



Comparison of effectiveness of coagulation with aluminum sulfate and pre-hydrolyzed aluminum coagulants

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ABSTRACT

The purpose of the present study was to compare the effectiveness of coagulation process conducted using a classical nonhydrolyzed coagulant—aluminum (VI) sulfate and pre-hydrolyzed coagulants: PAX XL 19H and Flokor 1.2A. Water samples subjected to coagulation were collected from selected water treatment plant after the pre-ozonation process. Research has been conducted in the winter season. True color of water amounted to 7.0 mg Pt/L and turbidity was 7.29 NTU. The study in a laboratory scale was carried out with the usage of a six-beaker flocculator. Volumetric coagulation was performed in water samples of the volume of 1 L. In six beakers, rapid mixing (3 min at the rotational speed of 200 rpm) was followed by 30 min slow mixing (at 30 rpm). After coagulation, the samples were subject to 60 min sedimentation. The coagulant dose was optimized for minimum color, turbidity, and UV₂₅₄ absorbance values using conventional jar testing procedures. The optimum dose for aluminum (VI) sulfate amounted to 2.2 mg Al/L. For prehydrolyzed coagulants, the doses were much lower, i.e. 1.2 mg Al/L. Also less wear of water alkalinity was obtained than in the case of aluminum (VI) sulfate. The effectiveness of removal of turbidity for ALK, PAX XL 19H, and Flokor 1.2A was 63, 70, and 74% respectively.

Keywords: Drinking water; Coagulation; Aluminum (VI) sulfate; Pre-hydrolyzed coagulants

1. Introduction

Selection of water treatment methods depends primarily on the quality of raw water and requirements which must be satisfied by treated water. Coagulation is one of the most important and

common unit processes used in surface water treatment technology. Properly conducted coagulation insures effective elimination of hydrophobic and hydrophilic colloids from water [1]. The former is responsible for water turbidity (clay, loams, etc.) and the latter for the color of water. Hydrophilic colloids include especially fulvic and humic acids. Humic substances can be derived from different sources, such

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as agricultural and wastewater return, aquatic organisms, and soil humic material [2–4]. Humic substances account for 60–90% of the dissolved carbon in natural water [5]. Content control of organic substances in water is very important because they are precursors of the oxidation by-products [6]. The effectiveness of a coagulation process depends primarily on: physico-chemical composition of water and technological parameters such as a dosage, a type of coagulant, pH, and temperature [7].

Aluminum and iron salts are widely used coagulants for removing dissolved impurities from water [8]. Most commonly used inorganic coagulants are aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3$), ferric chloride (FeCl_3), and ferric sulfate ($\text{Fe}_2(\text{SO}_4)_3$) [9]. The course of dissociation and hydrolysis of aluminum (VI) sulfate is presented in Table 1.

Added to the water chlorides or sulfates of aluminum, depending on the pH, may form the following monomers: Al^{3+} , $\text{Al}(\text{OH})^{2+}$, $\text{Al}(\text{OH})_2^+$, $\text{Al}(\text{OH})_3$, and $\text{Al}(\text{OH})_4^-$. Besides, various polymers are formed having the general formula: $\text{Me}_n(\text{OH})_y^{3n-y}$. In alkaline conditions, connections with negative charge are dominated, while in acidic environment—polyocations [13]. Aluminum sulfate is a well-known coagulant which has been used at water treatment plants for many years. However, water quality guidelines become more stringent, and it is needed to find more effective solutions of water treatment such as changing the chemistry of the coagulation process by introducing a new type of pre-hydrolyzed coagulants.

