

# The use of turbidity and particle number measurements in monitoring filtrate quality in conventional water treatment with polyaluminium chloride

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## Abstract

The excessive amount of hydrolysis products of PACl (polyaluminium chloride) results in the formation of a large number of very small non-agglomerated particles (1–2  $\mu\text{m}$ ) when reaching and exceeding the isoelectric point. It was found that the residual aluminium occurred mostly in the form of  $\text{Al}_{13}$  species. In the study the influence of PACl overdosing on filtrate quality was tested in the aspect of the choice of the best tool to control treatment process. It was reported that although the filtrate turbidity was compliant with drinking water requirements and did not exceed 0.37 NTU, the coagulant hydrolysis products in the form of fine particles were not all retained in the filter bed and were observed in filtrate. Non-agglomerated particles were especially observed in the initial phase of filtration run. There was no direct correlation between the number of fine particles and turbidity in filtrate e.g. if to compare filtrate samples of similar turbidity (sample 2 – filtrate turbidity was 0.21 NTU and sample 5 with turbidity 0.17 NTU). The number of 1  $\mu\text{m}$  particle was 82  $1/\text{cm}^3$  and 1184  $1/\text{cm}^3$  for sample 2 and 5, respectively. Contrary to turbidity measurement, particle count allows to identify the negative results of PACl overdosing.

## Keywords

conventional treatment, particle number, turbidity, coagulation control

## 1. INTRODUCTION

Coagulation with hydrolysing coagulants has been studied by many researchers, but the coagulation behaviour of pre-hydrolysed coagulants such as polyaluminium chloride (PACl), especially the coagulation mechanisms has not been systematically investigated (Duan and Gregory, 2003; Wei et al., 2015; Ye et al., 2007). Different coagulation mechanisms occur for aluminium sulphate (alum) and PACl (Wu et al., 2007). Pre-hydrolysed coagulant is becoming more and more popular in the coagulation/flocculation process. PACl has many benefits over conventional hydrolysing coagulant i.e. much better performance at low temperature, less aluminium residuals, less effect on the pH value of raw water, and more rapid flocculation (Aguilar et al., 2002; Gregory and Dupont, 2001; Van Benschoten and Edzwald, 2007; Yang et al., 2010; Yang et al., 2011; Yu et al., 2007). PACls effectively remove contaminants and form larger and more robust flocs which can settle more rapidly.

Recently, polyaluminium coagulants are very useful in drinking water treatment. Many types of polyaluminium coagulants are commercially available for water treatment. PACl is an inorganic pre-polymerised hydroxyl aluminium cationic polymer. It is formed by the slow titration of alkali to aluminium salts, resulting in the formation of positively charged polymers of aluminium hydroxide. The ratio of polymers to monomers and

the stability of the polymeric species, are dependent on the conditions of the production process. That is why products can differ in their basicity (degree of neutralization). Basicity is calculated as the molar ratio of  $[\text{OH}^-]$  to  $[\text{Al}_T]$  in the PACl (O'Melia and Dempsey, 1982; Snoeyink and Jenkins, 1980; Wang et al. 2002; Zarchi et al., 2012).

Theoretically, the molar ratio can vary within the range of 0–3, which corresponds to changes in basicity from 0% to 100%. In fact, the ratio of the number of moles of  $\text{OH}^-$  to  $\text{Al}^{3+}$  is less than 3.0, which means that among the polyaluminium chloride hydrolysis products, in addition to  $\text{Al}(\text{OH})_3$ , aluminium monomers and polymers are also present. The number of monomers and polymers in polyaluminium chloride solutions is different and depends mainly on the basicity value of the coagulants.

