyzed salts, polyaluminium chlorides with commercial names PAX18, PAX-XL61, PAX-XL69, PAX-XL19F, PAX-XL1905, and PAX-XL1910S, were used as coagulants, produced by KEMIPOL company in Police (Poland). The characteristics of the coagulants are shown in Table 1.

Basicity of polyaluminium chlorides varied from 41 to 85%. The basicity is determined by the ratio of the number of OH^- moles to Al^{3+} in the coagulant, defined as the coefficient $R=[OH^-]/[Al^{3+}]$. The relation between the R value and the basicity of the coagulant is as follow:

Table 1. Characteristics of the coagulants

Parameter	Unit	PAX					
		18	XL61	XL69	XL19F	XL1905	XL1910S
Basicity	%	41.0±3	70.0±10	60.0±10	85.0±5	85.0±5	85.0±5
Al_2O_3	%	17.0±0.6	10.2±0.4	11.3±0.9	16.0±0.9	11.3±0.9	19.8±0.4
Chlorides	%	21.0±2.0	11.0±2.0	11.0±2.0	5.5±0.5	5.0±1.0	7.5±1.0
pH	-	1.0±0.2	3.0±0.5	2.5±0.5	4.0±0.5	3.6±0.4	3.5±0.5

basicity (%) = $R/0.03$. Commercial solutions of PAX contained from 10.2 to 19.8% of Al_2O_3 . For the analyses, coagulant solutions were prepared by diluting commercial products so that they contained 1.0 gAl dm^{-3} .

In the first stage, the coagulation process was conducted in glass beakers with 2 dm^3 volume, to each beaker 1.5 dm^3 of analysed water was measured. The coagulants were introduced in amounts of 2, 3 and 4 mgAl dm^{-3} , and with the use of a mechanical stirrer fast stirring was executed for 2 minutes (applying 250 RPM), and then slow stirring for 15 minutes (25 RPM). After this time the samples were subjected to 1 hour sedimentation. Afterwards 0.5 dm^3 of water was decanted and analysed. The scope of the analysis of water after the coagulation included: pH, turbidity, colour, total organic carbon, oxidisability.

In the second stage, execution of the coagulation process was repeated using a coagulant dose equal to 4 mgAl dm^{-3} . The scope of the analysis of water after the coagulation included: dissolved organic carbon, ultraviolet absorbance at 254 nm, aluminium, trihalomethane formation potential.

The physicochemical parameters of water were measured with the following methods: pH – potentiometrically; turbidity – nephelometric with Eutech Instruments TN-100 nephelometer; colour – colometrically with platinum-cobalt standard method; dissolved (DOC) and total organic carbon (TOC) – by infrared spectrophotometry with carbon analyser Multi N/C, Analytik Jena (to measure DOC concentration the samples of water were filtered through $0.45 \mu\text{m}$ filtration membrane); oxidisability (OXI) by potassium permanganate method; aluminium – with Aquaquant 14413 aluminium test. Absorbance in the ultraviolet UV at wavelength of 254 nm was determined (spectrophotometer M501 by Camspec) using 1 cm quartz cells. $SUVA_{254}$ index was calculated as the ratio of absorbance at UV_{254} to the DOC content. Connecting the values of absorbance and DOC into single coefficient $SUVA_{254}$ enables to determine properties of dissolved organic matter in water, and vulnerability to their removal in the coagulation process.

In order to determine the THM-FP, measured into dark bottles surface water samples before treatment and after the coagulation, adjusted to pH 7 with phosphate buffer, were chlorinated using chlorine water prepared with sodium hypochlorite. Such dose was used to achieve, after a period of 24 hours at the temperature of 25°C ,

