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Analysis of the Physical and Chemical Composition of Sludge from the Water Treatment Plant

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Abstract: The properties and composition of the sludge generated in water treatment systems depend primarily on the type and composition of the water to be treated, the treatment methods, and the type and doses of chemical reactants. The sludge produced in the water treatment plant (WTP) under study follows the technological processes of coagulation, flocculation, sedimentation, and filtration. The analyses aimed to characterize the sludge in terms of its physico-chemical properties and classify it in terms of its potential discharge into the river and management. Four series of sediment tests were conducted over a calendar year (March, June, September and December), analysing selected parameters using various test methods, including the X-ray crystallography (XRF) method. The publication's authors showed that the sediment consists mainly of sand, clay, and silt particles with grain sizes ranging from 0.001 mm to 1 mm. Silica (53.78%), alumina (23.58%), calcium oxide (8.28%), iron (III) oxide (5.61%), and potassium oxide (2.36%) represent the main chemical constituents present in the sediment. The authors characterized the sediment in terms of the content of biogenic compounds: various forms of phosphorus and nitrogen, organic compounds – determined as total organic carbon (TOC), selected metals, and the content of individual elements (carbon, hydrogen, oxygen, nitrogen, sulfur). In addition, the sludge samples were also characterized in terms of calorific value, ash content, water content, and heat of combustion. Discharging WTP sludge into rivers, ponds, and lakes or storing dewatered sludge is an environmentally unfriendly form of disposal for this type of waste. The authors see the possibility of conducting further research on using WTP sludge in wastewater treatment, removing heavy metals from aqueous solutions, producing cement and construction materials, and recovering or recycling.

Keywords: coagulation sludge, water treatment sludge, metals in sludge, X-ray crystallography

1. Introduction

A factor affecting the suitability of water for human consumption is its quality, i.e., its physico-chemical and bacteriological composition. The primary task of water treatment plants (WTP) is to apply appropriate technological processes to provide consumers with water of suitable composition in line with the requirements for drinking water (Regulation 2017). The tightening of EU and national legislation on the quality of treated water, including the new Directive of the European Parliament and the Council of Europe on the quality of water intended for human consumption (2020/2184) (Directive EU 2020, Wysowska et al. 2024), determines water supply companies to implement new, reliable water treatment methods.

The treatment of water, especially surface water, produces a large amount of sludge (WTS) (between 2% - 5% of the treated water), which is difficult to manage. The location, type, and composition of the water to be treated – surface or groundwater, the method of treatment, and the type and doses of chemical reactants are used to determine the composition and properties of the sludge generated after the treatment process (Ahmad et al. 2017, Crittenden et al. 2012).

Discharging WTS into rivers, streams, ponds, lakes or storing dewatered WTS is currently the most common method of disposal, which is environmentally unfriendly due to the possible presence of toxic chemicals (Ippolito et al. 2011, Przydatek et al. 2017, Zwolińska & Basta 2024). The authors, seeing a gap in research on the subject, attempted to identify physical and chemical properties characterizing the sludge generated at the WTP over a calendar year. This research will form the basis for developing a sustainable strategy for managing this type of sludge in other industries and economies (Generowicz et al. 2011, Kowalski et al. 2012).

Surface waters are characterized by high variability in composition, resulting in sediments with large fluctuations and differences in quantity and quality. Pollutants removed from surface waters are mainly:

- clay minerals,
- clay and sand particles,
- dissolved and colloidal organic substances,
- plant and animal residues (Anjithan 2016, Crittenden et al. 2012, US EPA 2011).

Groundwater can additionally produce sediments containing precipitated iron and manganese compounds. Conversely, softening produces sediments rich in calcium and magnesium compounds through sedimentation and filtration (Ahmad et al. 2016a, Sales et al. 2011). Post-agglomeration sediments are also formed from the removal of solid, liquid, and gaseous organic and inorganic colloidal substances from water. The amount of post-aggregation sludge generated ranges from 0.1% to 5.0% of the daily capacity of a water treatment plant and varies throughout the year (Ahmad et al. 2016b). The amount of sludge generated is influenced by:

- quantity of water treated,
- quality of treated water, mainly: suspended solids, turbidity, color, organic compounds,
- dose and type of coagulants,
- water treatment technology and water quality after treatment (Babatunde & Zhao 2007, Bahadori et al. 2013).

A typical water treatment plant is estimated to produce about 100,000 tonnes of hydrated sludge per year, while globally, the literature reports an estimated daily sludge production of about 10,000 tonnes (Babatunde & Zhao 2007). The scale of the problem appears to be enormous; thus, the management of sludge from water treatment plants requires the introduction of carefully selected and sustainable solutions (Delpla et al. 1999, Molinos-Senante & Guzman 2018, Ciuła et al. 2019). The hydration of such sludge varies from 98.5% to 99.9% and depends on the method and frequency of discharge from the settling tanks and also on the seasonal variability of its physico-chemical composition (Tantawy 2015, Gronba-Chyła et al. 2024). Significant amounts of sludge are produced when sludge is continuously discharged from the settling tanks, whereas periodic discharges produce much less sludge due to compaction (Sozanski 1999, Ciuła 2022). Sediments that result from coagulation of water pollutants with pre-hydrolyzed aluminum polychloride and iron (III) sulfate are more susceptible to dewatering than sediments that result from coagulation with non-hydrolyzed aluminum sulfate (Qrenawi & Rabah 2021, Tomtas et al. 2021). Colloidal and suspended contaminants such as sand, silt, clay, and humic particles present in the raw water are removed during the coagulation process by charge neutralization, sweep coagulation mechanism, and adsorption (Trinh & Kanga 2011, Rybicki & Wiewiórska 2017). During the treatment of groundwater rich in iron salts, they are hydrolyzed to $Fe(OH)_2$ and then oxidized by oxygen present in the water to form iron (III) hydroxide, which is hardly soluble in water. Iron in water can also occur in combination with organic compounds, removed by coagulation. The precipitates produced in this process are a mixture of post-coagulation and ferric precipitates (Verlicchi et al. 2001). Manganese compounds in the form of MnO_2 are also a component of sediments following the treatment of manganese-rich groundwater (Bal Krishna et al. 2016, Banaś & Hilger 2024). Lime decarbonation results in sludge containing mainly $CaCO_3$ as well as, to a lesser extent: colloids and hardly soluble suspended solids, phosphates, organic compounds, heavy metals, deactivated viruses and bacteria, and iron (III) hydroxides when an iron coagulant is used (Dziubek & Maćkiewicz 1996). When water is softened by lime-soda, lye-soda, and sodium hydroxide methods, a mixture of $CaCO_3$ and $Mg(OH)_2$ is formed in the sediment. In the case of softening with phosphates, hardly soluble calcium and magnesium phosphates are found in the sediments (Szerzyna 2013).