PACl coagulants, contrary to aluminium sulphate, contain pre-polymerised aluminium hydroxide complexes which also appear during hydrolysis in coagulated water. These complexes are more stable. The hydrolysis process leads to the formation of various aluminium species, including monomers, dimers, and larger polynuclear complexes. The specific aluminium species formed during hydrolysis can influence the effectiveness of coagulation. The most effective specie of PACl hydrolysis products is tridecamer  $\text{Al}_{13}$  (polycationic species with a high positive surface charge  $[\text{AlO}_4\text{Al}_{12}(\text{OH})_{24}(\text{H}_2\text{O})_{12}]^{7+}$ ). The maximum share of  $\text{Al}_{13}$  is determined for basicity of

about 70% (some sources indicate 80%), which corresponds to the value of  $r = 2.1$ . Further increase in the basicity of the reagent causes a decrease in the share of these forms (Edzwald and van Benschoten, 1990; Lu et al., 1999; Pernitsky and Edzwald, 2006; Wang et al., 2004; Yang et al., 2010; Zhao et al., 2009; Zhao et al., 2010).

Most studies have focused on the efficiency of PACI during coagulation/flocculation–sedimentation processes, while hardly any have examined subsequent rapid filtration. In the paper the measurement of turbidity and particle count was examined in the aspect of the most reliable method of assessing the quality of filtrate in a conventional coagulation system with the use of highly – polymerised polyaluminium chloride.

## 2. MATERIALS AND METHODS

### 2.1. Experiment procedures

The study was conducted in the pilot treatment system and in the full-scale water treatment plant. The pilot installation comprised a double section flocculation tank and a lamella settling tank, preceded by rapid mixing using a static mixer. After rapid mixing, in collected samples streaming current value and particle number were measured. The system operated continuously at the flow  $0.75 \text{ m}^3/\text{h}$ . Flocculation time was approximately 20 minutes and hydraulic load of the settling tank was  $1.5 \text{ m}^3/\text{m}^2\text{h}$ . The study was conducted for 8 weeks.

The study in the pilot system was carried out to evaluate the coagulation mechanism based on SC (streaming current) values and particle number. Streaming current measurement allows to analyse the impact of a coagulant dose on the zeta potential value as an indicator of colloid stability. Based on the relationship between the SC value and the coagulant dose, the ranges of a coagulant dose with different coagulation mechanisms (charge neutralization, sweep coagulation) may be determined.

The aim of the study in the technical scale was to compare turbidity and particle count measurements in the aspect of monitoring filtrate quality in conventional treatment with polyaluminium chloride. The technical – scale study was conducted at the selected water treatment plant for 6 weeks. For the maximum capacity of the treatment system, i.e.  $13,000 \text{ m}^3/\text{d}$ , the flocculation time is about 20 min, while for the nominal capacity it is about 35 min. The hydraulic load of the settling tanks is in the range of  $0.8$  to  $1.5 \text{ m}^3/\text{m}^2\text{h}$ . The filtration rate is  $4 - 5 \text{ m}/\text{h}$ . The technological process consisted of a coagulation process carried out in the rapid mixing tanks with mechanical stirring, which was followed by flocculation carried out in the hydraulic mixing tanks combined with the vertical settling tanks. Clarified water supplied the gravity rapid filters. The reliability of turbidity measurements as an indicator of filtrate quality was analysed. Particle number was a comparative measurement.

Treatment effectiveness was assessed based on turbidity. Additionally, particle number and particle size distribution as supplementary indicator was measured. When a particle size is less than  $1 \mu\text{m}$ , turbidity is especially suitable for monitoring water quality. However, turbidity measurement is not a reliable tool for water quality analysis if larger particles are present in the water.

In the pilot study, PSD (Particle Size Distribution) was analysed with a particle size analyser (ARTI WPC21, Hach) which is an accurate particle counter with up to  $1 \mu\text{m}$  sensitivity. Streaming current values were measured with the SC analyser.

In the full-scale research, water samples were taken after each unit process and the number and size distribution of particles were determined with an analyser IPS LCW, Kamika Instruments. Turbidity was measured with Turbimax, Endress+Hauser. Total organic carbon was analysed with TN/TN Multi N–C analyser from Analytik Jena. TOC was measured as a monitoring parameter of raw water supplying treatment systems and the quality of filtrates. Absorbance  $\text{UV}_{254}$  was measured using a Hach DR5000 spectrophotometer. Each sample was measured three times. On those measurements the average value was calculated.