Pre-hydrolyzed coagulants are increasingly replacing nonhydrolyzed coagulants. They differ from nonhydrolyzed coagulants regarding the possession of a hydroxyl group, which determines their higher basicity [14]. It is believed that they are more effective and less sensitive to changes in pH and temperature [15–20]. They exhibit a high ability to neutralize the negatively charged particles present in the purified water due to the large positive surface charge. The structure of the precipitate during hydrolysis forms of aluminum is different in the case of pre-hydrolyzed coagulants from aluminum (VI) sulfate [21]. Poly-aluminum chlorides and sulfates are classified as

pre-hydrolyzed reagents. In the polyaluminum chloride solutions, additionally presence of many hydrolyzed aluminum hydroxy complexes and polymer “A₁₃” $[\text{Al}_{13}\text{O}_4(\text{OH})_{24}]^{7+}$ was noted [22]. This polymer is considered as the most facilitating, stable, and effective in the destabilization among all aluminum polymers [23]. Polyaluminum chlorides represented by the general formula $\text{Al}_n(\text{OH})_m\text{Cl}_{3n-m}$ are primarily used. The alkalinity of polyaluminum chlorides is defined by the coefficient r and is treated as a measure of polymerization degree [24].

$$r = [\text{OH}^-]/[\text{Al}^{3+}]$$

There is a following dependence between r and coagulant alkalinity:

$$\text{Alkalinity } [\%] = \frac{r}{0.03}$$

Inorganic flocculants are primarily used for drinking and industrial water purification [25,26]. They are also used in clarification of wastewater. In the case of chemicals, paper and food processing, and textile industries, polymeric inorganic flocculants are used for wastewater treatment [27]. Currently, there are more literature data on research conducted on new combined dual-coagulant, which can be more effective than a single coagulant [28–31].

The aim of the present study was to compare the effectiveness of coagulation process carried out using classical nonhydrolyzed coagulant—aluminum (VI) sulfate and pre-hydrolyzed coagulants: PAX XL 19H and Flokor 1.2A.

2. Materials and methods

2.1. Materials

2.1.1. Water used to coagulation

Water used in a coagulation process was collected once (single collection in one day) from selected water treatment plant in southern Poland after pre-ozonation process. Instantaneous samples were taken in the winter season—in February. Due to the fact that the water quality indicators changed according to the seasons, it is necessary to conduct further research also in the summer season in order to verify the effectiveness of pre-hydrolyzed coagulants. Analyzed samples were grab samples. Water samples for analysis were collected from waterworks taps before coagulation. The samples were stored at +4°C.

In the chosen water treatment plant, there are two production lines operating simultaneously. Water for

Table 1

Aluminum hydrolysis reactions and solubility constants for Al^{3+} for zero ionic strength and 25°C [10–12]

Reactions	K_S
$\text{Al}^{3+} + \text{H}_2\text{O} \rightleftharpoons \text{Al}(\text{OH})^{2+} + \text{H}^+$	4.95
$\text{Al}(\text{OH})^{2+} + \text{H}_2\text{O} \rightleftharpoons \text{Al}(\text{OH})_2^+ + \text{H}^+$	5.60
$\text{Al}(\text{OH})_2^+ + \text{H}_2\text{O} \rightleftharpoons \text{Al}(\text{OH})_3 + \text{H}^+$	6.70
$\text{Al}(\text{OH})_3 + \text{H}_2\text{O} \rightleftharpoons \text{Al}(\text{OH})_4^- + \text{H}^+$	5.60

treatment is collected from two independent water supply sources. Raw water transferred to the first line is derived from one surface water intake (dam reservoir). In the case of the second line, water is taken from two independent surface sources. Water samples for analysis were collected after pre-ozonation in first treatment line, Fig. 1.

Selected indicators of water quality used for coagulation are presented in Table 2.

2.1.2. Coagulants

In research studies, three coagulants were used:

- $Al_2(SO_4)_3 \cdot 14H_2O$ —ALK, in the solid form,
- two pre-hydrolyzed coagulants:
 - polyaluminum chloride—PAX XL 19H,
 - aluminum chloride hydroxide—Floklor 1.2A.

Both ALK and PAX XL 19H were produced by KEMIPOL in Police (Poland). Floklor 1.2A was manufactured by DEMPOL-ECO in Opole (Poland).

