Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2022.28372

254 (2022) 151–159
April

Analysis of a coagulation sludge contamination with metals using X-ray crystallography

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Received 27 December 2021; Accepted 24 February 2022

ABSTRACT

Characteristics of both surface and groundwater as well as water treatment itself determine properties of sludge produced as a by-product of technological processes. However, most of the currently available literature data and research focuses rather on the evaluation of water treatment processes, comparing the quality of raw water and the quality of water after treatment. The sludge produced at the water treatment plant, presented in the research, is the most environmentally sensitive by-product of the water treatment processes, that is, volumetric and contact coagulation with the use of coagulants. The chemical composition of the produced sludge varies, depending on the raw water quality and the type and amount of chemical reagents dosed. Two types of coagulants were dosed to the system over the period (year) of sludge sampling. They were: an aqueous solution of a mixture of polyaluminium chloride and a mixture of aluminum sulphate hydroxychloride and a complex solution of aluminum hydrochloride. Therefore, it is very important for the plant operators, to accurate determine the chemical composition of the sludge, especially in terms of the presence of metals in water. In the article, the authors offered an innovative approach in order to assess contamination of a coagulation sludge with selected metals. The work summarized several years of research conducted at the water treatment plant, which obviously increased the application value of the work. The authors described the principles the water treatment processes and tried to assess their effectiveness with a qualitative analyzes of sludge generated at particular stages of water treatment. The sludge was analyzed with qualitative methods of X-ray crystallography, that is, X-ray fluorescence and X-ray diffraction. Additionally, the laboratory and technological conditions required to proceed with these methods were presented. The methods, more accurate than in conventional laboratory, allow for a better determination of the metals presence in the sludge.

Keywords: Coagulation sludge; Coagulation; X-ray crystallography; Aluminum forms in sludge; Metals in sludge

1. Introduction

Implementation of the new Directive of the European Parliament and of the Council of Europe on the quality of water intended for human consumption (2020/2184) requires Member States to carry out a risk assessment and risk management approach regarding the quality of drinking water at every stage of its production and distribution. According to the Directive, the priority action to be taken is to minimize, or even eliminate, the risk of metal contamination. Such approach creates a space for testing water quality not only at the consumer ends but also at the intake and along the treatment line. In accordance with the Directive, Member States should implement special programs to monitor whether drinking water meets the

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Presented at the 2nd International Conference on Critical Infrastrcture of Cities (CIC 2021), 6–8 October 2021, Rytro, Poland

quality requirements; responsibility for monitoring will be largely borne by water suppliers. Water Safety Plans, as well as identification of potential hazardous events leading to the occurrence of hazards, and then risk assessment and risk management will be obligatory. These plans must cover virtually the entire supply system, that is, water source, water intake, then treatment lines and finally external and internal water distribution systems. The practical implementation of Directive 2020/2184 requires verification of opinions and an operating practice so that water suppliers (water supply companies) would eventually end up with a better knowledge about metals in order to effectively improve the water quality. The information would involve not only facts about the presence (concentration) of metals in water, but most of all about the forms of their occurrence (and other pollutants).

The presence of metals (and other pollutants) in drinking water results from a number of factors, often overlapping each other. They are usually involved with a contact with water on its way from the intake to the consumer and include:

- metal compounds in raw water – due to a geological structure of the area where water is withdrawn (mainly groundwater);
- metal compounds in intake waters – caused by a sewage discharge into the river above the cross-section with a water intake (mainly surface waters);
- lower content of these substances in water due to water treatment, for example, precipitation of some metals;
- increase of a metals and metalloid content during the water treatment process;
- increase of a metals and metalloid content during water distribution;
- introduction of metal compounds into water in households systems.

The issue of aluminum may be of a particular importance, since aluminum is not only present in natural waters but also serves as a popular coagulant. Apart from its positive effects, the coagulation process may be responsible for such changes in the physico-chemical composition of water [1]. Letterman and Driscollch [2] in their studies showed the beneficial effect of aluminum coagulants on the physical–chemical and bacteriological quality of treated water at the water companies in the United States. These studies have been also confirmed by other researchers, such as Rybicki and Wiewiórska [3], Tong et al. [4] and Wysowska et al. [5]. Additionally, Włodarczyk-Makuła et al. [6] have also confirmed a positive effect of coagulation on the removal of xenobiotics from water.