a concentration of residual free chlorine in the range of 3 to $4 \text{ mgCl}_2 \text{ dm}^{-3}$ [APHA 1998]. The concentration of chlorine in the chlorine water was determined iodometrically, and the chlorine remaining after 24 hours in water samples by means of spectrophotometric method using orthotolidine as a factor in the colour reaction. For the determination of THMs in water samples before and after the chlorination process, 10 cm^3 of water was collected into tubes, 1 cm^3 of n-pentane was added, and it was intensively shaken for 1 minute. After separation of the layers, 2 mm^3 of the extract was collected with a microsyringe and introduced into a chromatography column. Nitrogen was used as a carrier gas. Distribution of the compounds was performed on a capillary column DB-5 ($30\text{m} \times 0.25\text{mm} \times 0.25\mu\text{m}$) and analyzed by means of gas chromatography with microelectron capture detector μECD (chromatograph Agilent 6890N). Temperature program: starting at 40°C , ramp $40^\circ\text{C}/\text{min}$ to 120°C , then $15^\circ\text{C}/\text{min}$ to 250°C . As a standard, solution by CPAchem Ltd was used, containing 4 components (CHCl_3 , CHBr_3 , CHCl_2Br , CHClBr_2) with a concentration of $10 \mu\text{g cm}^{-3}$ each in iso-octane.

The potential for trihalomethane formation was calculated by subtracting the initial THMs concentration from the concentration of these compounds determined after 24 hour contact of water with chlorine.

RESULTS AND DISCUSSION

Surface water was characterized by colour equal to 25 gPt m^{-3} and turbidity of 7.6 NTU. The TOC and the DOC content amounted to 8.8 and 7.4 gC m^{-3} respectively, and the absorbance at UV_{254} – 17.6 m^{-1} . Thus the $SUVA_{254}$ value was equal to $2.38 \text{ m}^2 (\text{gC})^{-1}$. The $SUVA_{254}$ value in the range of $2\text{--}4 \text{ m}^2 (\text{gC})^{-1}$ suggests that in the surface water occurred a mixture of hydrophilic and hydrophobic humic substances and other natural organic compounds, both of low and high molecular weights. At the same time, as the literature states [Mołczan et al. 2006], in case of waters with a value of this index $<3 \text{ m}^2 (\text{gC})^{-1}$, the degree of DOC removal generally does not exceed 30%.

The effectiveness of turbidity and colour removal by various coagulants are presented in Table 2, as well as changes in OXI and TOC concentration during coagulation. Based on the results of turbidity removal efficiency it was stated that the

Table 2. Effect of coagulation with various agents on selected physicochemical properties of water

Coagulant	Dose [gAl m ⁻³]	pH -	Turbidity [NTU]	Colour [gPt m ⁻³]	PV [gO ₂ m ⁻³]	TOC [gC m ⁻³]
Raw water	-	7.42	7.6	25.0	5.4	8.8
PAX18	2	7.36	2.9	15.0	3.9	6.1
	3	7.28	1.4	10.0	3.4	5.7
	4	7.22	0.9	5.0	3.2	5.3
PAX-XL61	2	7.38	3.5	15.0	3.9	6.2
	3	7.31	2.6	10.0	3.8	5.9
	4	7.26	1.8	7.5	3.5	5.7
PAX-XL69	2	7.37	3.3	20.0	4.4	6.5
	3	7.30	2.2	15.0	4.2	6.4
	4	7.24	1.5	10.0	3.8	6.1
PAX-XL19F	2	7.40	2.4	10.0	3.4	5.5
	3	7.38	1.5	7.5	3.1	5.1
	4	7.34	0.9	7.5	3.0	4.8
PAX-XL1905	2	7.39	2.2	10.0	3.4	5.6
	3	7.33	1.6	5.0	3.2	5.4
	4	7.31	0.9	5.0	2.9	4.9
PAX-XL1910S	2	7.40	2.1	12.5	3.6	5.6
	3	7.36	1.3	7.5	3.2	5.2
	4	7.32	0.8	5.0	3.1	4.9

most effective agent (under the conditions of coagulation process: T=19–21 °C, pH 7.2–7.4) was PAX-XL1910S. Application of this product at dose of 4 mgAl dm⁻³ decreased turbidity of water from 7.6 to 0.8 NTU. Also PAX18, PAX-XL19F, PAX-XL1905 agents were effective in removing of turbidity (decreased turbidity to 0.9 NTU).