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

Table 2

Average values of selected indicators of water quality intended to coagulation

Indicator	Value
Temperature, °C	8.9
pH	6.61
Acidity, mval/L	0.20
Alkalinity, mval/L	1.60
True color, mg Pt/L	7
Turbidity, NTU	7.29
Absorbance UV_{254nm}^{1m} (unfiltered samples), m^{-1}	8.5
Absorbance UV_{254nm}^{1m} (filtered samples*), m^{-1}	5.2
Absorbance UV_{272nm}^{1m} (unfiltered samples), m^{-1}	7.6
SUVA, $m^3/g\ Cm$	2.02
Aluminum, mg Al/L	0.044

Note: * filtered through a 0.45 μm membrane filter.

2.2. Experimental procedure of coagulation

The study in a laboratory scale was carried out with the usage of a six-beaker flocculator. The process of volumetric coagulation was conducted in glass beakers with a capacity of 2 L. Each beaker was filled with 1 L of sampled water. Then, suitable doses of coagulants were added and were quickly mixed with a mechanical stirrer within 3 min (with 200 rpm) followed by slow mixing within 30 min (30 rpm). Afterwards, the

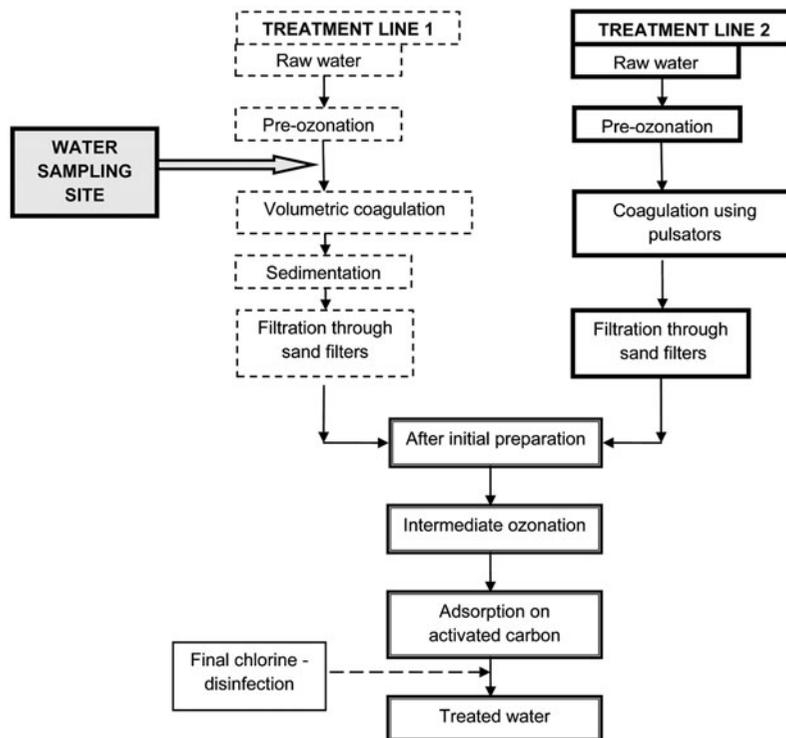


Fig. 1. Technological scheme of chosen water treatment plant with marked water sampling site.

Table 3
Characteristics of tested coagulants

Parameter	Coagulant		
	ALK	PAX XL 19H	FLOKOR 1.2
Density (20°C), g/cm ³	1.580	1.340	1.280
pH	3.4	3.5	4.2
Basicity, %	0.0	85.0	80.0
[Al], wt. %	9.1	12.5	11.0
[Al ₂ O ₃], wt. %	17.20	23.60	20.79
[Cl], wt. %	0.0	8.5	7.0
Viscosity, mPas	0	20	115

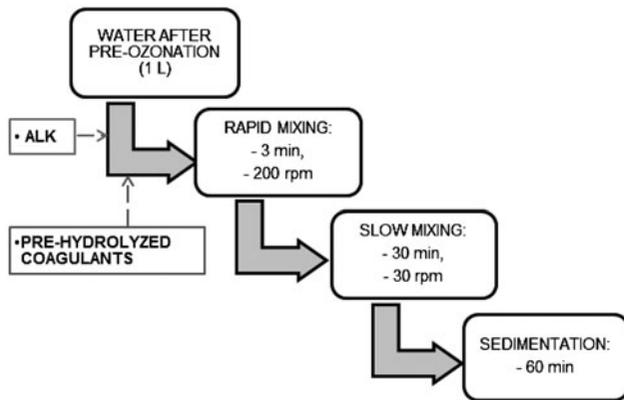


Fig. 2. Scheme of coagulation process.

samples were subject to sedimentation for 60 min. Successive stages are presented in Fig. 2. After sedimentation, 0.6 L of water was decanted for analysis.