Aluminum coagulants may also increase the concentration of aluminum ions in drinking water, which may be potentially hazardous to human health. High concentrations of aluminum in food (including water) are likely to contribute to the development of, for example, Alzheimer's disease [3]. Strauber et al. [7] conducted some epidemic studies on a group of people drinking tap water treated with aluminum coagulant and clean water. It was shown that 1%–2% of the daily intake of Al came from alum treated drinking water and about 0.3%–0.4% of the Al in alum treated drinking water was absorbed by the body – the same percentage as that absorbed from food. It was estimated that drinking 1.6 L/d of alum treated water containing 140 μg Al/L would contribute only 0.4%–1.1% of the lifetime body burden of Al.

Ahmad et al. [8] highlighted some physical and chemical properties of water treatment sludge and the possibility of its reuse, as for example, coagulant in wastewater treatment, processes, adsorbent of pollutants and heavy metals, substrate in wetlands under construction and as a cement material and sand substitute during concrete and mortar production. It may also be used for co-conditioning and dewatering of sewage sludge, in cement, bricks, ceramics and artificial lightweight aggregates, in agricultural practice and in land applications. Researchers have found that the properties of the water treatment sludge largely depend on its chemical composition, which may have a major impact on its potential future reuse. They also pointed out that physical and chemical properties of the sludge varied along the treatment process line at water treatment plant (WTP).

Studies have shown that the use of pre-hydrolyzed aluminum coagulants, that is, polyaluminium chlorides (including PAC, PAX-18, PAX XL-3, PAX-XL61) instead of aluminum sulphate reduced negative effects of the coagulation process and improved process efficiency (especially for alkalinity ≥70) [9]. It was also found that concentrations of residual aluminum detected after coagulation with polyaluminium chloride were lower than after coagulation with aluminum sulphate. Zouboulis et al. [9] and Pernitsky, Edzwald [10] proved that a pH drop observed after coagulation with pre-hydrolyzed aluminum coagulants was much lower compared with 'conventional' alum [10] and coagulant aggressiveness decreased with an increase of their alkalinity. Such observations have been widely reported in literature, for example, [11]. Polymeric forms of aluminum in solutions of polychlorides of aluminum produce a coagulation sludge with better sedimentation properties and this way improve removal of aluminum-containing coagulation products [1]. The coagulation sludge contains also oxides of varying content. There are mostly SiO_2, then Al_2O_3, Fe_2O_3 (depending on the coagulant used (Al or Fe); the remaining oxides (CaO, MgO, Na_2O, K_2O, P_2O_5, TiO_2) accounts for a small percentage of sludge content. The studies show also that heavy metals may be present in the sludge; their source is either the raw water or the coagulant. A good knowledge of the origin and forms of aluminum compounds in water is necessary to assure a good quality safety.

The study presents the results of research carried out under the Project "Technologically advanced, intelligent (critical) infrastructure for collective water supply systems".

1.1. Scope

The research focused on a detailed analysis of the chemical composition of the coagulation sludge collected at the WTP using the X-ray fluorescence (XRF) method. Additionally, a chemical structure of compounds present in the sludge samples (in particular aluminum forms) was determined using X-ray diffraction (XRD) analysis. This way the coagulation processes parameters and operation conditions could be verified.

1.2. Plant description

The study was conducted during the day-to-day operation of the WTP at Stary Sącz; the plant is one of the three treatment plants operating within the company "Sądeckie Wodociągi" LLC. Until mid – 2011, the maximum daily flow of the Stary Sącz WTP was 7,500 m^3 and water was drawn from the Dunajec River and eight deep wells. The water treatment system comprised: open vertical settling tanks with volumetric coagulation and closed pressured filters (until the year 2002). From the year 2002, the pressured filters were substituted with the Dyna Sand filters and disinfection with a UV lamp was added to a regular disinfection with chlorine gas dosed to water and water tanks. After disinfection water was collected in two clean water reservoirs with total capacity of 600 m^3.

In 2011–2012, the WTP was rebuilt and upgraded to increase the plant's efficiency and improve the quality of treated water; the project was co-financed by the European Union funds. Currently, the Stary Sącz WTP supplies water to the agglomerations of Nowy and Stary Sącz. The current production of water meets the needs of residents and ranges from 8,000 to 10,000 m^3/d, with the maximum of 14,000 m^3/d. The WTP takes water from both the Dunajec River (116 + 000 km) and infiltration wells; the wells are supplied with a river water through infiltration basins. The water from the Dunajec River passes through a bottom infiltration intake and then is transported to two storage reservoirs; then it is pumped to two technological lines (soil infiltration and vertical settling tanks/filters at the WTP). Annually, about 100 tons of sludge are produced on the premises of the WTP.

2. Materials and methods

All the tests and studies were carried out under dynamic conditions in an actual scale. The plant maintained the continuous water supply and complied with the drinking water quality standards [9]. The dose of the coagulant is given as grams of active metal per unit of water volume.

