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Electrocoagulation of model wastewater in the recirculation system — liquid and solid phase testing

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ABSTRACT

This paper presents the results of wastewater treatment and the properties of sludge aggregates obtained by model wastewater electrocoagulation. Wastewater treatment was effected in a recirculation flow system using aluminium electrodes, at an amperage of 0.1 A. Electrocoagulation of wastewater with the following initial chemical oxygen demand (COD_o) was performed: 1,260 and 780 mg/L. Wastewater with a lower COD_o and lower initial impurities load produced better treatment results: 63% COD removal, 98% turbidity, color, and 100% suspension removal. The fractal properties of the aggregates obtained during the electrocoagulation process were also examined. Sludge aggregates formed in wastewater with a higher purification level had larger fractal dimensions and smaller real dimensions. Data on sludge characteristics can be useful in selecting the treatment method and designing devices for its separation.

Keywords: Electrocoagulation; Fractal dimension; Model wastewater; Sludge aggregates

1. Introduction

Legal regulations implemented around the world necessitate actions aimed at limiting environmental pollution. Community or industrial wastewater, including wastewater from the paper industry [1,2], discharged to natural aquatic ecosystems must have a very low pollution load. Commonly applied biological, chemical, and physico-chemical treatment methods do not always prove efficient and cost-effective.

In an alternative method of wastewater treatment, biological and physico-chemical treatment processes can be combined in the treatment tank with an electric current flowing through the wastewater. Owing to a considerable improvement in electric current effectiveness and an increase in its yield, the electrocoagulation (EC) method became competitive and effective, for example, in the removal of metals [3,4], dyes [5,6], organic matter [7,8], solid suspensions [9], and even arsenic [10,11] from water and wastewater.

Electrocoagulation relies on coagulant formation in situ via electrolytic dissolution of the electrodes. Metal ions traversing to the solution create hydroxides, which possess the capability to absorb the pollution. The electrocoagulation process is therefore similar to traditional coagulation. It is, however, accompanied by numerous phenomena associated with the electric current flow, such as water electrolysis and hydrogen production. The bubbles of gaseous hydrogen carry the absorbed pollution to the surface of the liquid, where they can be removed, for example, by scraping. There is a lot of relevant literature confirming the effectiveness of electrocoagulation in wastewater treatment. The literature provides examples of electrocoagulation applications in purification and neutralisation: wastewater from the textile industry [12,13], from the food industry [14,15], from a car wash [16], electroplating industry [17], and other impurities [18].

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For electrocoagulation to perform a significant role in wastewater treatment, further research is needed focusing on interactions between electrochemistry, coagulation, and floatation, and their effect on the quality of the liquid. The creation of large amounts of sludge remains a relevant problem associated with wastewater treatment processes. The sludge needs to be dehydrated, removed, and then safely disposed of. In the electrocoagulation process, sludge is also created, although in lesser amounts compared to chemical coagulation. The sludge structure is closely associated with the process of the treatment. For example, the exact method of performing the electrocoagulation can permanently affect the created aggregates – flocs. The high removal efficiency of EC depends on the aggregation of flocs with large, strong structures. There have been studies that have focused on the settling characteristics and sludge volume index of electrocoagulation sludge. However, the floc growth during the EC has not been thoroughly studied, and their structures have not been described in detail, compared to the flocs formed by chemical coagulation.

The paper-pulp wastewater is a particularly difficult group of industrial wastewaters, characterized by high levels of organic waste that are harmful to the receiver. According to the author's knowledge, a few papers have been published on the treatment of pulp and paper wastewater by EC that describe the structure of the formed flocs.

The aim of this paper was to compare the effectiveness of treating model paper-pulp wastewater of different loads by electrocoagulation performed in a recirculation system against the duration of the process under the constant conditions of current density = 4.5 mA/cm^2, pH = 6. The paper also aims to characterize the created sludge in the EC and discuss the influence of the wastewater output load on the structure of the created aggregate's flocs. A study was performed to verify whether there is a correlation between the degree of effectiveness of wastewater treatment and the size of the fractal post-coagulation sludge.