The applied doses of coagulants are presented in Table 4. Doses of aluminum (VI) sulfate ranged from 1.6 to 2.6 mg Al/L. In case of pre-hydrolyzed aluminum coagulants, this range was 0.2–1.4 mg Al/L.

Furthermore, an additional series of tests was performed for the same dose for all coagulants (1.7 mg Al/L).

2.3. Analytical methods

Designation of pH was made by potentiometric method. A titrimetric method was used for determina-

Table 4
Doses of tested coagulants

Coagulant	Dose (mg Al/L)					
	I	II	III	IV	V	VI
Aluminum sulfate – ALK	1.6	1.8	2.0	2.2	2.4	2.6
PAX XL 19H	0.2	0.4	0.6	0.8	1.0	1.2
FLOKOR 1.2A	0.4	0.6	0.8	1.0	1.2	1.4

tion of total acidity and alkalinity of water samples. True color was indicated in accordance with ISO 7887—Method C [32], using a spectrophotometer M501 Camspec Ltd. Quartz cuvette with path length of light 50 mm was used. It was determined after filtration of the water sample through a membrane filter of pore size 0.45 μm. Color of the sample was calculated using following equation:

$$C = \frac{A_{410}}{ad} [\text{mg Pt/L}]$$

where C is true color of the sample, A_{410} —absorbance of the sample at $\lambda = 410$ nm, a —specific absorption coefficient of the calibration solution of potassium hexachloroplatinate and cobalt chloride [$\text{mm}^{-1}(\text{mg Pt/L})^{-1}$], d —optical pathlength [mm] [32].

Turbidity was designated using a turbidity meter—the nephelometric method. Determination of UV_{254} absorbance was performed in accordance with the method given by US EPA [33] using a spectrophotometer Camspec M501 Ltd. Quartz cuvette with path length of light 10 mm was used. For DOC analysis, the sample was filtered through a membrane. DOC concentration was performed using a carbon analyzer TN/TC model multi NC produced by Analytik Jena. DOC and UV_{254} are used in the calculation of the specific UV absorbance (SUVA).

$$SUVA = \frac{UV_{254nm}^{1m}}{DOC} [\text{m}^3/\text{g C m}]$$

where SUVA is specific UV absorbance at 254 nm and DOC—dissolved organic carbon [$\text{g C}/\text{m}^3$] [33].

Aluminum concentration in water samples was determined with the method of inductively coupled plasma atomic emission spectrometry using Perkin Elmer Optima 8000 spectrometer. Measurements were performed at a wavelength of 396.153 nm.

Determinations of all indicators selected to the analysis were performed in three replications.

2.4. Statistical analysis

Effectiveness of coagulants was compared on the basis of concentration of selected water quality indicators (turbidity, absorbance at 254 nm) after coagulation process (for optimal doses). For the assessment, if the differences were statistically significant, Student's t test for difference of two independent trials was used. The number of results in both trials was the same. The following formula was used in calculation:

$$t_d = \frac{|\bar{x}_1 - \bar{x}_2|}{\sqrt{\frac{\sum_{i=1}^n (x_{i1} - \bar{x}_1)^2 + \sum_{i=1}^n (x_{i2} - \bar{x}_2)^2}{2(n-1)}}} \cdot \sqrt{\frac{n}{2}}$$

where t_d —the value calculated from the experimental data, t_t —theoretical value read from the table at the appropriate level of significance α and the number of degrees of freedom, \bar{x}_1 —average value of the first trial, \bar{x}_2 —average value of the second trial, x_{i1} —the individual results of the first trial, x_{i2} —the individual results of the second trial, n —number of results in the trial, $2(n-1)$ —the number of degrees of freedom [34].