### 2.2. Reagents

The coagulant applied in the pilot – scale testing was a commercial product commonly used in water treatment plants in treatment process. Table 1 presents the basic characteristics of the tested coagulant.

Table 1. Basic characteristics of the tested coagulant (Floklor 1.2A, the manufacturer – DEMPOL-ECO, Poland).

Parameters	PACI
Specific gravity $[\text{g}/\text{cm}^3]$	1.28
pH	4.2
Al [% w.]	11
Cl [% w.]	7
Al/Cl	1.57
Basicity (OH/Al) [%]	80

### 2.3. Raw water

In the pilot study low polluted water was tested. The quality of water was quite stable. The basic indicators of water quality were on average:

- turbidity – 5 NTU,
- TOC –  $5.5 \text{ mg C}/\text{dm}^3$ ,
- absorbance  $\text{UV}_{254}$  –  $5.6 \text{ l}/\text{m}$ .

The detailed analyses of the raw water in chosen samples collected in the technical treatment system are presented in Table 2. The individual samples differed in the time of intake.

### 3. RESULTS AND DISCUSSION

#### 3.1. Pilot study

The change of SC value resulting from the increasing dose of PACI depends mostly on the surface charge of the hydrolysis products, which is used to neutralize the charge of colloidal compounds present in the water. The tested PACI doses were in the range 0.8–8.1 gAl/m<sup>3</sup>.

Figure 1 shows that with the increase of SC value, the number of 1 µm particles collected for analysis after 28 s of rapid mixing significantly increased when reaching and exceeding the isoelectric point which was noted at the dose 2 gAl/m<sup>3</sup> (SC value was –10 SCU). At that dose the number of 1 µm particles was about 200 1/cm<sup>3</sup> and gradually increased to approximately 19,000 1/cm<sup>3</sup> at the maximum coagulant dose 8.1 gAl/m<sup>3</sup> when the SC value was 57 SCU. The measurements of particle number of a larger size (2, 5 and 10 µm) did not show such changes. For comparison only the results for 10 µm particles are presented (Fig. 2). No noticeable changes for this particle size in relation to SC values were noted. PSD was not analysed for the tested doses because only fine particles of the size 1 µm, as it was confirmed in the technical scale study, are responsible for PACI overdosing.

No relationship was found between turbidity and particle count, both for 1 µm and 10 µm particle size. Figure 3 presents the relationship between turbidity and 1 µm particle number after rapid mixing in a static mixer. Due to the selectivity of turbidimeters (turbidity measurement is influenced by particles up to 1 µm size), there was no need to analyse the relationship between the 10 µm particle count and turbidity values. The increased number of 1 µm particles reflected a significant amount of non-agglomerated particles due to the same surface charge.

#### 3.2. The technical scale research

The PACI doses applied during treatment in the technical system were in the range of 3.2–4 gAl/m<sup>3</sup>. The dose of PACI was stated every day by the system operator. The raw water,

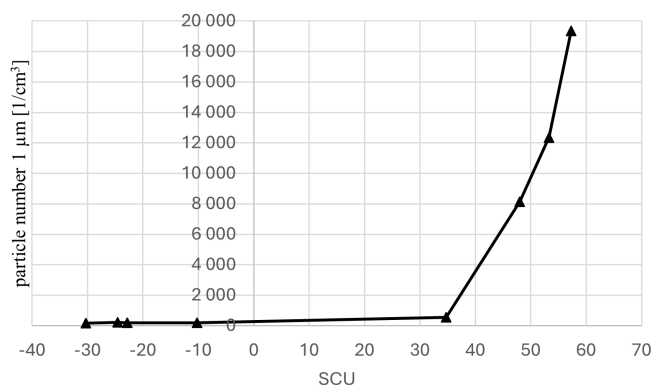


Figure 1. Relationship between SC (streaming current) value and 1 µm particle number after rapid mixing in a static mixer.