Until mid-May of 2002, only aluminum sulphate (VI) was periodically dosed to the vertical settling tanks with volumetric coagulation. Then, a change of coagulant took place when the pressure filters were replaced with the DynaSand filters. Pre-hydrolyzed coagulants such as: PAX XL-3, PAX XL-19F, PAX XL-10 and Flokor 1.2A have been dosed to a volumetric coagulation, contact coagulation and to the lamella separator used for pretreatment of backwash water from sand filters.

Additionally, the coagulant dosing points were modernized and new technical solutions were introduced:

- possibility of dosing two different coagulants upstream of the primary settling tanks (the gate valve chamber) directly into the pipe with water from the Dunajec River, using two separate valves and installations feeding coagulant solutions;
- possibility of dosing two different coagulants by two separate valves and feeding installations upstream from contact filters;
- expansion of the coagulant dosing station; new dosing pumps and tanks for different types of coagulants are located in a separate room of the technological building;
- automation of the coagulant dosing process; algorithms controlling coagulant dosing pumps based on turbidity and flow rate of the Dunajec River water as well as after preliminary settling tanks. Measurements of flow (on-line) and water turbidity are carried out with a flow meter and a turbidimeter located in the gate valve chamber, ahead of preliminary settling tanks, and on the pipe in the technological hall ahead of sand filters;
- remote control of the coagulation process (dosage and type of coagulant) from the operator's station located in the WTP dispatch room.

During plant operation, the basic water quality parameters including the following metals: iron, chromium, zinc, cadmium, manganese, copper, nickel, lead, potassium, sodium and calcium were analyzed, using the inductively induced plasma atomic spectrometry method. Their initial identification served as the basis for further determination of aluminum concentrations in sludge, as some aluminum compounds could seep from the sludge into solution.

During the sludge sampling period (1 y), two types of coagulants were dosed successively (one after another) into the technological system: an aqueous solution of a mixture of polyaluminium chloride with the trade name PAX XL-10 and a mixture of aluminum sulphate hydroxychloride and a complex solution of aluminum chlorohydride with the trade name Flokor 1.2A.

The sludge samples for physical–chemical tests were collected at three points of the technological system (Fig. 1):

- after vertical settling tanks with volumetric coagulation (sludge settling time 20 h; sludge withdrawal – 10 min);
- after a lamella separator that treats backwash water from the Dyna Sand filters (sludge settling time – 240 min; sludge withdrawal – 15 min);
- from the sludge tank, where sludge from the entire WTP was stored (2–4 weeks).

Individual sludge samples (about 1 kg) were dried at 105°C in a thermal chamber, according to the PN-C-04616-01: 1975 standard. The analysis were done at the Laboratory of the Wastewater Treatment Plant owned by the Sądeckie Wodociągi LLC. Physical and chemical properties of dried and ground sludge collected in: March (March 15) – sample no. 1, May (twice: May 22 – sample no. 2 and May 28 – sample no. 3), October (October 2) – sample no. 4 and December (December 15) – sample no. 5, were analyzed at the Laboratory of the Faculty of Chemistry and Chemical Technology of the Institute of Inorganic Chemistry and Technology, Cracow University of Technology. The analyzes were conducted using the following methods:

- comprehensive thermal analysis combining differential thermal analysis (DTA) and thermogravimetric analysis (TG) and differential thermogravimetric analysis (DTG) using the EZTAR SII TG/DTA 7300 analyzer;
- elemental analysis of C, H and N performed with the PerkinElmer type 2400 CHN analyzer; the instrument