2. Materials and methods

2.1. Wastewater characteristics

The wastewater treatment was conducted in laboratory conditions. This study used model wastewater to ensure the repeatability and reproducibility of results [19]. The wastewater composition was similar to real wastewater from a paper industry site. The wastewater was obtained by treating pinewood sawdust with chemicals at an increased temperature. The experiment was performed on: wastewater I – high-load, with an initial COD_o of 1,260 mg/L, and wastewater II – low-load, with an initial COD_o of 780 mg/L. The wastewater treatment was affected by electrocoagulation with a recirculation system for 4 h.

2.2. Experimental procedures

The study can be divided into two stages:

- examining the wastewater after electrocoagulation treatment – liquid phase examination,
- examining the structure of sludge resulting from treatment – solid phase examination.

2.2.1. Wastewater liquid phase examination

The wastewater electrocoagulation was performed in a recirculation system. This method increases the possibility of contact between the flocs of hydroxide created during the process and the absorbed pollution. The use of recirculation can also provide a partial neutralization of the electrokinetic potential of the wastewater pollution due to the shielding effect from the surface charge of the recirculated post-coagulation suspension. Another benefit of recirculatory electrocoagulation is the ability to track the kinetics of the treatment process. A recirculation system applied in wastewater treatment enables full process control. An electrocoagulation recirculation system enables the performance of actions aimed at supporting additional wastewater purification between the quality control point and the outlet. The possibility of full process automation makes electrocoagulation an alternative to chemical wastewater coagulation. Therefore, the process can be used for effective impurity removal from wastewater.

An electrocoagulation system (Fig. 1) was applied in the following design: wastewater tank – dosing pump – electrolyser – power supply system.

The electrocoagulation system used in the process enabled wastewater to flow from the tank to the electrolyser. Wastewater recirculation was possible owing to the use of a dosing pump (1), which pumped wastewater from the tank (4) to the electrolyser (2) and back to the tank in a continuous manner. Electrolyser (2) with a capacity of 80 cm^3 was built of six aluminium electrodes separated from each other by 1 cm. Each electrode had dimensions of 10/1/0.1 cm. The electrode deployment enabled their optimal contact with flowing wastewater. With the use of the pump dosing the wastewater at a constant rate of 0.233 cm^3/s, 1 dm^3 was pumped from tank (4) to the electrolyser (2) and then back to the tank (4). The wastewater in the tank was stirred with the use of a magnetic stirrer (5), which prevented the accumulation of sludge on the bottom of the wastewater allowing it to pump along with the wastewater. An original power

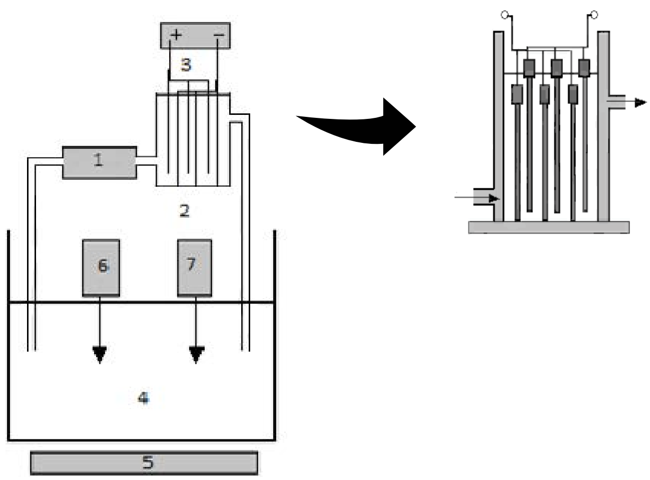

Fig. 1. The wastewater treatment set in the recirculation system with an electrolyser: 1 – dosing pump, 2 – electrolyser, 3 – power supply system, 4 – wastewater tank, 5 – magnetic stirrer, 6 – pH-meter, 7 – 1 M HCl.

supply system (3) was applied, which enabled running the process at a constant current density of 4.5 mA/cm^2 and an alternating current direction. The recirculatory electrocoagulation of the wastewater was performed for 15,000 s. To avoid the over-polarisation of the electrodes during the process and their periodic self-cleansing, the current direction was automatically alternated every 256 s. During the electrocoagulation of the wastewater, due to occurring processes, the pH is changed, which influences the entire treatment process. To avoid significant changes in the pH, a control system was introduced, maintaining the pH at around 6.0. It was achieved by using the pH-meter (6) and the burette to dose the HCl solution (7) with a concentration of 1 mol/dm^3.