PAX18, PAX-XL1905, PAX-XL1910S were also the most effective products for colour removal (decreased colour from 25 to 5 gPt m⁻³). PAX-XL61 and PAX-XL19F agents reduced colour to 7.5 g Pt m⁻³, whereas PAX-XL69 to 10 gPt m⁻³.

The efficiency of removal of organic compounds depended both on the type of the coagulant, and on its dose. The best results were obtained with a dose of coagulants equal to 4 mgAl dm⁻³. The content of organic compounds after the coagulation process carried out using low-alkaline PAX18, determined as OXI and TOC, decreased respectively from 5.4 gO₂ m⁻³ and 8.8 gC m⁻³ to 3.2 gO₂ m⁻³ and 5.3 gC m⁻³. The absorbance value UV₂₅₄ decreased by 8.4 m⁻¹. With the usage of medium alkaline polyaluminium chlorides PAX-XL61 and PAX-XL69, a reduction in TOC content was obtained, respectively to 5.7 and 6.1 gC m⁻³, and the OXI to 3.5 and 3.8 gO₂ m⁻³. The best results of organic compound content reduction were obtained with the usage of highly

alkaline coagulants: PAX-XL1905, PAX-XL19F, and PAX-XL1910S. The TOC content in water after the coagulation was equal to 4.8–4.9 gC m⁻³, and the OXI – 2.9–3.1 gO₂ m⁻³. A higher basicity of the coagulant usually indicates its higher efficiency, resulting from a greater portion of polymerized forms. This is confirmed by various laboratory tests [Rak and Świdorska-Bróz 2003; Yang et al. 2011a; Yang et al. 2011b; Dąbrowska and Rosińska 2013]. However, the experience gained in water treatment plants have shown that even under the same operating conditions of the coagulation (pH, coagulant dose), the outcome of this process does not always confirm this relation [Gumińska 2012]. Therefore, in each case, it is necessary to choose an appropriate coagulant, its dosage and the process parameters.

Lowering the DOC content in water after the coagulation was in the range of 22–39%, and the UV₂₅₄ absorbance in the range of 48–59%, depending on the used coagulant – Table 3. The largest reduction in values of those indicators were stated using highly alkaline hydrolyzed chlorides PAX-XL19F and PAX-XL1910S. It is believed [Mołczan et al. 2006], that significant reduction in the value of the absorbance during the coagulation is obtained by removing the hydrophobic and remaining the hydrophilic fraction of the DOC.

Table 3. Effect of coagulation with various agents on selected physicochemical properties of water

Coagulant	Dose [gAl m ⁻³]	pH -	Al [gAl m ⁻³]	DOC [gC m ⁻³]	UV ₂₅₄ [m ⁻¹]	SUVA [m ² (gC) ⁻¹]
Raw water	-	7.42	<0.05	7.4	17.6	2.38
PAX18	4	7.22	0.18	5.1	8.4	1.65
PAX-XL61	4	7.26	0.25	5.5	8.8	1.60
PAX-XL69	4	7.24	0.30	5.8	9.2	1.59
PAX-XL19F	4	7.34	0.10	4.5	7.3	1.62
PAX-XL1905	4	7.31	0.05	4.8	7.8	1.63
PAX-XL1910S	4	7.32	0.07	4.7	7.5	1.60

The SUVA value was equal to 1.56–1.65. This confirms that in water after the coagulation, organic substances remained, which are unsusceptible to the removal in this process. As research has shown [Szlachta and Adamski 2008], the coagulation is the most effective in the removal of compounds with molecular weight of 2.2–5.3 kDa, whose content in water was reduced by about 62%. Whereas for organic substances with lower molecular weights 1.5–2.2 kDa and 1.0–1.6 kDa amounted to 25 and 11%, respectively.

The effect of alkalinity of aluminium coagulants and water pH on the removal of organic substances was also studied by Świdarska-Bróz et al. [2008]. The authors stated that with increasing dose and alkalinity of coagulants and decreasing water pH, the coagulants efficiency in removal of analyzed fractions of organic substances increased. DOC share in TOC had also a significant meaning. With increasing participation of DOC in TOC, efficiency of organic compounds removal decreased.