Critical value for four degrees of freedom ($n=3$) and confidence level (95%) was adopted using tables. Value from tables of t -Student amounts $t_t = 2,776$.

3. Results and discussion

3.1. Determination of the optimal dose of tested coagulants

Optimal coagulant doses were determined on the basis of selected indicators of water quality, such as color, turbidity, and absorbance at 254 nm. The changes of color values for investigated coagulants are presented in Fig. 3. In Figs. 4 and 5, the values of turbidity and absorbance at 254 nm were shown, respectively. Points marked on bright gray indicate the optimum dose of each coagulant.

The coagulation performance was examined with a range of dosages 0.2–1.2 mg Al/L for PAX XL 19H and 0.4–1.4 mg Al/L for Flokor 1.2A. In the case of aluminum (VI), sulfate ranged from 1.6 to 2.6 mg Al/L. The lowest optimal dose on the basis of color was obtained for Flokor 1.2A. It amounted to 0.8 mg Al/L.

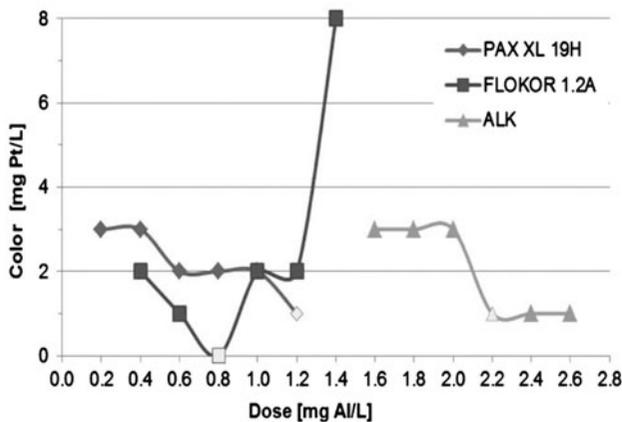


Fig. 3. Optimal doses of selected coagulants designated on the basis of color.

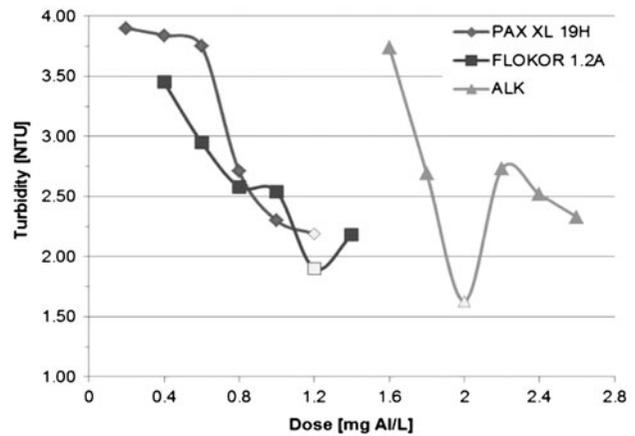


Fig. 4. Optimal doses of selected coagulants designated on the basis of turbidity.

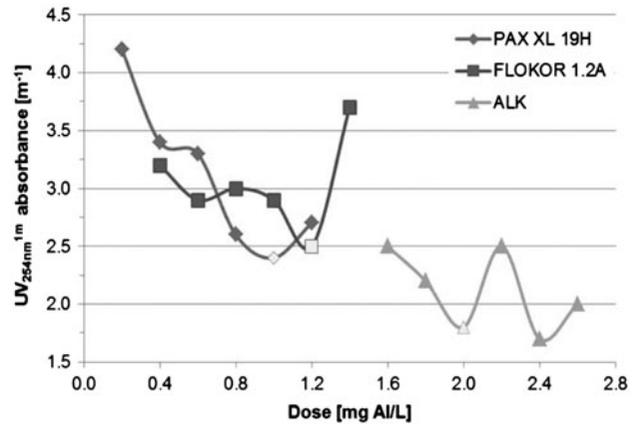


Fig. 5. Optimal doses of selected coagulants designated on the basis of UV₂₅₄ absorbance.