Management of sludge generated during water treatment is understood as:

- discharging sludge safely into the environment,
- landfill,
- use in industry or the economy,
- use in wastewater treatment processes by recovering reactants or recirculating the recovered filtrate water,
- use for H_2S binding,
- disposal with sewage sludge or waste (joint composting) (Ahmad 2016b, Basta & Szewczyk 2024, Gaska et al. 2019, Ciuła et al. 2023a).

The most important waste treatment and disposal process in water treatment plants is the thickening of washings and sludge. In Poland, gravity thickening is the most commonly used method (Jaroszyński et al. 2011). Still, others are also emerging, e.g., mechanical sludge dewatering on a filter press or using mobile sludge dewatering stations (Wei et al. 2018). To dewater sludge even more efficiently, new coagulants and flocculants are being developed, which should be characterized by: a high dewatering rate, efficiency, as well as being environmentally friendly and cost-effective (Gronba-Chyła et al. 2021). To develop a new, effective coagulant/flocculant, knowledge of the structure and activity of the sludge and coagulant is essential. The knowledge of different types of inorganic metals, ionic and non-ionic functional groups, with different molecular weights and chain conformations on different types of sludge, is still unsystematic and limited (Ciuła et al. 2023b, Wei et al. 2018).

According to the Regulation of the Minister of the Environment of 11 May 2015 on the recovery of waste outside installations and facilities (Regulation 2015a), the use of clarification sludge for tidying up and protecting from water and wind erosion the slope and crown surface of a closed landfill or part of it is allowed (Verrelli et al. 2010). Water clarification and decarbonation sludge can be stored non-selectively (Regulation 2015b, Gronba-Chyła et al. 2022). The Regulation of the Minister of the Environment of 10 November 2015 on the list of types of waste that the holder of waste may transfer to individuals or organizational units that are not entrepreneurs and the permitted methods of their recovery (Regulation 2015c) allows the use of waste from water clarification (decarbonation for the liming of acidic soils (Ahmad et al 2016, Sales et al. 2011) or for the production of building materials (cement, bricks, ceramic tiles, roof tiles, ceramic pipes, concrete, lightweight aggregates, mortar) as also mentioned in their work: American Water Works (Association & Edzwald 2011, Cremades et al. 2011) and (Luo et al. 2008). Aluminum sludge can also be used efficiently to remove nitrogen, phosphorus, and organic matter (Babatunde et al. 2010). Recently, there have also been literature reports on using sludge from water treatment as adsorbents for heavy metals from wastewater (Abo-El-Enein 2017). Another way to manage sludge is by recovering coagulants from post-coagulation sludge and CaO and CO_2 from calcareous sludge containing high amounts of $CaCO_3$ (Bishop 1987). Hydrated iron sludges are used to bind hydrogen sulfide generated in sewage networks, and scrubbers to remove hydrogen sulfide generated during anaerobic digestion of sewage sludge. (Szerzyna 2013, Jaroszyński et al. 2011, Kowalski et al. 2020). Sludge from water treatment can also be directly discharged in liquid form to a wastewater treatment plant without reducing the efficiency of wastewater treatment (Kowal & Świderska-Bróż 2009, Kowalski et al. 2022).

The results of a pilot study on the modification of a classic sweep coagulation system by introducing recirculation of post-coagulation sludge (Wang et al. 2004, Xu et al. 2011) also showed that treating water in a coagulation system with recirculation of post-coagulation sludge to the flocculation chamber as well as to the static mixer reduces the negative effects of sweep coagulation, i.e., causes a reduction in the aluminum remaining in the water after the coagulation process (Yang et al. 2011, Yan M. et al. 2007).

In the available literature, there is a lack of comprehensive studies on the physico-chemical composition of sludge from full-scale WTPs. This study aimed to determine in detail the physico-chemical composition of the analyzed samples of post-agglomeration sludge generated at the WTP during a calendar year, including physical parameters such as particle size analysis, calorific value, and a detailed analysis of the chemical and elemental composition, using, among others, an innovative test method- X-ray fluorescence (XRF) (Wiewiórska & Rybicki 2023). Comprehensive studies of post-coagulation sludge fill an important research gap and should be used to develop strategies for WTP users to deal with this type of sludge, which to date has been predominantly discharged to receiving bodies, i.e., rivers, lakes and streams, sewers (Ahmad et al. 2016a, Ahmad et al. 2016b).

2. Methodology

The study was conducted under real-world conditions in a dynamically operating WTP, respecting the requirements for water quality for human consumption (Regulation 2017) and continuity of supply. The water production of the selected WTP, resulting from the water demand of the residents, is, on average, 8,000 m^3/d. The plant treats surface water from the river and water from infiltration wells. The technological processes of water treatment that have a direct impact on the quantity and quality of the sludge generated at the WTP include: volumetric coagulation of river water in vertical settling tanks, filtration in dynamic sand bed filters enriched with contact coagulation and a compact Lamella separator with a flocculation chamber for the pretreatment of washwater generated continuously by the filter battery (Wiewiórska 2023a, Wiewiórska 2023b). During the study period, a pre-hydrolyzed coagulant, i.e., a mixture of aluminum sulfate hydroxychloride and aluminum chloride complex solution with the trade name Flokor 1,2A, was continuously dosed into the volumetric coagulation system, the contact coagulation system in the sand filter bed and the flocculation tank of the Lamella separator.