2.2.1.1. Analysis

Liquid samples were taken for analysis from above the deposit after the electrocoagulation process was completed, after 30 min of sedimentation.

The effectiveness of electrocoagulation in wastewater treatment was determined by measurement of the following liquid phase parameters: COD, turbidity (TU), suspensions (SS), and colour. They were determined in three replications. They were carried out according to procedures given in the standard methods for the examination of water and wastewater – HACH method. The measurements were taken with a spectrophotometer, Hach Lange Dr 3900. The pH was measured using a CP-411 meter (Elmetron). The conductivity was measured using a conductivity meter CX-461 (Elmetron). The temperature of the wastewater was kept constant at about 25°C ± 2°C during the experiments.

The treatment efficiency (E) was determined in terms of maximum COD, colour, turbidity, and suspensions elimination percentage from effluent by the following equation:

$$\text{Treatment efficiency}\left(E\%\right)=\frac{C_o-C_t}{C_o}\times 100\% \quad (1)$$

where E = treatment efficiency (%), C_0 = initial concentration of pollutants, C_t = concentration of pollutants after the time (t).

2.2.2. Sludge structure analysis

The solid-phase examination included an analysis of sludge aggregates obtained following the wastewater electrocoagulation. The sludge structure analysis was based on the determination of the fractal dimensions D of the aggregates following their purification.

The sludge formed in the process was taken for analysis after 1 h of sedimentation. The fractal dimension was determined by the photographic method. Sludge aggregates were introduced to a column filled with distilled water and the route of their sedimentation was observed. For one experiment, 15 photographs were taken to capture an image of depositing aggregates and recorded in one frame. After the image was obtained, the dimension of a particle, R was measured and the floccule sedimentation rate, v was determined. Parameters of 100–126 aggregates were determined for one process.

The fractal dimensions were determined and all the calculations related to solid-phase aggregates were made with the FRACTAL 2 software based on the Microsoft Excel Spreadsheet.

3. Results and discussion

3.1. Description of the EC process

During the first stage, electrocoagulation is effected by electric charge flow between electrodes immersed in wastewater, which causes electrode reactions. The proposed mechanism of chemical reactions during the process is as follows:

$$\text{on the cathode (–)}\ \ 2H_2O+2e\rightarrow H_2\uparrow+2OH^- \quad (2)$$

$$\text{on the anode (+)}\ \ Al^{o}_{(s)}\rightarrow Al^{3+}_{(aq)}+3e \quad (3)$$

In this method, the coagulant is produced directly in the reaction as ions are released from electrodes by the flowing current. Electrochemically formed Al^{3+} cations react spontaneously and produce the respective hydroxide and/or poly-hydroxides in accordance with the complex precipitation kinetics. The aluminium ions that enter the solution form, depending on the pH, are monomeric or polymeric hydroxyaluminium ions: Al^{3+}, $Al(OH)_2^{+}$, $Al(OH)^{2+}$, $Al(OH)_3$, $Al(OH)_4^{-}$, $Al(H_2O)_6^{3+}$, $Al(H_2O)_5(OH)^{2+}$, $Al_2(OH)_4^{4+}$, $Al_6(OH)_{15}^{3+}$, $Al_8(OH)_{20}^{4+}$ [20]. The presented compounds differ in solubility, which affects their capability to coagulate pollution. the insoluble hydroxides $Al(OH)_3$ are the best coagulants. Their larger surface area and amphoteric nature enhance the coagulation process. According to Gupta et al. [21] their concentration is highest at a pH value between 6 and 7.