While for ALK and PAX XL 19H, these doses were 2.2 and 1.2 mg Al/L, respectively. Additional series of tests were performed for the same dose for all coagulants (1.7 mg Al/L).

In the case of turbidity, lower doses were also obtained for the pre-hydrolyzed aluminum coagulants. The optimal dose for both tested coagulants was 1.2 mg Al/L. For ALK, it was 2.2 mg Al/L.

Based on a comparison of the UV₂₅₄ absorbance, the lowest optimal dose was obtained for the PAX XL 19H (1.0 mg Al/L). For Flokor 1.2A, it amounted to 1.2, while for ALK 2.0 mg Al/L.

Additional series of tests were performed for the same dose for all coagulants (1.7 mg Al/L). These studies revealed that in the case of pre-hydrolyzed coagulants, doses above 1.6 mg Al/L are too high and lead to a breakdown of the coagulation process.

Analysis of the results made it possible to determine the following optimal doses (on the basis of their color, turbidity, and UV absorbance at 254 nm) of investigated coagulants:

- ALK: 2.2 mg Al/L,
- PAX XL 19H: 1.2 mg Al/L,
- FLOKOR 1.2A: 1.2 mg Al/L.

The highest value of the three received optimal doses for the various indicators was adopted. Optimal doses of pre-hydrolyzed coagulants were much lower than in the case of aluminum (VI) sulfate. In the next section, there are some results of water samples analysis after the coagulation process using the optimum dose of each of examined coagulants.

Dependency between UV absorbance at 254 nm and the value of turbidity is presented in the Fig. 6(A–C).

There was a clear linear correlation between the two examined indicators in the case PAX XL 19H, the coefficient of determination amounted to 0.87. For ALK, a moderate linear correlation was observed, and the determination coefficient was equal to 0.74. In the case of Flokor 1.2A, a coefficient of determination was very low and amounted to 0.18.

3.2. Comparison of coagulants effectiveness

The values of analyzed indicators after the coagulation process using the optimal doses of selected coagulants: aluminum (VI) sulfate, PAX XL 19H, and Flokor 1.2A are presented in Table 5.

The lowest pH value was obtained for ALK. For the other two (PAX XL 19H and Flokor 1.2A) coagulants, value was higher and was in the range 7.40–7.47. Lower wear of alkalinity was obtained for pre-hydrolyzed aluminum coagulants. It was lower by 13 and 19%, for PAX XL 19H and Flokor 1.2A, respectively, than in the case of ALK. The use of optimal doses of the tested coagulants resulted comparable results in the removal of water true color (decrease by 71–86%), Fig. 7. The effectiveness of removal of water turbidity for ALK, PAX XL 19H, and Flokor 1.2A was 63, 70, and 74%, respectively. The lowest concentration of residual aluminum in the water samples was achieved after the coagulation process using Flokor 1.2A. This concentration was about 53% lower than in the case of ALK. However, in the case of all coagulants, concentration of aluminum after the coagulation process increases significantly in comparison with raw water. Further research is required to optimize the process parameters of coagulation, such as time of: rapid mixing, flocculation, and sedimentation.