During operation, qualitative determinations were carried out for the main contaminants in the intake water, including suspended solids, turbidity, color, absorption of light in the UV part of the light spectrum at 254 nm (UVA_{254nm}), permanganate index and aluminum, iron, zinc chromium, cadmium, manganese, copper, nickel, lead, potassium, sodium, calcium by inductively excited plasma atomic spectrometry.

Sludge for physico-chemical testing was collected from the sludge chamber, the collection point for all sludge generated at the WTP. The sludge chamber collects sludge from the volumetric coagulation settling tanks and from the lamella separator that purifies the rinsing water generated after rinsing the sand bed in the filters. The sludge is collected in a chamber for 2 to 5 weeks, after which it is dewatered on a belt press. Approximately 100 tonnes of dewatered post-coagulation sludge are generated annually at the WTP.

The physico-chemical properties of the dried and crushed sediments of approximately 1 kg, taken in 2023 in March – sample 1, June – sample 2, October – sample 3, December – sample 4, were tested in the accredited laboratories SGS Polska Sp. z o.o. and ALS Polska Sp. z o.o.

To accurately identify the composition of the sludge in terms of physico-chemical parameters, analyses were carried out on the following: particle size distribution, dry mass content, water content, calorific value and ash content, pH reaction, elemental content (selected metals and non-metals), nitrogen and phosphorus compounds, total organic carbon (TOC) and organic acids.

The authors also presented a novel and non-routine approach to the topic of assessing the composition of post-coagulation sediments using a qualitative method from the field of X-ray crystallography, i.e., X-ray fluorescence (XRF), to determine the percentage of chemical compounds and elements in sediment samples.

Analyses were performed according to the procedures and methods shown in Table 1.

Table 1. Test parameters and test methodology

Test parameters	Research methods	Standards
determination of the percentage composition of chemical compounds and elements	X-ray fluorescence (XRF) using a Bruker S1 Titan LE spectrophotometer. Each analysis was repeated 3 times and averaged.	non-routine testing
preparation of solid samples for analysis	crushing, grinding, and pulverizing drying and sieving a sample with grain size < 2 mm	-
metals – sample preparation	acid mineralization in the heating block	-
grain size distribution	sieve analysis, sieving, and measurement with a laser analyzer in the range of > 0.0001 mm to < 1 mm	-
gross calorific value net calorific value emission factor	colorimetric, calculation	CSN ISO 1928, CSN EN ISO 18125, CSN EN 21654, CSN EN 15170, CSN DIN 51900-1, CSN DIN 51900-2, CSN DIN 51900-3, CSN P CEN/TS 16023
dry matter (d.m.) at 105°C, water content	calculation	CSN ISO 11465
ash, roasting losses at 550°C	gravimetric, computational	CSN EN 15169, EN 15935, EN 13039
analytical water, gross water, total moisture	by weight, calculation	CSN 44 1377, CSN EN ISO 18134-1, CSN EN ISO 18134-2, CSN EN ISO 18134-3, CSN P CEN/TS 15414-1, CSN P CEN/TS 15414-2, CSN EN ISO 21660-3, CSN EN12880, CSN EN14346, CSN EN 15002
pH	Electrochemical determination of pH in a suspension of a soil/sediment sample in water (pH-H_2O) or KCl, $CaCl_2$, $BaCl_2$. The resulting pH value with reference to 25°C	CSN ISO 10390, CSN EN 12176:1999, CSN EN 13037, CSN EN 15933, CSN 46 5735, ÖNORM L 1086-1, US EPA 9045D, US EPA 9040C

Table 1. cont.

Test parameters	Research methods	Standards
elements	Atomic emission spectrometry with inductively excited plasma. Samples were homogenized and mineralized in royal water before analysis.	US EPA 200.7, CSN EN ISO 11885, US EPA 6010, SM 3120, US EPA 3050, CSN EN 13657, ISO 11466 chapters 10.3 to 10.16, 10.17.5, 10.17.6, 10.17.9 to 10.17.14.
ammonium nitrogen and ammonium ions, nitrites, and fully oxidized nitrogen ions	discrete spectrophotometry	ISO 11732, CSN ISO 13395
Kjeldahl nitrogen	spectrophotometry	CSN EN 25663, EN 13342 CSN, CSN ISO 7150-1
P_2O_5 in silicates	spectrophotometry	CSN 72 0116-1
total carbon (TC), total organic carbon (TOC), total sulfur, and hydrogen	combustion with IR detection, determination of total nitrogen by TCD, determination of oxygen by calculation method	CSN ISO 29541, CSN EN ISO 16994, CSN EN ISO 16948, CSN ISO 19579, CSN EN 15408, CSN ISO 10694
total organic carbon (TOC)	combustion method with IR detection	CSN ISO 10694, CSN EN 13137:2002, CSN EN 15936
organic acids	capillary electrophoresis with UV detection	ISSN 1406-0124

Source: own compilation based on Certificates of Analysis issued by ALS Polska Sp. z o.o. and SGS Polska Sp. z o.o.

3. Results

3.1. Physical characteristics

The characteristics of the sediments in terms of physical parameters are shown in Table 2. The pH value of the sediments ranged from 7.7 (in October) to 7.9 (in June), due to seasonal variations in the quality of water abstracted from the river. (Dassanayake et al. 2015) Obtained pH values of 5.12-8.0 in the post-coagulation sediments studied.