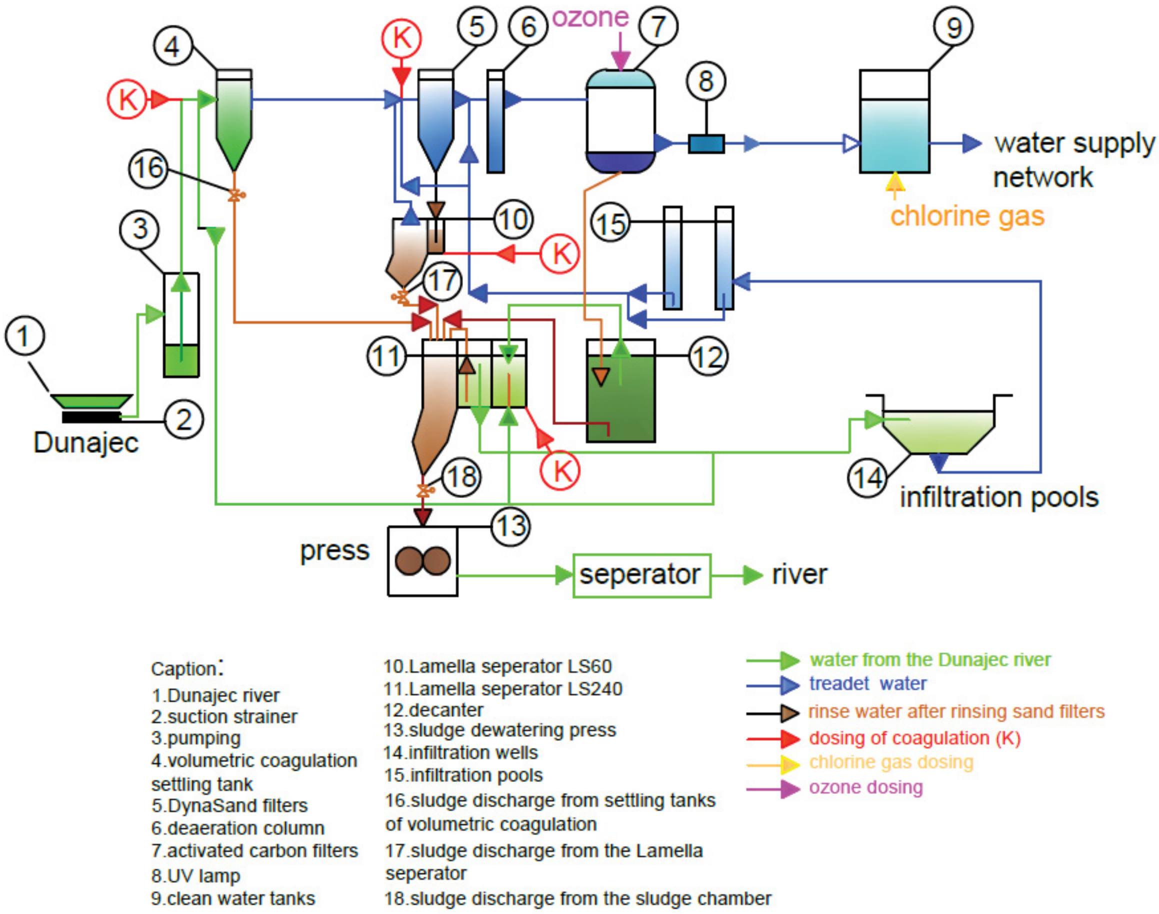

Fig. 1. Layout of the Stary Sącz WTP with coagulation sludge sampling points.

determines the mass percentage of chemical elements in the sludge sample;

- X-ray fluorescence (XRF) with the PANalyticalMinipal 4 apparatus; the analysis allows for identification of the particular elements in the sample and their percentage content;
- X-ray diffraction (XRD) using the PHILIPS XPert PW1830 analyzer; the analysis shows the composition of crystalline and liquid crystal structures, but does not detect amorphous (non-crystalline) substances in the sample.

Additionally, non-metallic inorganic parameters and extractable metals (mainly cations) were analyzed as well as percentage of C, H, N and TOC in samples no. 3 and no. 4 was measured. These particular samples were taken under extreme weather conditions, at high turbidity and color levels in raw water.

The analyzes were carried out in the accredited laboratories and followed the applicable standards and procedures. Solid samples were crushed, ground and pulverized within the following ranges: <0.07, <0.03 and <2 mm. The dry matter and moisture contents were determined according to CSN ISO 11465; the contents of TOC, carbon C and hydrogen H were determined by the combustion method with IR detection, while the nitrogen content by the TCD method, according to CSN EN ISO (CSN ISO 29541, CSN EN ISO 16994, CSN EN ISO 16948, CSN EN 15407, CSN ISO 19579, CSN EN 15408, CSN ISO 10694, CSN EN 13137).

The content of the particular elements was determined by inductively excited plasma atomic emission spectrometry, according to US EPA 2007, ISO 11885, US EPA 6010, SM 3120. Before the analysis, the samples were homogenized and mineralized in aqua regia, according to US EPA 3050, CSN EN 13657, ISO 11466 chap. 10.3 to 10.16, 10.17.5, 10.17.6, 10.17.9 to 10.17.14.

3. Results and discussion

The sludge produced at the WTP is a by-product of the technological process, that is, volumetric and contact coagulation with aluminum coagulants. The chemical composition of the sludge varies, depending on the quality of the raw water and the type and amount of dosed coagulant.

3.1. XRF results

The research focused on the detailed analysis of the chemical composition of the coagulation sludge samples using the XRF method. The results of the XRF analysis are presented in the tables (Tables 1 and 2) and in a diagram – a printout from the instrument (Fig. 2).