The solution pH changes during electrocoagulation, with the final pH considerably affecting the effect of impurities removal [22]. It has been indicated in several previous studies that the use of soluble anodes changes the solution pH during electrocoagulation. The wastewater pH during the process is mainly affected by OH^- ions formed by oxygen-related cathode polarisation:

$$1/2O_2\left(\text{water-soluble}\right)+H_2O+2e\rightarrow 2OH^- \quad (4)$$

According to Sharma et al. [23], Sridhar et al. [24], Shankar et al. [25], the optimum pH for pulp and paper wastewater treatment with electrocoagulation lies between 6.0 and 7.0. This is also the range within which aluminium hydroxide is the least soluble. Conducting the process in this pH range ensures the optimum wastewater purification and the minimum Al^{3+} residue in the effluent. As a result, the pH of the wastewater was monitored and adjusted with HCl solution to maintain it within this range.

3.2. Effect of sewage initial load and electrocoagulation time on wastewater treatment

Two types of wastewaters were used in this study, whose initial characteristic parameters are presented in Table 1.

Figs. 2 and 3 show the results of the wastewater liquid phase analysis following electrocoagulation, obtained for high-load wastewater I (Fig. 2) and low-load wastewater II (Fig. 3). Subsequent graphs show the percentage removal of COD, colour, turbidity, and suspensions depending on the duration of the process. In the initial period, up to around 15 min of electrocoagulation, both in high-load wastewater I and in low-load wastewater II, an increase in colour, turbidity, and suspensions was observed in the treated wastewater. A similar dependence was observed by Kumar et al. [26], the increase in colour in the first 10 min of the electrocoagulation process of the paper-pulp wastewater.

The initial increase of the aforementioned parameters might be caused by the production of the Al ions on the anode, however, their concentration is relatively low. The presence of a small amount of $Al(OH)_3$ during the initial phase of the continuous flow prevented impurities from adsorbing and increased the above parameters. Aggregation and flocculation of wastewater colloids did not take place until the threshold charge was exceeded, with an increase in Al^{3+}, or other aluminium species in the system. In the case of low-load wastewater II (COD_o = 780 mg/L), after 30 min, a significant raise in suspension elimination was observed, up to 50% (Fig. 3). For the heavy-load wastewater (Fig. 2), only 4% of the suspension SS was removed. A similar trend was observed in turbidity removal, with a 20% turbidity reduction for low-load wastewater in 30 min and no significant change for heavy-load wastewater, despite a minor increase.

The difference in effectiveness for the initial electrocoagulation period comes from the amount of introduced electric charge and the resulting concentration of corresponding Al ions. For the case of heavy-load wastewater, the concentration turned out to be insufficient to destabilize the system, aggregate wastewater colloids, and then sediment the resulting solution.

Finally, in the case of wastewater II (COD_o = 780 mg/L), almost 100% removal of turbidity, suspension, and colour was observed after 15,000 s of electrocoagulation (Fig. 3). The optimal time, however, remains 120 min, after which more than 90% of the colour, turbidity, and suspension are removed. After that time, the degree of COD removal amounted to 60%. The increase in the duration of the electrocoagulation did not lead to any notable improvements in the result.

Table 1
Initial parameters of the model wastewater under study

Parameters	Wastewater I	Wastewater II
pH	5.75	5.62
COD, mg·O_2/L	1,260	780
Colour, mg/L	1,595	1,140
Turbidity (TU), mg/L	375	230
Suspensions (SS), mg/L	145	75
Conductivity, mS/cm	2.85	2.44