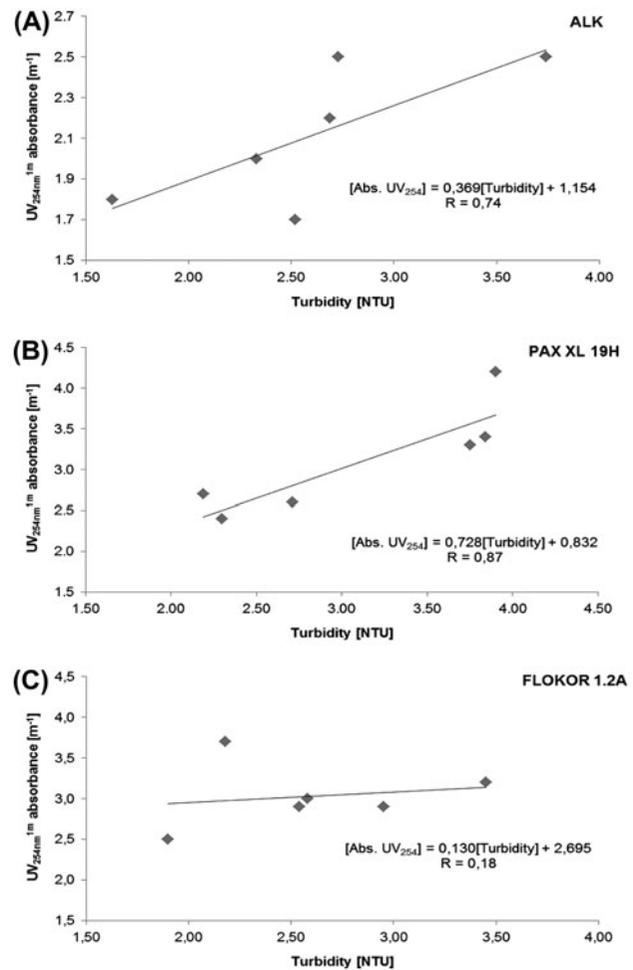


Fig. 6. Dependency between UV_{254nm}^{lm} absorbance and water turbidity for various coagulants: (A) ALK, (B) PAX XL 19H, and (C) Flokor 1.2A.

Optimization of these parameters may allow for reduction of the residual aluminum concentration to the limit value (0.2 mg Al/L).

The indicators of water contamination by organic substances as absorbance at wavelength 254 nm (unfiltered and filtered samples) and 274 nm (unfiltered samples) and SUVA are listed in Table 6. In the case of UV_{254nm}^{lm} measured in unfiltered samples, best efficiency was obtained for Flokor 1.2A (58%). In the case of UV_{254nm}^{lm} measured in filtered samples, the same effectiveness (52%) was obtained for the two coagulant ALK and Flokor 1.2A. The degree of removal of DOC in the coagulation process was small and amounted to 0.8–13%. The maximum value was obtained for Flokor 1.2A. SUVA value before coagulation was $2.02 \text{ m}^3/\text{g C m}$. The obtained results are confirmed by the literary data. Molczan et al. [35] reported that the SUVA value equals more or less to

Table 5
The values of analyzed indicators after coagulation process

Coagulant	Optimal dose (mg Al/L)	pH	Alkalinity (mval/L)	True color (mg Pt/L)	Turbidity (NTU)	Aluminum (mg Al/L)
ALK	2.2	6.49	1.30	1	2.73	0.868
PAX XL 19H	1.2	7.40	1.50	1	2.19	0.643
FLOKOR 1.2A	1.2	7.47	1.60	2	1.90	0.409

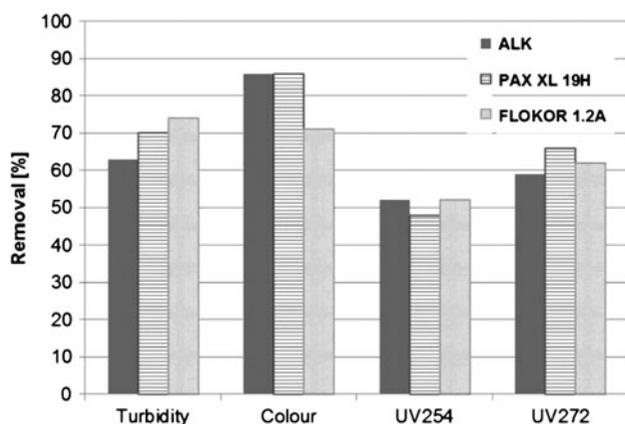


Fig. 7. The percentage of the removal of turbidity, color, and organic substances expressed as UV_{254nm}^{1m} (filtered samples), and UV_{272nm}^{1m} from water.

$2.0\text{ m}^3/\text{g C m}$ DOC removal in the coagulation process does not exceed 25%. On the basis of obtained results, it can be concluded that addition of pre-hydrolyzed aluminum coagulants, in comparison with aluminum

(VI) sulfate, is more effective destabilizing the negative colloids, which are primarily responsible for water turbidity and color.