XRF analysis confirmed the presence of the following elements in the coagulation sludge samples: Al, Si, P, Mg, S, Cl, K, Ca, Ti, V, Cr, Mn, Co, Ni, Cu and Zn (Tables 2 and 3, Fig. 2). All sludge samples taken at the same location, that is, vertical settling tank, lamella separator or sludge tank showed a similar content (%) of elements; the value remained at a similar (constant) level throughout the calendar year. Silicon and aluminum showed the highest content in the sludge samples; it was 32.2%–47.9% and 17%–39%, respectively. This is due to the nature of the coagulation processes where precipitation of turbidity compounds (including silica) with aluminum coagulants is expected.

Then, the quantitative analysis (mg/kg of dry mass) of the elements in the sludge samples was carried out using atomic emission spectrometry with inductively excited plasma. The analysis such as: XRF, elemental C, H, N, quantitative elements, DOC and TOC proved that the levels of turbidity, color, aluminum, phosphorus, UVA_{254nm} absorbance, DOC

Table 1
Percentage of different elements in a coagulation sludge collected: after settling tanks with volumetric coagulation – O, after lamella separator – L and from the sludge tank – K

Day	Sampling point	Sample symbol	Content (%)							
			Mg	Al	Si	P	S	Cl	K	Ca
	O	OW1		30	40.3	0.81	0.85		4.84	7.17
15.03	L	OW2		30	41.0	0.77	0.79		4.81	7.30
	K	OW3		27	42.8	0.86	0.81		4.29	10.30
	O	OW1		24	46.0	0.57	0.46	0.59	6.59	5.15
22.05	L	OW3		33	39.3	0.56	0.77	0.60	6.16	5.81
	K	OW5		25	39.9	0.73	1.00	0.48	4.59	14.80
	O	OW2		23	47.7	0.53	0.29	0.45	6.73	4.51
28.05	L	OW4		35	37.1	0.70	0.87	0.54	5.31	6.72
	K	OW6		25	43.3	0.51	0.65	0.51	5.57	9.34
	O	B1	2	17	47.9	0.57	0.46	0.55	4.54	13.90
02.10	L	B2		36	35.5	0.94	1.50	0.70	3.01	11.00
	K	B3		26	42.2	0.75	0.98	0.60	3.79	13.50
	O	O		22	44.4	0.73	0.81	0.60	4.04	15.50
15.12	L	L		39	32.2	0.78	1.40	0.63	4.20	10.90
	K	K		24	42.9	0.67	0.87	0.59	4.10	14.60

Table 2
Percentage of different elements in a coagulation sludge collected: after settling tanks with volumetric coagulation – O, after lamella separator – L from the sludge tank – K

Day	Sampling point	Sample symbol	Content (%)							
			Ti	V	Cr	Mn	Fe	Ni	Cu	Zn
	O	OW1	1.020	0.072	0.020	0.440	14.30			
15.03	L	OW2	0.975	0.059	0.020	0.430	13.90			
	K	OW3	0.968	0.060	0.042	0.510	12.60			0.081
	O	OW1	1.250	0.085	0.043	0.420	14.20	0.03	0.030	0.066
22.05	L	OW3	1.020	0.079	0.041	0.390	12.20	0.03	0.040	0.066
	K	OW5	0.999	0.066	0.039	0.510	11.50	0.01	0.040	0.067
	O	OW2	1.290	0.091	0.044	0.370	14.40	0.03	0.030	0.078
28.05	L	OW4	1.010	0.060	0.041	0.480	12.20	0.02	0.047	0.064
	K	OW6	1.140	0.073	0.043	0.450	12.90	0.02	0.040	0.075
	O	B1	1.040	0.056	0.020	0.400	11.40		0.020	0.054
02.10	L	B2	0.630	0.046	0.030	0.978	9.46		0.050	0.110
	K	B3	0.885	0.064	0.020	0.610	10.70		0.030	0.050
	O	O	0.880	0.056	0.037	0.570	10.30		0.040	0.067
15.12	L	L	0.730	0.060	0.030	0.490	9.88		0.056	0.095
	K	K	0.900	0.059	0.030	0.530	10.60		0.030	0.050