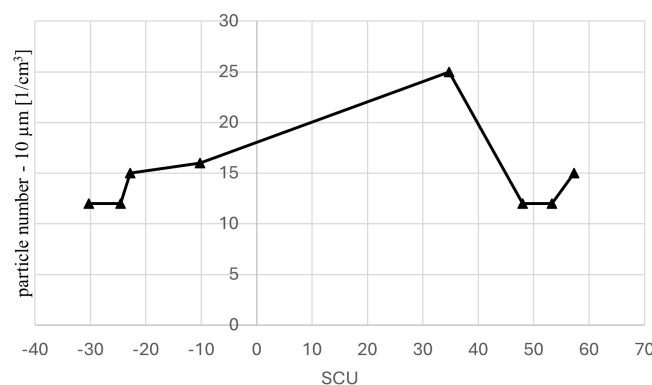


Figure 2. Relationship between SC (streaming current) value and 10 µm particle number after rapid mixing in a static mixer.

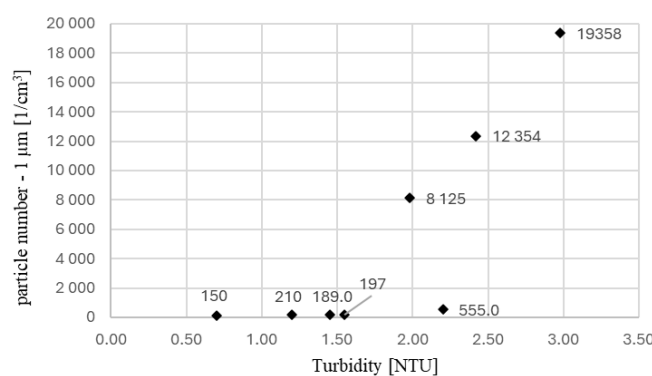


Figure 3. Relationship between turbidity and 1 µm particle number after rapid mixing in a static mixer.

Table 2. Raw water characteristics supplied to the technical water treatment plant for the selected samples.

Raw water quality	sample 1	sample 2	sample 3	sample 4	sample 5	sample 6
Turbidity [NTU]	5.3	3.8	5.9	4.2	3.7	5.7
Absorbance UV <sub>254</sub> [1/m]	21.3	21.5	22.9	20.5	20.3	15.2
Colour [gPt/m <sup>3</sup> ]	45	46	50	54	45	46
TOC [g/m <sup>3</sup> ]	5.47	5.01	5.11	4.95	4.76	4.7

Table 3. Filtrate quality collected in the technical system after treatment of raw water (samples 1–6).

Filtrate quality	sample 1	sample 2	sample 3	sample 4	sample 5	sample 6
Turbidity [NTU]	0.29	0.21	0.34	0.37	0.17	0.1
Absorbance UV <sub>254</sub> [1/m]	3.1	4.1	3.5	3.3	3.1	2.5
Colour [gPt/m <sup>3</sup> ]	4	4	4	4	5	3
TOC [g/m <sup>3</sup> ]	2.46	2.23	2.29	2.2	1.98	1.8

after settling and after filtration samples were taken every day for the analysis of the basic water quality parameters. Additionally, PSD was measured in the exemplary samples. Particle size distributions (PSDs) for 6 chosen samples of filtrates were analysed. For the sample 1 PSD for raw water, clarified water and filtrate was presented.

The detailed analysis of filtrate quality, collected in the technical system, is presented in Table 3.

The filtrate quality indicates high effectiveness of treatment process. However, when analysing the changes of particle number in treatment process, the adverse effects of PACl overdosing were observed which were not reflected in filtrate turbidity. It resulted in the increase of the residual aluminium in water after treatment processes. The increase of aluminium concentration is especially dangerous because polymeric species may not be detected in standard monitoring analysis.