Table 2. Physicochemical parameters of sediments

Parameter	Unit	Sediment samples 1-4								
		1	2	3	4	min.	av.	max.	stand. dev.	median
		March	June	September	December					
residue after incineration at 550°C	% d.m.	73.0	68.4	81.9	77.0	68.4	75.1	81.9	4.98	75.00
loss on roasting at 550°C	% d.m.	27.0	31.6	18.1	23.0	18.1	24.9	31.6	4.98	25.00
dry mass at 105°C	%	31.0	26.3	41.0	27.4	26.3	31.4	41.0	5.79	29.20
reaction	pH	7.8	7.9	7.7	7.8	7.7	7.8	7.9	0.07	7.80
calorific value (calorific value) in dry matter	$MJ \cdot kg$ $d.m.^{-1}$	3.12	3.44	1.87	3.07	1.87	2.87	3.44	0.60	3.10
water content	%	72.3	72.2	58.2	72.4	58.2	68.8	72.4	6.11	72.25
ash content in dry matter at 550°C	% d.m.	71.0	69.4	81.7	72.0	69.4	73.5	81.7	4.81	71.50
analytical ash at 550°C	%	19.7	19.3	0.91	19.9	0.91	14.9	19.9	8.11	19.50
heat of combustion on dry matter	$MJ \cdot kg$ $d.m.^{-1}$	3.42	4.02	2.59	3.53	2.59	3.39	4.02	0.51	3.48

The dry matter in the four sediment samples ranged from 23.3%, with an average of 31.4% and a max. 41.0%, while the moisture content ranged from 58.2% to 72.4%, with an average value of 68.8%. The roasting losses averaged 24.9%, and the residue after combustion averaged 75.1%. The ash content of the dry matter of the tested sludges was found to be high, with an average of 73.5%, a minimum of 69.4% for the sample taken in June and a maximum of 81.7% for the sample taken in October, while the roasting losses at 550°C ranged from 18.1% to 31.6%. Similar results for ash content (89.78%) in sludge samples from the WTP in India were obtained by (Ahmad et al. 2016a). The heat of combustion on a dry weight basis averaged 3.39 $MJ \cdot kg$ $d.m.^{-1}$. The analytical results indicate that the sludge is mainly inorganic (Table 2). The average calorific value of the tested sludge was

2.87 MJ·kg d.m.$^{-1}$, and the maximum was 3.44 MJ·kg d.m.$^{-1}$. These values are low compared to sewage sludge, which, according to (Cao & Pawlowski 2012), can range from 8.9 MJ·kg d.m.$^{-1}$ to 23.0 MJ·kg d.m.$^{-1}$, depending on the type and site of formation.

Figure 1 shows the particle size distribution of the sediments. Approximately 63.5% of the sediments, on average, consisted of solid particles with a grain size between 0.01 mm and 0.1 mm. 18.4% of the sediments consisted of particles between 0.1 mm and 1 mm, and 17.7% of the sediments consisted of particles between 0.001 mm and 0.01 mm. No particles smaller than 0.001 mm and larger than 1 mm were observed in the sediment samples tested. All samples tested (1-4) contained a comparable percentage particle size distribution. The morphology and particle size distribution indicate the presence of mainly sand and clay grains in the sediments (Ahmad T. et al. 2016a).

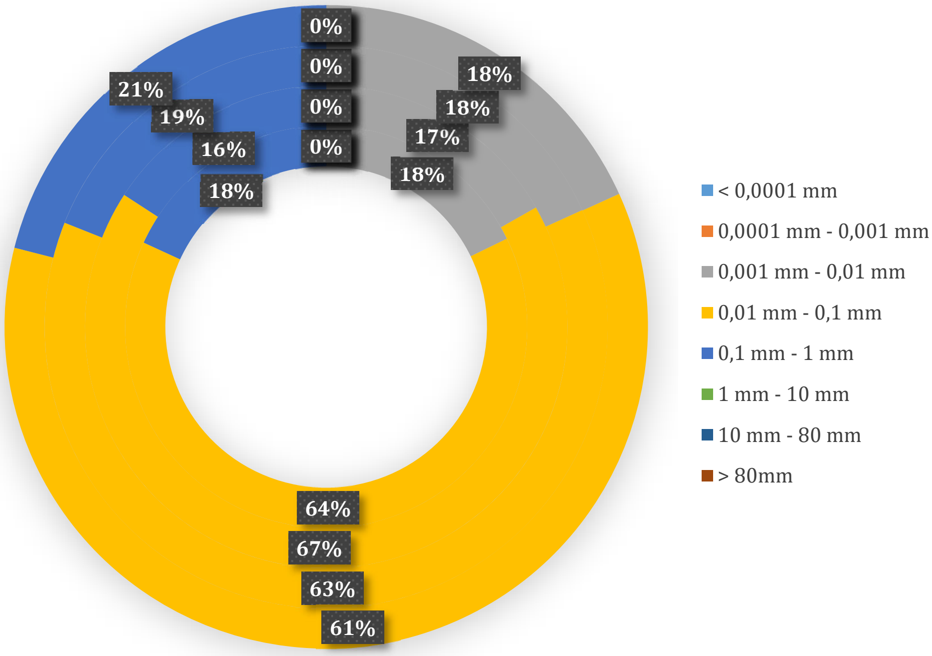

Fig. 1. Particle size distribution [%] in WTS samples (samples 1-4)

3.2. Chemical characteristics

An important step in the study was identifying the chemical composition using the XRF method (Table 3). The XRF analysis showed that the main component of the post-agglomeration sediment is silicon (IV) oxide from river water, with an average content of 53.781% from four measurements over the year. Aluminum oxide (mean – 23.582%) was further identified in the samples, the content of which is directly influenced by the Al-based coagulant dosed into the water. Calcium carbonate was further identified, with an average content of 23.582% – the main component of many minerals – and iron (III) oxide with 5.610%. Potassium oxide was present in the sediments at an average of 2.358%, phosphorus (V) oxide, manganese (II) oxide, and titanium oxide in smaller amounts. Sulfur, chlorine, cobalt, zinc, and arsenic were found in less than 1% of the sediments studied. Similar results on the percentage of chemical components in sediments were obtained in the work of Breesem et al. (2016). The authors examined sediments from the WTP using the XRF method. Huang et al. (2005) used an energy dispersive spectrometer (EDS), an inductively coupled plasma atomic emission spectrometer (ICP AES), and an atomic adsorption spectrophotometer (AAS) to study sediments. Chiang et al. (2009) used the X-ray diffraction (XRD). All researchers noted that the main components of post-coagulation sludge from surface water treatment at WTP are: SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, Na_2O, K_2O, and P_2O_5.