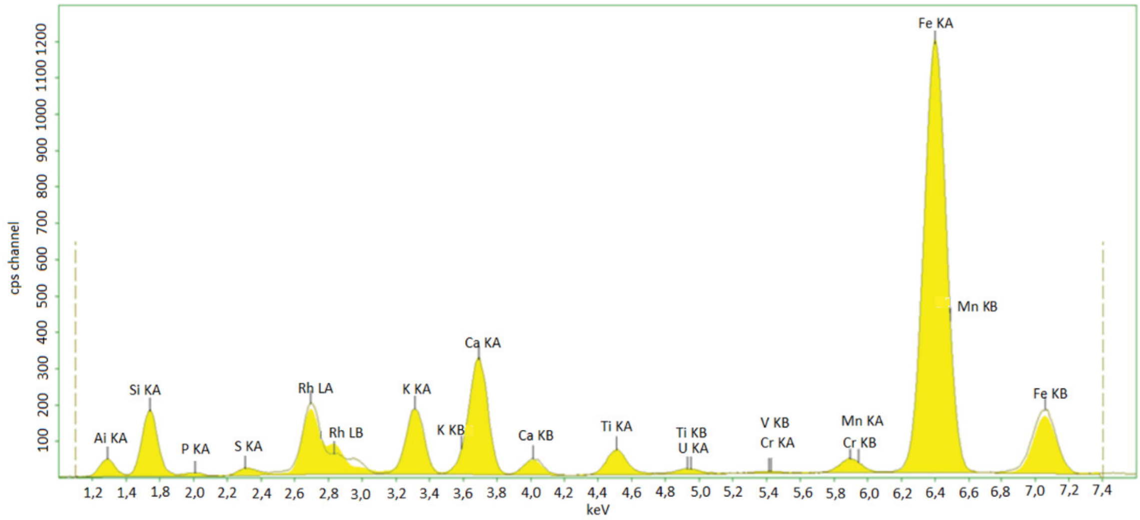

Fig. 2. Percentage of elements in the sample of sludge from vertical settling tanks with volumetric coagulation (sample no. 1).

and TOC in the water determine the coagulation process at the WTP. The remaining elements are of no technological importance and do not take part in the process. They are removed with amorphous $Al(OH)_3$ and their content depends on the raw water quality. The aluminum content in a coagulation sludge from the vertical settling tank, lamella separator and the sludge tank are shown in Fig. 3.

3.2. XRD results

To accurately determine the chemical structure of the compounds present in a coagulation sludge (mainly aluminum forms) and to verify whether the coagulation processes operate well, XRD analysis was performed.

The results of the XRD analysis for the sludge samples (1–5) collected: after vertical settling tanks with volumetric coagulation – O, after lamellas separators – L and from the sludge tank – K are presented in Tables 3 and 4 as well as in a diagram (printout) from the instrument (Fig. 4). The content of compounds of a crystalline nature is shown as a percentage (%) or as – "presence", when only trace amounts of the compound were detected.

XRD analysis showed that the sludge was mainly composed of silica that precipitated during the process of coagulation. In all analyzed samples, silicon oxide (IV) was found. Another compound that was found in almost all samples is calcium carbonate (up to 22.1%), which is the key component of many minerals. In sample no. 5, the trace amounts of potassium aluminum silicate of the formula $KAl_3Si_3O_{11}$ were identified. The sludge samples no. 1 had also kaolin ($Al_2Si_2O_5(OH)_4$), which is the main component of clays and loams. In six samples collected in May, traces of titanium oxide (TiO_2) were detected; its presence in the sludge may suggest that raw water was contaminated

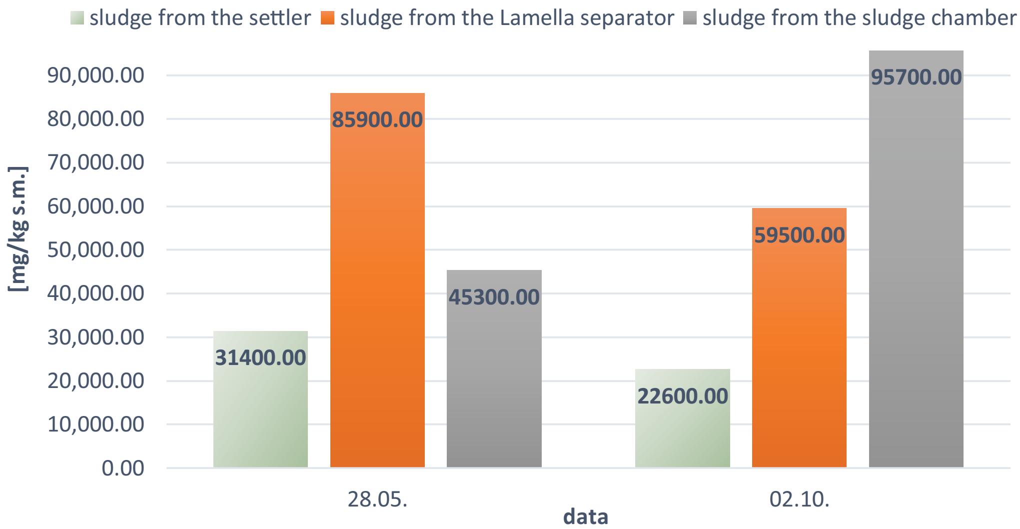

Fig. 3. Aluminum (Al) content in the samples nos. 3 and 4.