Changes in concentrations of selected water quality indicators such as turbidity, absorbance at 254 nm (filtered samples), and absorbance at 272 nm (unfiltered samples) were compared by analyzing its concentration after the coagulation process using optimal doses of two different coagulants. Aluminum (VI) sulfate with PAX XL 19H, aluminum (VI) sulfate with Flokor 1.2A, and PAX XL 19H with Flokor 1.2A were compared. Table 7 shows the values of Student's t test which determine the significance of different coagulant in the same process. The resulting statistical analysis showed that differences in tested coagulants were not statistically significant in case of:

- ALK vs. FLOKOR 1.2A: absorbance at 254 nm and 272 nm,
- PAX XL 19H vs. FLOKOR 1.2A: absorbance at 272 nm.

In those cases, calculated values were lower than theoretical ($t_d < t_i$).

Table 6
The values of selected indicators of organic pollutants

Coagulant	Optimal dose (mg Al/L)	Absorbance UV_{254nm}^{1m} (unfiltered samples) (m^{-1})	Absorbance UV_{254nm}^{1m} (filtered samples) (m^{-1})	Absorbance UV_{272nm}^{1m} (unfiltered samples) (m^{-1})	SUVA ($\text{m}^3/\text{g C m}$)
ALK	2.2	4.1	2.5	3.1	1.06
PAX XL 19H	1.2	4.8	2.7	2.6	1.13
FLOKOR 1.2A	1.2	3.6	2.5	2.9	1.19

Table 7
Values of student- t distribution (t_d) with $t_i = 2.776$

Coagulant	Value of student- t distribution (t_d)		
	Turbidity	Absorbance UV_{254nm}^{1m} (filtered samples)	Absorbance UV_{272nm}^{1m} (unfiltered samples)
ALK vs. PAX XL 19H	18.462	4.243	3.474
ALK vs. FLOKOR 1.2A	87.338	1.414	1.414
PAX XL 19H vs. FLOKOR 1.2A	9.330	2.828	2.012

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

4. Conclusions

The studies performed on the effectiveness of non-hydrolyzed aluminum (VI) sulfate and pre-hydrolyzed aluminum coagulants PAX XL 19H and Flokor 1.2A led to the following conclusions:

- (1) The use of pre-hydrolyzed aluminum coagulants improves the efficiency of the coagulation process, and reduces the wear of coagulants in comparison with previously used aluminum (VI) sulfate.
- (2) After application of Flokor 1.2A, aluminum chloride hydroxide with the 80% basicity, the best results (among the investigated coagulants) in the removal of chosen contaminant were obtained: more than 74% decrease of turbidity and 13% reduction of DOC.
- (3) The lowest concentration of residual aluminum in the water samples was after the coagulation process using Flokor 1.2A.

In addition, it is necessary to estimate the costs of changing the coagulant from aluminum (VI) sulfate for pre-hydrolyzed aluminum coagulants. This will make it possible to assess whether the change of coagulant beyond improved water quality will also reduce the operating costs of a water treatment plant.

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Symbols

ALK	—	aluminum(VI)sulfate in the solid form
C	—	true color of the sample
A_{410}	—	absorbance of the sample at $\lambda=410$ nm
a	—	specific absorption coefficient of the calibration solution of potassium hexachloroplatine and cobalt chloride [$\text{mm}^{-1}(\text{mg Pt/L})^{-1}$]
d	—	optical pathlength [mm]
SUVA	—	specific UV absorbance at 254 nm
$\text{UV}_{254\text{nm}}^1$	—	absorbance at 254 nm [m^{-1}]
DOC	—	dissolved organic carbon [g C/m^3]
t_d	—	the value calculated from the experimental data

t_t	—	theoretical value read from the table at the appropriate level of significance α and the number of degrees of freedom
\bar{x}_1	—	average value of the first trial
\bar{x}_2	—	average value of the second trial
x_{i1}	—	the individual results of the first trial
x_{i2}	—	the individual results of the second trial
n	—	number of results in the trial
$2(n-1)$	—	the number of degrees of freedom

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