The behaviour of particles in the initial stages of filtration after backwashing of granular bed (anthracite/sand filter bed) is complicated. Immediately after backwashing, larger particles and particles with lower surface charge are retained more effectively than smaller particles. The capture of particles is largely dependent on the amount of previously retained particles in the filter bed, and filtration efficiency increases with the increase in the previously retained contaminant load. Fig. 4 presents the exemplary PSDs after each unit processes where coagulant overdosing was observed.

The increase in the particle count in the water after sedimentation indicated non-optimal conditions for the simultaneous removal of dissolved organic matter and suspensions. Histograms of the particle distribution show that these are mainly particles with a size of up to 10 µm, of which the majority are particles with a diameter of up to 2 µm. This increase can be explained by too high concentration of PACl hydrolysis products. The increase in particle count in the water after sedimentation stage compared to the raw water had a direct impact on the operation of the filters. Non-agglomerated particles are very difficult to remove during sedimentation and rapid filtration process. Fig. 5 shows particle size distribution in water collected after rapid filtration in samples 2–6. Turbidity of filtrate was also presented.

The analysis of the water quality collected in the filter outflow indicates that although the filtrate turbidity was significantly lower than that required for drinking water and did not exceed 0.37 NTU in analysed samples, the number of fine particles indicated that not all particles of the post-coagulation suspension were retained in the filter bed (Fig. 5). Such a phenomenon was noticed in the sample number 1 and the sample 5 collected at the early stage of the filtration cycle, when the separation efficiency of the filter bed was the lowest. It should also be noted that there was no direct correlation between the number of particles and turbidity in filtrates, e.g. if to compare filtrate samples of similar turbidity (sample 2 – filtrate turbidity was 0.21 NTU and sample 5

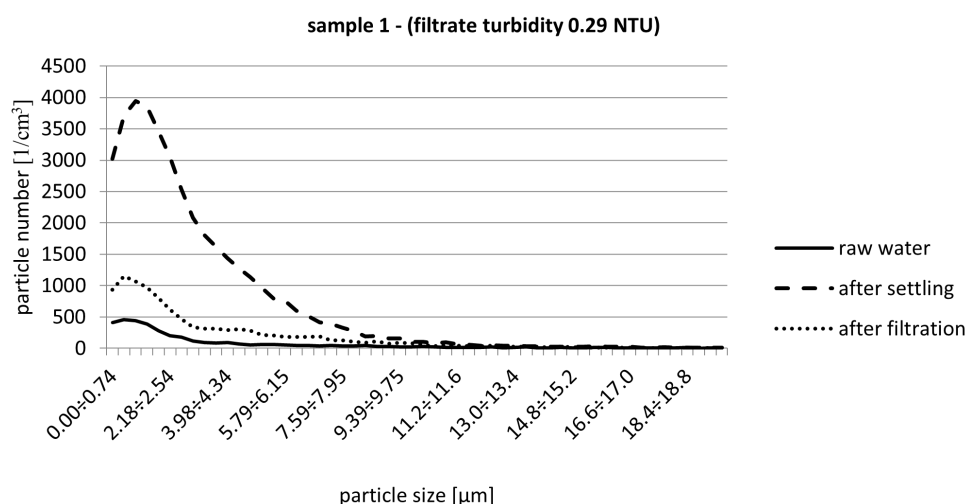


Figure 4. PSD after unit processes in PACl overdosing condition – sample 1.