Table 3
XRD results for a coagulation sludge

Day	Sampling point	$CaCO_3$	SiO_2	Fe_3O_4	$KAl_3Si_3O_{11}$	$Al_2Si_2O_5(OH)_4$	NH_4NO_3
	O	present	present			present	
15.03	L		74.1			25.9	
	K*	17.4*	41.5*			17.0*	24.1*
	O	present	present				
22.05	L	present	present				
	K	present	present				
	O		present				
28.05	L	present	present				
	K	present	present				
	O	18.3	81.7				
02.10	L	23.6	75.1				
	K	12.2	86.5				
	O	15.4	86.8				
15.12	L	present	present	present	present		
	K	22.1	70.9	7.1			

Table 4
XRD results for a coagulation sludge

Data poboru	Punkt poboru	TiO_2	KCl	$AlSi_2P_3O_{12}$	$Al_2(SO_4)_3{\cdot}16H_2O$	Al_2O_3	Fe_2TiO_4	$Fe_5Al_4Si_6O_{22}(OH)_2$
	O							
15.03	L							
	K*							
	O	present	present	present	present			
22.05	L	present	present	present		present		
	K	present		present			present	
	O	present		present			present	present
28.05	L	present	present	present				
	K	present	present	present				
	O							
02.10	L							
	K							
	O							
15.12	L							
	K							

with industrial sewage. Also iron orthotitanate – a ferromagnetic mineral, the so-called ulvospinel (Fe_2TiO_4), was found in the sludge samples no. 2 and no. 3. Fe_2TiO_4 is formed in igneous rocks (deep and effusive) as well as in transformed formations and crumb rocks. Another mineral, ferrogedrite ($Fe_5Al_4Si_6O_{22}(OH)_2$) was detected in trace amounts in the sludge from the vertical settling tank, only.

In the samples collected in May, trace amounts of potassium chloride (KCl), occurring naturally in the form of the mineral – sylvin, were found. In some single samples, traces of Al_2O_3 and $Al_2(SO_4)_3{\cdot}16H_2O$ were detected. The sample no. 1 (sludge from the sludge tank marked with *) was found unreliable in terms of a NH_4NO_3 content. This sample was taken in severe weather conditions, that is, a sudden change of weather from a stable winter (cold and snowy) to spring thaw. Soon there was a sudden rise of flow in the Dunajec River followed by deterioration of physic-chemical parameters of raw water. Also several other technological factors that had happened simultaneously could have influenced the results, that is, temporary turn off of coagulant dosing to vertical settling tanks with volumetric coagulation. It was done to verify the assumption that coagulation of water with a very low turbidity is not only ineffective, but also increases the residual aluminum concentration in the water after the settling tanks.

The lack of a stable, crystalline form of aluminum hydroxide, that is, gibbsite (hydrargilite) in the samples proves an amorphous nature of coagulation sludge