Table 3. Results of XRF analyses of post-coagulation sediments

Parameter	Unit	Sediment samples 1-4								
		1	2	3	4	min.	average	max.	stand. dev.	median
		March	June	September	December					
Al_2O_3	%	25.949	25.387	20.959	22.034	20.959	23.582	25.949	2.130	23.710
SiO_2	%	53.797	53.566	57.799	49.962	49.962	53.781	57.799	2.774	53.681
P_2O_5	%	0.254	0.234	<LOD	0.293	0.234	0.260	0.293	0.025	0.254
S	%	0.354	0.904	0.305	0.635	0.305	0.550	0.904	0.241	0.495
Cl	%	1.074	1.108	1.069	1.234	1.069	1.121	1.234	0.067	1.091
K_2O	%	2.354	2.032	2.774	2.273	2.032	2.358	2.774	0.268	2.314
CaO	%	9.155	9.233	5.744	9.007	5.744	8.285	9.233	1.469	9.081
TiO_2	%	0.070	0.703	0.929	0.761	0.070	0.616	0.929	0.326	0.732
MnO	%	0.202	0.264	0.193	0.370	0.193	0.257	0.370	0.071	0.233
Fe_2O_3	%	5.377	5.274	6.107	5.684	5274	5.610	6.107	0.324	5.530
Co	%			0.760	0.685	0.685	0.722	0.760	0.038	0.722
Zn	%			0.012	0.012	0.012	0.012	0.012	0.000	0.012
As	%		0.260	0.065	0.282	0.065	0.202	0.282	0.097	0.260

Ahmad et al. (2016a) also studied the chemical properties of post-coagulation sediments by Energy dispersive X-ray fluorescence (ED-XRF) and Wavelength Dispersive X-ray Fluorescence (WD-XRF) (from a water treatment plant in Ghaziabad, India. The researchers determined in the sediment sample: SiO_2 (52.78%), Al_2O_3 (14.38%) Fe_2O_3 (5.20%), CaO (4.9%), K_2O (3.62%), MgO (3.08%) and small amounts of Na_2O (0.97%), TiO_2 (0.61%), P_2O_5 (0.17%), MnO (0.08%) and ZnO (0.01%). The sludge was aluminous in nature because the Ghaziabad station, like the one analyzed by the authors of the publication, uses an Al-based coagulant (PACl) for water treatment.

The presence in the sediment samples of biogens, i.e., nitrogen (N_{Kj} , N-NH_4, $NH_4^{+)}$ and phosphorus (P_{og}, P_2O_5, PO_4), the content of elements forming chemical compounds (C, H, O, S), total organic carbon (TOC) was shown. No significant amounts of organic acids (acetic acid, lactic acid, propionic acid, isobutyl acid, isovalerian acid, valerian acid, caproic acid, formic acid) were found. The results of the analyses are shown in Table 4.

Table 4. Results of analysis of different forms of nitrogen, phosphorus, and analysis of C, H, O, S, TOC and organic acids

Parameter	Unit	Sediment samples 1-4								
		1	2	3	4	min.	av.	max.	stand. dev.	median
		March	June	September	December					
Kjeldahl nitrogen (N_{Kj})	mg·kg d.m.$^{-1}$	6780	9720	5500	7900	5500	7475	9720	1550	7340
total phosphorus (P)	% d.m.	0.115	0.133	0.095	0.148	0.095	0.123	0.148	0.020	0.124
phosphorus (P_2O_5)	% d.m.	0.2	0.3	0.22	0.34	0.20	0.27	0.34	0.06	0.26
ammonium ion (NH_4^+)	mg·kg d.m.$^{-1}$	0.93	2.24	0.72	0.66	0.66	1.14	2.24	0.64	0.825
total organic carbon (TOC)	% d.m.	6.96	10.1	6.00	7.01	6.00	7.52	10.10	1.54	6.985
ammonium nitrogen (N-NH_4)	mg·kg d.m.$^{-1}$	0.80	1.74	0.56	0.52	0.52	0.91	1.74	0.49	0.68
phosphorus (PO_4)	% d.m.	0.121	0.133	0.126	0.148	0.121	0.132	0.148	0.010	0.130
combustible sulphur in the dry matter	% d.m.	0.26	0.41	0.26	0.28	0.26	0.30	0.41	0.06	0.27
oxygen in the dry matter	% d.m.		15.2	9.2	13.9	9.2	12.8	15.2	2.6	13.9
nitrogen in the dry matter	% d.m.		1.13	0.51	0.86	0.51	0.83	1.13	0.25	0.86
hydrogen in the dry matter	% d.m.		2.81	1.48	2.24	1.48	2.18	2.81	0.54	2.24
carbon in the dry matter	% d.m.		11.1	6.86	10.7	6.86	9.55	11.1	1.91	10.7
lactic acid	mg·kg^{-1}	<7.5	<7.5	<7.5	<7.5					

Table 4. cont.