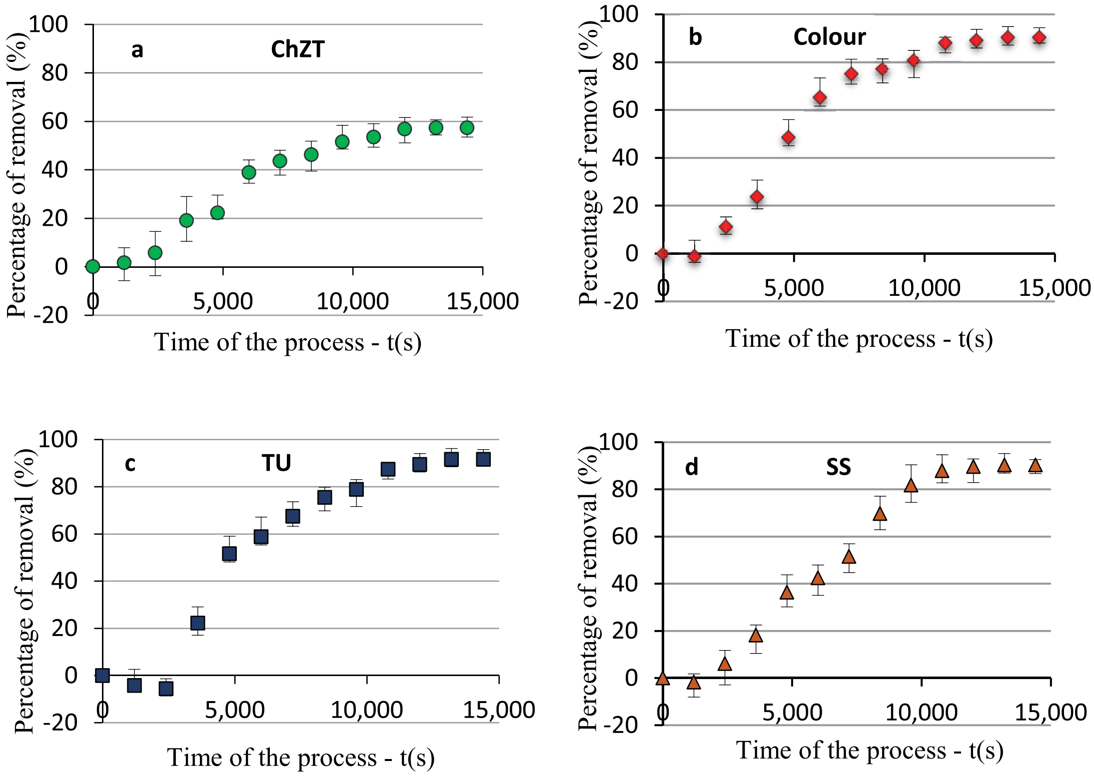

Fig. 2. Wastewater I parameter (COD_o = 1,260 mg/L) changes during the electrocoagulation process – the percentage of COD, colour, TU, and SS removal (% removal) vs. process duration (*t*).