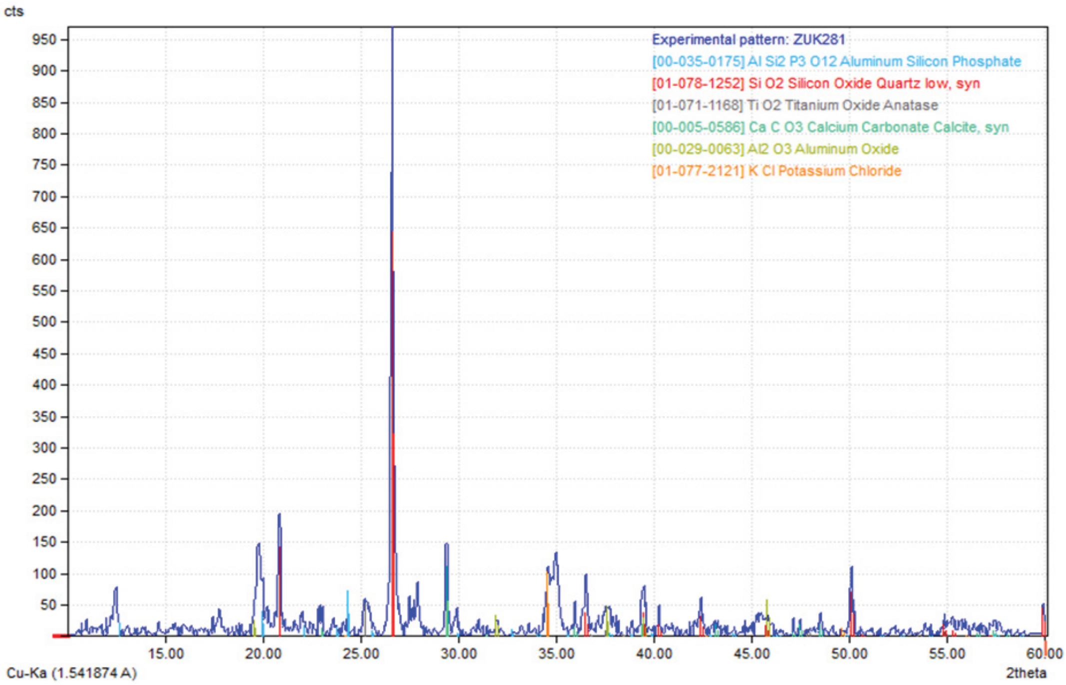

Fig. 4. Content (%) of compounds of a crystalline nature in the sludge from the lamella separator (L) – sample no. 2.

($Al(OH)_3$). The content of other remaining aluminum compounds present in the form of, for example, aluminosilicates is small, therefore it can be concluded that most aluminum compounds have the amorphous form, as precipitated $Al(OH)_3$. Coagulation carried out with the use of aluminum polychlorides $Al_x(OH)_yCl_{(3x-y)}$ and polymerized aluminum salts – aluminum polyhydroxycholor sulfates (VI) ($_xAl(OH)_{1.5}Cl_{1.5} \times {}_x(SO_4)_{0.2}$ does not result in formation of crystalline forms of $Al(OH)_3$ (no excessive dosing of the coagulant was observed). Aluminum hydroxide was entirely precipitated in the amorphous form with other contaminants. XRD analysis showed a negligible (trace) presence of iron in the sludge. It was present in as crystalline compounds, which proves that iron compounds precipitated with aluminum hydroxide mainly in the amorphous form are present in sludge samples. The XRD analysis correlates here with the XRF analysis of these elements. The pre-set doses of coagulants are the minimum doses required to achieve the expected technological effect, while maintaining a low level of total aluminum in the treated water. Multiple XRD analyzes proved that silica is the major crystalline phase present in the sludge. Also, the analysis of coagulation sludge with a scanning electron microscope (SEM) did not reveal the crystalline structure of $Al(OH)_3$, so it may be concluded that the sludge had a highly porous amorphous structure; this observation is consent with results reported by the study of Chiang et al. [12]. In the experiments, a relatively high content of aluminosilicate (a crystalline solid) in the aluminum sludge was found, confirming the results reported in the literature (e.g., [12–14]). Such unique structure makes this sludge a feasible component for ceramics, following the circular economy requirements. The tests revealed also that due to the amorphous and porous nature of Al and Fe ions, the sludge provides adsorption sites for most anions, which makes it an effective tool for other pro-ecological applications like: landfill recovery [15], groundwater resources' protection [16] or improvement of overall efficiency of municipal wastewater treatment plants [17].

4. Conclusions

The modern practice of water coagulation, to meet the ever-increasing demand concerning a drinking water quality, requires employing more advanced methods to determine the true nature of the aluminum compounds in the water at different stages of its treatment, including a sludge disposal process. It is particularly important to focus on sludge characteristic so as this by-product could be used in the most effective way. The methodology described in the article has been verified in real operational conditions as an effective tool for both research and a practical use of a coagulation sludge. The XRD analysis of fresh and mature sludge (after coagulation with aluminum coagulants) combined with a scanning electron microscope (SEM) provided accurate data concerning sludge characteristics, that is, porosity and its nature (amorphous and porous character). Such results provide a more detailed description of the coagulation results than the conventional methods used so far.

In the future, research is planned on the seasonal variability of the metal content in the sludge from water treatment.

Summary

In the nearest future, water utilities will be forced to remove pollutants (especially metals) from water more precisely and efficiently than they are doing it today to meet the formal requirements. Among the metals that can be a potential source of risk to consumers, aluminum deserves a special attention. On one hand, it can be present in the raw water while on the other hand, it must be in a controlled manner added to the water as a coagulant during the treatment process (e.g., aluminum sulphate or polyaluminium chloride).

Taking into account the above formal requirements, it is important that most of the aluminum is removed from water as bound in the coagulation sludge during the water treatment process. To better control this process more accurate measurement procedures are needed to monitor the content of aluminum.

The method of the X-ray diffraction analysis, presented and applied by the authors, allows to determine the structure and type of aluminum compounds in the sludge and provides the operator of a water treatment plant with a broader spectrum of knowledge about this specific pollutant.

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