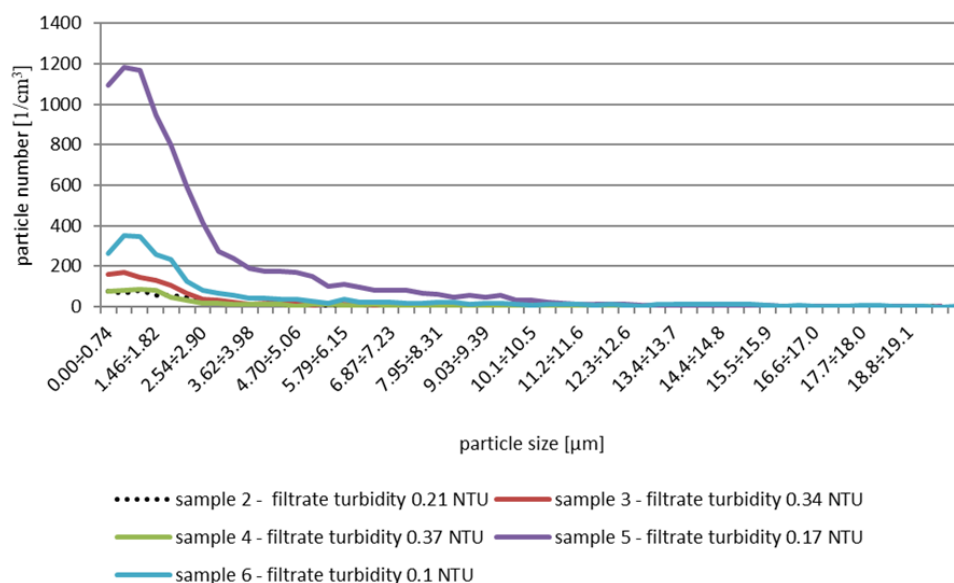


Figure 5. PSD and turbidity of filtrates – samples 2–6.

with turbidity 0.17 NTU). When analysing the particle size distribution in samples in which a significantly larger number of particles was recorded in relation to the other samples, clear discrepancies were noted. In the samples where the total number of particles was the lowest (samples 2, 3, 4, 6), most particles were of a size of 1–2  $\mu\text{m}$ .

In the case of pre-hydrolysed coagulants, due to the significantly higher surface charge of hydrolysis products compared to aluminium sulphate, the isoelectric point and the optimum dose of PACl for turbidity removal are significantly lower than the doses required for the removal of dissolved organic matter. This is because the surface charge of mineral particles is many times lower than the charge of natural organic matter (NOM) particles. In the pH range typical for most natural waters, the negative electric charge of mineral particles is in the range of 0.1 to 1  $\mu\text{eq}/\text{mg}$ , while the NMO charge is usually 10 to 100 times greater. If the coagulation process of mineral particles proceeds according to the charge neutralization mechanism, then the coagulant doses are proportional to the turbidity. The dose required to neutralise the contaminant charge does not allow for obtaining satisfactory removal efficiency of other contaminants, apart from turbidity. It follows that when using polymerised coagulants, the water purification process must be carried out based on the mechanism of sweep coagulation. The basic problem occurring during this mechanism of coagulation, resulting from the need to carry it out in a state of supersaturation of water with hydrolysis products, is the possibility of restabilisation of colloids due to too high concentration of hydrolysis products (Duan and Gregory, 2003). The increase of PACl dose exceeding the dose required to charge neutralization results in an increase in the number of non-agglomerated particles, which are very difficult to remove during sedimentation and rapid filtration. Only conducting the coagulation process based on the mechanism of sweep

coagulation, by appropriately increasing the coagulant dose, the efficiency of water clarification can be improved (Kłos and Gumińska, 2009). Therefore, when selecting coagulants, the criteria that are considered in the optimization of the process should be clearly defined.