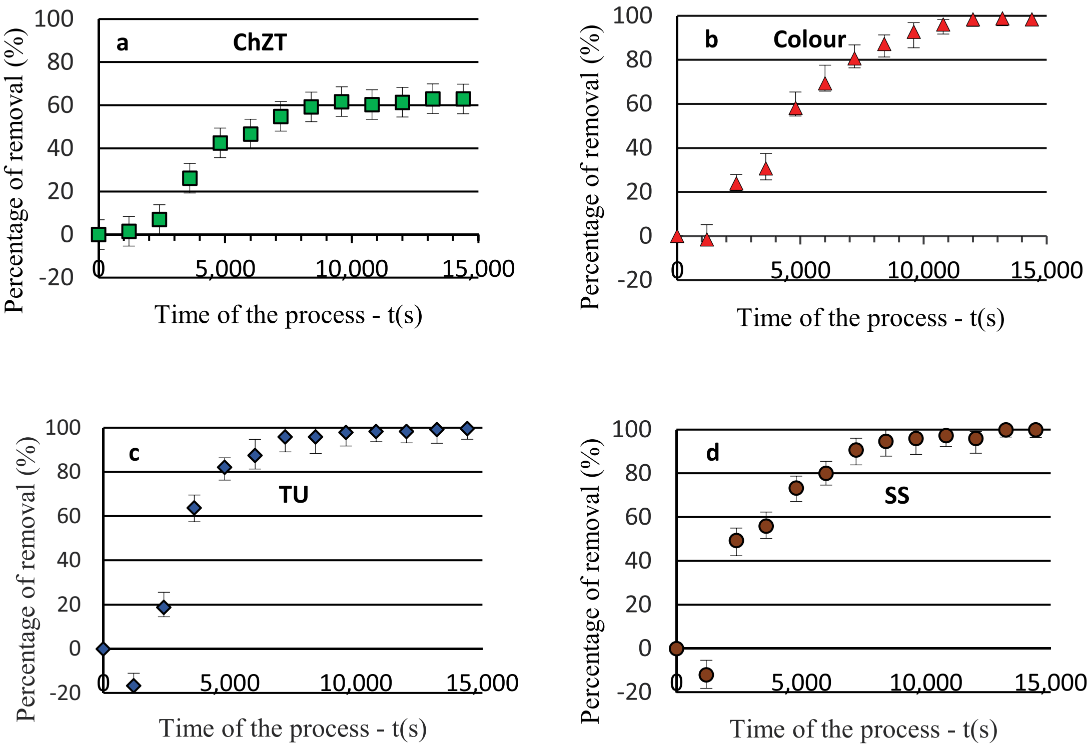

Fig. 3. Wastewater II parameter (COD_o = 780 mg/L) changes during the electrocoagulation process – the percentage of COD, colour, TU, and SS removal (% removal) vs. process duration (*t*).

Electrocoagulation in the recirculatory system also provided a high, almost 90% degree of TU and SS removal for the wastewater of COD_o = 1,260 mg/L (Fig. 2). To obtain such results, however, a time of 180 min was needed. Further increase in the process duration did not lead to improved results, ultimately failing to replicate the results obtained with electrocoagulation of wastewater with 780 mg/L loads. The COD degree of removal (Fig. 2) was increasing gradually, obtaining a value of 40% in the 100th min, and reaching 56% for the optimal time of 180 min. The remaining 70 min of the process, led to a minor increase of 2% in COD.

The duration of electrolysis in both cases turned out to be too long. The optimal duration for wastewater II (COD_o = 780 mg/L) was 120 min and for wastewater I (COD_o = 1,260 mg/L) was180 min. The effectiveness of impurities removal largely depends on the initial impurities load in the wastewater. For wastewater II (COD_o = 780 mg/L), more effective purification was observed (measured as nearly 63% of COD removal) than for the wastewater with initial COD_o = 1,260 mg/L. The purification of wastewater I reached the final constant level of 58% COD removal. The removal of turbidity, color, and suspensions was higher in wastewater II than in wastewater with a higher COD_o. A similar percentage removal of COD – 68% was obtained by Kumar et al. [26], in the electrocoagulation of pulp-paper wastewater with the initial load of COD_o = 584 mg/L, and initial pH of 7. In their study, the electrolysis time was shorter by 50 min, but the current density was almost six times higher – 25.20 mA/cm^2. The removal efficiency of 85% COD and 94% color has been achieved under the optimal experimental conditions of pH–7, a treatment time of 2 h, and a current density of 15 mA/cm^2 [23]. Shank et al. [25] reported that the optimum process conditions for the maximum removal of COD, and color from pulp and paper industry wastewater are pH ≈ 7, treatment time: 75 min, current density: 115 A/m^2. Under optimum operating conditions, the percentage removals of COD and color are ≈77, and 99.6%, respectively.

The extent of impurities removed from wastewater in the process was proportional to the electrolysis duration, that is, to the electric charge flowing through the solution during the electrocoagulation. Pollution destabilization mainly occurs through two separate mechanisms. One of them is the neutralization of negatively charged wastewater colloids by the products of cationic hydrolysis. The other one is the adsorption of the pollution on the surface of amorphic aluminum hydroxide. With lower COD_o values, that is, with generally smaller impurities load, the impurities load/adsorbent surface area ratio proved smaller, which perceptively increased the impurities adsorption capacity and resulted in a better effect of wastewater treatment.