Parameter	Unit	Sediment samples 1-4								
		1	2	3	4	min.	av.	max.	stand. dev.	median
		March	June	September	December					
acetic acid	mg·kg^{-1}	<7.6	<7.6	<7.6	<7.6					
propionic acid	mg·kg^{-1}	<7.7	<7.7	<7.7	<7.7					
isobutyl acid	mg·kg^{-1}	<7.8	<7.8	<7.8	<7.8					
isovalerian acid	mg·kg^{-1}	<7.9	<7.9	<7.9	<7.9					
valerian acid	mg·kg^{-1}	<7.10	<7.10	<7.10	<7.10					
caprylic acid	mg·kg^{-1}	<15	<15	<15	<15					
formic acid	mg·kg^{-1}	<7.5	<7.5	<7.5	<7.5					

The results for total nitrogen determined by the Kjeldahl method were as follows: nitrogen - from 6780 mg·kg^{-1} d.m. in March to 9720 mg·kg^{-1} d.m. in June, while TOC – from 6.0% d.m. in October, 6.96% d.m. in March to 10.1% d.m. in June. A significant relationship was observed between the amount of organic matter and nitrogen content in the water scaling sediments resulting from river water treatment, and to verify the possible existence of a regression-type relationship between the selected quality indicators, a simplified statistical analysis was carried out (Figure 2). The coefficient of determination (R2) value was determined for the regression type relationship, which was 1,0 – a very good fit. The amount of nitrogen and phosphorus in the post-accumulation sediments depends on their concentration in the water (Koc & Szyperek 1996). An important factor shaping the inflow of biogenic nitrogen and phosphorus compounds was the supply of these components together with anthropogenic pollutants, mainly sewage from areas without sewage systems above the intake and from sewage treatment plants and sewage from agricultural activities (arable fields). The presence of a dam reservoir located above the water intake is also important.

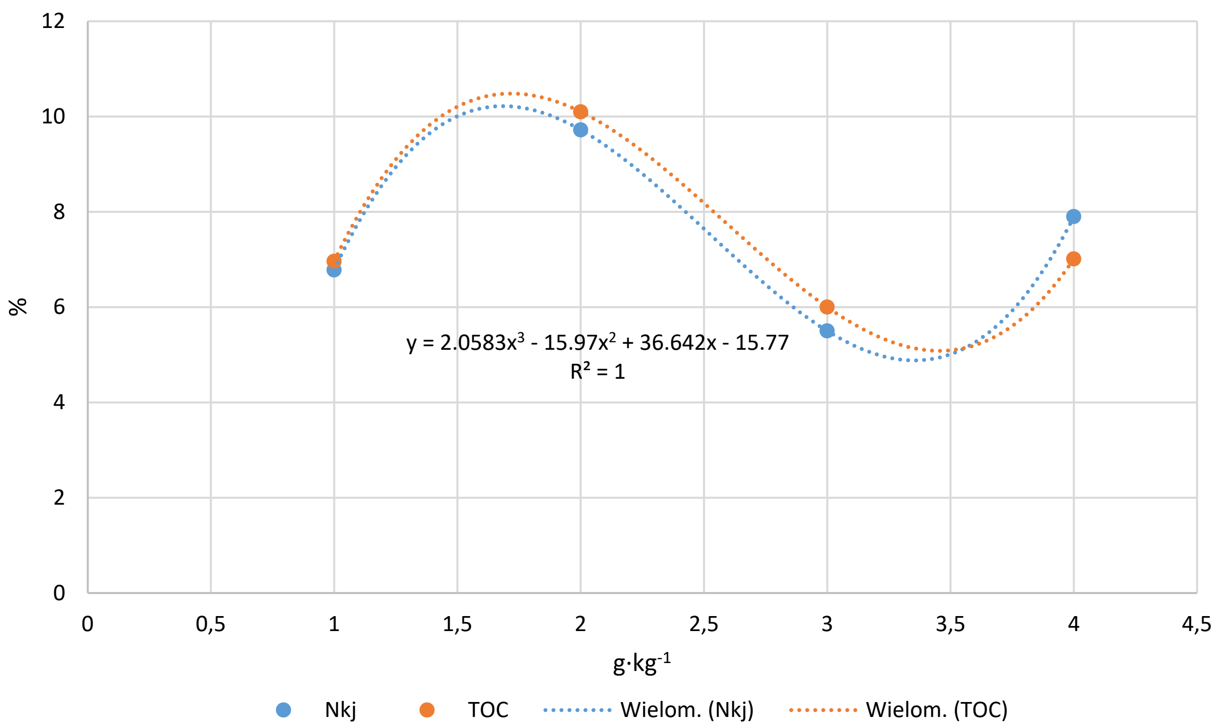

Fig. 2. Dependence of sediment nitrogen content on the amount of organic matter

This was followed by quantitative analysis (mg/kg d.m.) of the elements contained in the sediment samples by inductively excited plasma atomic emission spectrometry, and the results are shown in Table 5.