The consequence of the appearance of a significant number of fine particles in the filtrate was periodic exceedances of aluminium concentration (determined by the atomic spectroscopy method). To identify the species of aluminium responsible for the increase in the number of fine particles,  $k$ -value-based ferron assay was carried out (Ye et al., 2009). Based on the reaction rate constants and the measured of  $\text{UV}_{370}$  absorbance values, it was calculated that the residual aluminium was of the highly polymerised species ( $\text{Al}_{13}$ ) in 89%, the remaining share i.e. 11% was the species of aluminium with a lower degree of polymerisation. When an excessive dose of PACl was applied, at the first stage of the filter run, a large amount of coagulant hydrolysis products in the form of fine particles appeared in the water supplying the rapid filter. During the filter bed ripening phase, these positively charged aluminium precipitates were retained on the surface of the filter bed grains and repelled positively charged non-agglomerated fine particles, present in water feeding the filter, preventing their retention. In that case, the filter could not operate properly. The process of filtration had to be cancelled to wash the filter bed. The studies have shown that the negative effect of PACl overdosing is also the shortening of the filtration run. The research, whose results are presented in Fig. 6, were carried out at a similar water treatment plant with the same technological processes and equipment as in the WTP analysed in this paper. These results show the influence of positively charged particles on separation efficiency of coagulant hydrolysis products and turbidity from water supplying the filter (Gumińska and Kłos, 2015).

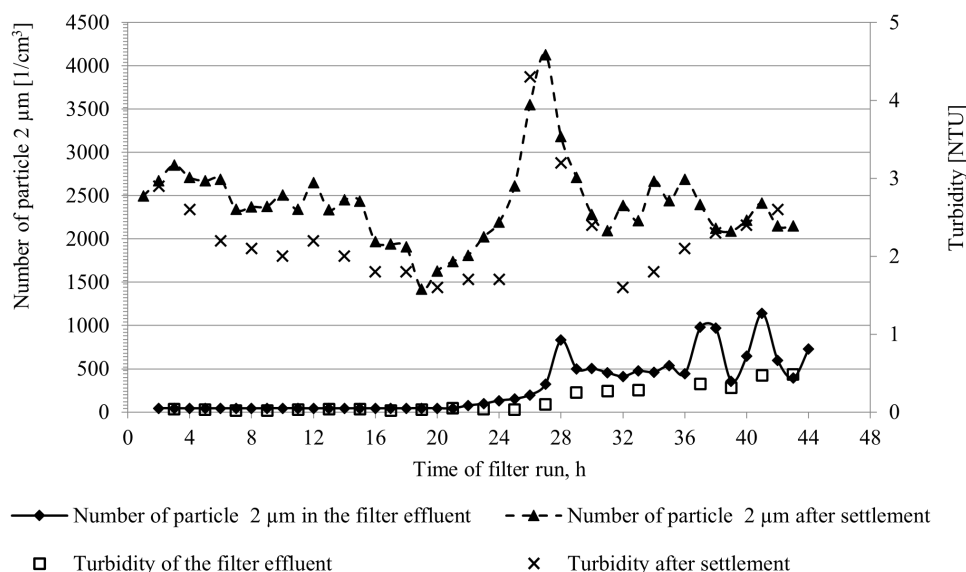


Figure 6. Particle number and turbidity measurements during the filter run (Gumińska and Kłos, 2015).

## 4. CONCLUSIONS

1. Pre-hydrolysed coagulant overdosing has negative effects on coagulation/flocculation and filtration process. It results from the formation of large quantities of fine non-agglomerated particles (1–2 μm particles) which are observed in the treated water. The consequence of PACl overdosing is an increase in the residual aluminium in water after treatment process. The increase of aluminium concentration is especially dangerous because polymeric Al species may not be detected in standard monitoring analysis.
2. Negative effects of PACl overdosing are not detected based on turbidity measurements but only using particle count measurement. Non-agglomerated particles are especially observed in the filtrate after backwashing, in the initial phase of a filtration run. When a large amount of PACl hydrolysis products in the form of fine particles appear in the filter influent, these particles create a coating with a positive electrical charge. As a result, the coating covering the filter bed repels the particles in the filter influent, preventing their retention.
3. Turbidity is one of the basic indicators of water quality that must meet the legal requirements for drinking water. Particle count measurements make it possible to control a coagulant dose. It is also the most reliable method of assessing the quality of filtrate in a conventional coagulation system with the use of highly – polymerised polyaluminium chloride.

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