The lowest concentration of remaining aluminium occurred in water after the coagulation, when highly alkaline PAX were applied. It ranged from 0.05 to 0.10 gAl m⁻³ and it was lower than the permissible value for drinking water of

0.2 gAl m⁻³ [Regulation 2015]. Also, using low alkaline PAX18, residual aluminium concentration of less than 0.2 gAl m⁻³ was obtained, while using medium alkaline PAX-XL61 and PAX-XL69 – it was higher than this value.

In the collected surface water, as well as in the water after the coagulation process, presence of trihalomethanes was not observed. However, their presence was demonstrated in these waters after the chlorination process. THM concentration in surface water and in water purified in the coagulation process, after 24 hour period of contact with chlorine, are shown in Table 4. Trihalomethane formation potential THM-FP is given in conversion to mgCHCl₃ m⁻³.

In the surface water subjected to chlorination, presence of CHCl₃ and CHCl₂Br was demonstrated, CHBr₃ and CHClBr₂ were not detected. CHCl₃ and CHCl₂Br concentration was equal to 15.36 and 2.64 mg m⁻³, respectively. In the water subjected to chlorination after the coagulation process, CHCl₃ concentration ranged from 8.52 to 9.45 mg m⁻³, while CHCl₂Br from 1.92 to 2.16 mg m⁻³. The lowest concentration of these compounds were obtained in the water subjected to chlorination after previous treatment using PAX-XL19F and PAX-XL1910S.

Table 4. THM concentration in surface water and in water purified in the coagulation process

Coagulant	THM [mg m ⁻³]				THM-FP [mg m ⁻³ as CHCl ₃]
	CHCl ₃	CHCl ₂ Br	CHClBr ₂	CHBr ₃	
Raw water	15.36	2.64	nd	nd	17.28
PAX18	8.76	2.08	nd	nd	10.28
PAX-XL61	9.12	2.16	nd	nd	10.69
PAX-XL69	9.48	2.10	nd	nd	10.94
PAX-XL19F	8.61	1.92	nd	nd	10.01
PAX-XL1905	8.88	2.04	nd	nd	10.37
PAX-XL1910S	8.52	2.10	nd	nd	10.05

nd – not detected

Despite various effects of dissolved organic carbon removal, depending on the used coagulant, THM formation potential in purified water did not differ significantly and ranged from 10.0 to 10.9 mgCHCl₃ m⁻³. It was by 37–42% lower than in surface water. Although during the coagulation process a significant portion of organic matter was removed, yet the remaining part was still characterized by a high THM formation potential. The content of organic carbon is, however, only one of the conditions determining the formation of THMs. Other factors influencing the mechanism of THM formation are mainly the pH, time, temperature and the presence of bromides [Abdullah et al. 2003; Hong et al. 2007; Platikanov et al. 2012].

CONCLUSIONS

The influence of the type of coagulant (low, medium, highly alkaline) on the efficiency of organic compound removal, determined as total organic carbon TOC, oxidisability OXI, ultraviolet absorbance at 254 nm was stated. Under the conditions of the coagulation (pH 7.2–7.4, temperature of 19–21°C), the best results were obtained using highly alkaline polyaluminium chlorides PAX-XL19F, PAX-XL1905 and PAX-XL1910S, decrease in TOC and OXI by 43–46%, slightly worse – 40–41% using low alkaline PAX18. Using the medium alkaline coagulants PAX-XL61 and PAX-XL69, 30–35% removal of organic matter was obtained. Considering the concentration of aluminium remaining after the coagulation process performed in these conditions, applying highly alkaline coagulants was most advantageous. Despite various effects of dissolved organic carbon removal, depending on the coagulant used, THM formation potential in purified water did not differ significantly and ranged from 10.0 to 10.9 mgCHCl₃ m⁻³. It was by 37–42% lower than in surface water.

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