Table 5. Results of analyses of selected elements

Parameter	Unit	Sediment samples 1-4								
		1	2	3	4	min.	av.	max.	stand. dev.	median
		March	June	September	December					
antimony (Sb)	mg·kg^{-1} d.m.	<0.50	<0.50	<0.50	<0.50					
barium (Ba)	mg·kg^{-1} d.m.	167	134	165	205	134	167.8	205	25.17	166.00
beryllium (Be)	mg·kg^{-1} d.m.	0.960	0.569	0.908	0.707	0.569	0.786	0.960	0.157	0.808
bismuth (Bi)	mg·kg^{-1} d.m.	<1.0	<1.0	<1.0	<1.0					
boron (B)	mg·kg^{-1} d.m.	14.9	17.9	16.8	20.1	14.9	17.4	20.1	1.9	17.4
chromium (Cr)	mg·kg^{-1} d.m.	30.1	33.2	40.9	41.3	33.2	38.5	41.3	3.7	40.9
tin (Sn)	mg·kg^{-1} d.m.	<1.0	1	1.3	<1.0	1.0	1.2	1.3	0.2	1.2
zinc (Zn)	mg·kg^{-1} d.m.	99.5	81.1	99.7	98.1	81.1	94.6	99.7	7.8	98.8
zirconium (Zr)	mg·kg^{-1} d.m.	<5.0	<5.0	<0.5	<5.0					
total phosphorus (P)	mg·kg^{-1} d.m.	1200	1600	1210	1910	1200	1480	1910	296	1405
aluminium (Al)	mg·kg^{-1} d.m.	45300	67100	39200	72800	39200	56100	72800	14161	56200
cadmium (Cd)	mg·kg^{-1} d.m.	<0.40	0.42	<0.40	0.45	0.42	0.44	0.45	0.02	0.44
cobalt (Co)	mg·kg^{-1} d.m.	11.2	7.05	12.6	9.45	7.05	10.08	12.60	2.07	10.33
silicon (Si)	mg·kg^{-1} d.m.	204	332	348	277	204	290.3	348.0	56.3	304.5
lithium (Li)	mg·kg^{-1} d.m.	68.0	37.6	69.2	48.8	37.6	55.9	69.2	13.3	58.4
magnesium (Mg)	mg·kg^{-1} d.m.	8440	4470	8630	5610	4470	6788	8630	1795	7025
manganese (Mn)	mg·kg^{-1} d.m.	1160	1320	1360	2570	1160	1603	2570	564	1340
copper (Cu)	mg·kg^{-1} d.m.	50.0	36.6	44.8	44.4	36.6	44.1	50.4	4.9	44.6
molybdenum (Mo)	mg·kg^{-1} d.m.	0.42	0.73	0.85	1.06	0.42	0.77	1.06	0.23	0.79
nickel (Ni)	mg·kg^{-1} d.m.	44.0	34.0	51.9	44.4	34.0	43.58	51.90	6.36	44.20
lead (Pb)	mg·kg^{-1} d.m.	23.4	11.4	20.0	15.6	11.4	17.6	23.4	4.5	17.8
potassium (K)	mg·kg^{-1} d.m.	2510	2220	3270	2320	2220	2580.0	3270.0	411.8	2415.0
selenium (Se)	mg·kg^{-1} d.m.	<2.0	4.2	<2.0	3.7	3.7	3.95	4.2	0.25	3.95
sulphur (S)	mg·kg^{-1} d.m.	2700	3720	2010	2560	2010	2748	3720	618	2630
sodium (Na)	mg·kg^{-1} d.m.	277	157	134	158	134	182	277	56	158
silver (Ag)	mg·kg^{-1} d.m.	<0.50	<0.50	<0.5	<0.50					
strontium (Sr)	mg·kg^{-1} d.m.	67.7	60.1	68.4	82.6	60.1	69.7	82.6	8.1	68.1
thallium (Tl)	mg·kg^{-1} d.m.	<0.50	<0.50	<0.50	<0.50					
tellurium (Te)	mg·kg^{-1} d.m.	<1.0	<1.0	<1.0	<1.0					
titanium (Ti)	mg·kg^{-1} d.m.	41.5	61.2	75	55.6	41.5	58.3	75.0	12.0	58.4
vanadium (V)	mg·kg^{-1} d.m..	33.5	28.3	39.5	32.2	28.3	33.4	39.5	4.0	32.9
calcium (Ca)	mg·kg^{-1} d.m.	33300	34000	26700	34800	26700	32200	34800	3219	33650
iron (Fe)	mg·kg^{-1} d.m.	29500	17800	30200	25300	17800	25700	30200	4931	27400

Analysis of the content of individual elements in the sediments showed aluminum content ranging from 39200-72800 mg·kg^{-1} d.m., calcium content of 26700-34800 mg·kg^{-1} d.m., iron content ranging from 17800-30200 mg·kg^{-1} d.m., magnesium at 4470-8630 mg·kg^{-1} d.m., sulphur (2700-3720 mg·kg^{-1} d.m.), potassium (2220-3270 mg·kg^{-1} d.m.), manganese (1160-2570 mg·kg^{-1} d.m.), silicon (204-348 mg·kg^{-1} d.m.) and sodium (134-277 mg·kg^{-1} d.m.) – Table 5. These analyses correspond with the preliminary exploratory analysis of compounds and elements contained in the sediments by XRF (Table 3). The sediments also contain barium, boron, chromium, zinc, copper, nickel, lead, cobalt, strontium, titanium, and vanadium (Table 5). Heavy metals mainly come from industrial and agricultural activities and chemicals (Ahmad et al. 2017, Crittenden et al. 2012, Anjithan 2016).

Phosphorus is the main element that causes the eutrophication of freshwater bodies (Gao et al. 2013). The analytical results of total phosphorus in the tested sediments in the selected WTP ranged from 1200 mg·kg^{-1} d.m. to 1910 mg·kg^{-1} d.m. According to Karunanithi et al. 2015 and Tekile et al. 2015, biogenic compounds, including phosphorus, can cause several water quality problems in the WTP, such as unacceptable odor and taste of the treated water and technological problems with filter bed clogging. It is, therefore, of utmost importance that phosphorus is effectively removed in coagulation treatment processes. Muisa et al. 2020 compiled the phosphorus contents removed in selected WTPs in treatment processes using aluminum salt coagulation, which varied considerably (from 2000-43000 mg P·kg^{-1} d.m. of sludge). The differences were due to the quality of the raw water subjected to the treatment processes and the chemical reactants used (Hou et al. 2018).

The sediments studied were formed by coagulating surface water from the river and infiltration water. The content of individual elements in the sediment samples is a result of the quality of the water flowing into the WTP, which is sine dependent on natural (surface run-off, floods, droughts, snowmelt) and anthropogenic conditions, as well as the type and amount of coagulant dosed. The hydrated sludge in the chamber is collected for 20-30 days, sedimented, and the supernatant water is separated from the dense sludge. The sludge sample is, therefore, concentrated and averaged.

The catchment area of the river that feeds the WTP is made up of formations belonging to the Magurian Mantle flish bedrock, and Miocene sediments made up of: clays (mainly siltstones), silts, and sands of marine origin, red and green-colored mudstones, Miocene-Pliocene formations – gravels, conglomerates and silts (Nickel et al. 2015). On the other hand, quaternary formations are mainly alluvial covers, loess-like clay and clayey covers, and slope formations. Literature data (Kicinska 2012) show a high average percentage (% concentration) of the following oxides in siltstone/sandstone rocks: SiO_2 (57/61%), Al_2O_3 (19/13%), CaO (8/16%), Fe_2O_3 (8/5%), K_2O (4/2%), MgO (3/2%), SO_3 (1/1%).

The water quality of the river is also affected by anthropogenic activities: the dam above the water intake, economic and industrial activities in the catchment area, lack of sanitary sewerage in areas located above the intake, and sewage treatment plants located above the water intake.

Comprehensive studies of post-coagulation sludge fill an important research gap and should be used to develop strategies for WTP users to deal with this type of sludge and to develop good operational practices in managers. The results of the WTP sludge studies clearly support the claim that this type of sludge cannot be discharged directly into natural water reservoirs (rivers, lakes, streams) and sewers (Ahmad et al. 2016a, Ahmad et al. 2016b).

WTS can be reused in agriculture as a soil component to improve soil nutrient properties (organic matter content, nutrients) and/or structure (hydraulic conductivity, water retention) (Moodley et al. 2004, Dassanayake et al. 2015). Research has been ongoing since 1970 to test the effectiveness of WTS admixtures on plant growth and environmental impacts (Russell 1975, Ippolito et al. 1999, Moodley et al. 2004, Lombi et al. 2010). The use of WTS in agriculture requires further research due to the presence of heavy metals, aluminum, and salt ions in the sediments (Babatunde & Zhao 2007).

As mentioned earlier, the possibility of using water decarbonation wastes for liming acidic soils (Ahmad et al. 2016, Sales et al. 2011) or in the production of building materials (cement, bricks, ceramic tiles, roof tiles, ceramic pipes, concrete, lightweight aggregates, mortar) has also been put forward (Cremades et al. 2011, Luo et al. 2008). Clay sludge can also be used efficiently to remove nitrogen, phosphorus, and organic matter (Babatunde et al. 2010) or as a heavy metal adsorbent from wastewater (Abo-El-Enein 2017). Another way to manage sludge is by recovering coagulants from post-coagulation sludge and CaO and CO_2 from calcareous sludge containing large amounts of $CaCO_3$ (Bishop 1987). Hydrated iron sludges are used to bind hydrogen sulfide generated in sewage networks in scrubbers to remove hydrogen sulfide generated during the anaerobic digestion of sewage sludge and slurry (Szerzyna, 2013, Jaroszyński et al., 2011). WTS can also be used to modify the classical sweep coagulation system by introducing recirculation of post-coagulation sludge (Wang et al. 2004, Xu et al. 2011), which reduces the negative effects of sweep coagulation, i.e., results in a reduction of aluminum remaining in the water after the coagulation process (Yang et al. 2011, Yan et al. 2007).

4. Conclusion

The analyses showed that the sediments are mainly inorganic in nature, consisting of sand, clay, and silt particles with grain sizes ranging from 0.001 mm to 1 mm. Silica (53.781%), alumina (23.582%), calcium oxide (8.28%), iron (III) oxide (5.610%), and potassium oxide (2.358%) – accounted for the predominant percentage of chemical constituents present in the sediments. The heat of combustion of the sludge on a dry weight basis averaged 3.39 MJ·kg d.m.$^{-1}$ – this result disqualifies the sludge as a potential energy source. A significant correlation was observed between the amount of organic matter (TOC) and nitrogen content (N_{Kj}) in the sediment scaling water, which developed similarly over the year. Site-specific sediment samples – the sediment chamber – had comparable percentages of elements, remaining at a similar level over the calendar year. Silicon and aluminum had the highest percentages in the sediment samples, which is justified by the nature of the coagulation processes carried out, i.e., the precipitation of, among others, turbidity-causing compounds with aluminum coagulants. Subsequently, the sediments also contained higher amounts of magnesium, calcium, and phosphorus. Admixtures of metals were also determined in the sediment samples. Analytical results: XRF, quantitative elemental, TOC, proved that the water content of turbidity, colour, aluminium, phosphorus, UVA254 and TOC determines the coagulation process in the studied WTP. The studied sediments were formed by the coagulation process of surface water from the river and infiltration waters. The content of individual elements in the sediment samples is a result of the quality of the water flowing into the WTP, which is sine dependent on the anthropogenic conditions and the type and amount of coagulant dosed.

Comprehensive studies of post-aggregation sludge fill an important research gap and should be used to develop strategies for WTP users to deal with this type of sludge and to develop good operating practices in managers. The results of the WTS studies clearly support the thesis that this type of sludge cannot be discharged directly into natural water reservoirs (rivers, lakes, streams) and sewers. WTS sludge can be used for, among other things: soil enrichment in agriculture, production of construction and filtration materials, absorption of heavy metals from wastewater, improvement of the efficiency of the sweep coagulation process, and the removal of hydrogen sulfide. The authors will continue to research and explore alternative methods for disposing and safely reusing WTP sludge in other industries.

Statements and Declarations

CRediT authorship contribution statement

J. Ciuła: Conceptualization, Methodology, Software, Validation, Formal analysis, Investigation, Resources, Data curation, Writing – original draft, Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition. **I. Wiewiórska:** Conceptualization, Methodology, Software, Validation, Formal analysis, Resources, Data curation, Supervision. **J. Kulczycka:** Formal analysis, Resources, Writing – original draft, Visualization, Conceptualization, Software, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review & editing. **J. Willson:** Methodology, Software, Validation, Formal analysis, Investigation, Resources, Writing – original draft, Writing – review & editing, Visualization, Supervision, Project administration.

Declaration of generative AI and AI-assisted technologies in the writing process

Statement: During the preparation of this work the authors used ChatGPT, an AI language model in order to improve the readability and language of this work. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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