3.3. Sludge characteristics

Solid particles formed by electrocoagulation, depositing at the bottom, tend to aggregate, that is, to join and form structures of a complex geometrical structure [27]. The shape and internal structure of an aggregate are described with a fractal dimension. This study made use of the possibility of describing aggregate particles with the mass fractal [28]. A mass fractal is a mass aggregate made of particles connected with each other – self-similar, that is, each sub-unit of an object has the same features as the entire aggregate. The mass

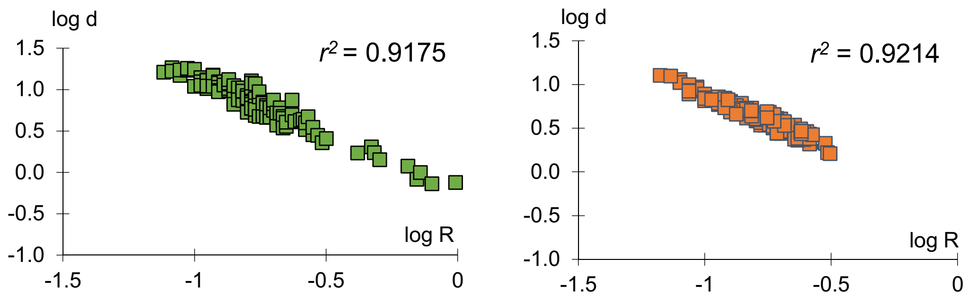

Fig. 4. Relationship log$d$ ~ f(log$R$) for wastewater I (r^2 = 0.9175) and wastewater II (r^2 = 0.9214).

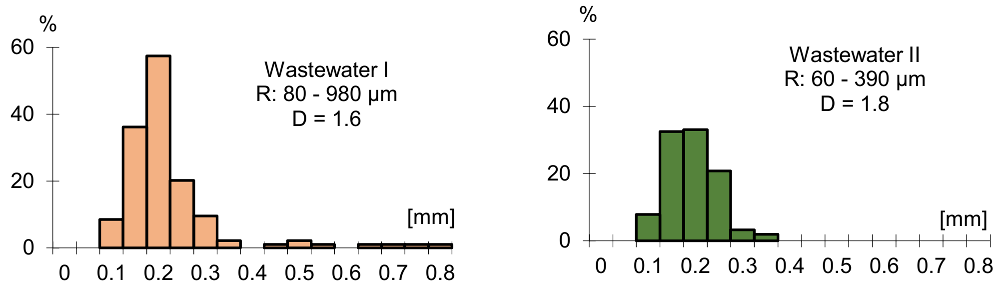

Fig. 5. Percentage of aggregates in sludge depending on their actual dimension – wastewater I and II

fractal D was determined from the relationship: $m(R) \sim R^D$, where: $m(R)$ – the mass of suspension particles, R – linear particle dimension.

The fractal dimension adopts the values from the range: $1 < D > 3$. In order to determine the mass distribution, one has to know the effective aggregate density. The density d of a mass fractal of dimension D was determined as a function of the distance from its centre: $d(R) \approx R^{D-3}$. The data were used to determine a logarithmic relationship: log$d$~f(log$R$), and the slope of the curves (Fig. 4) was used to read the value of D. Selected results are presented in Fig. 4.

The average slope index determines the fractal dimension of the aggregates. A high value of the determination indices r^2 determines the self-similarity of the objects under study [29]. Aggregate self-similarity suggests their fractal nature, and the mathematical model applied, log$d$~f(log$R$), fits an image analysis for the sludge obtained by purification. The presented (selected) aggregates had a fractal dimension of D = 1.60 for wastewater I and D = 1.80 for wastewater II. Załęska-Chróst and Wardzyńska [30], observed that there was a relationship between the wastewater purification extent and the fractal dimension: a higher purification extent – a higher D. Higher values of the fractal dimension denote sludge of lower hydration, which is easier to separate by further processing.

For both cases, D < 2 fractal sizes were obtained, suggesting the same aggregation mechanism for both – the cluster-cluster model, presented by Meakin et al. [31]. According to the model, in a given volume of space, there are identical and evenly-spaced molecules. When both molecules undergo contact, they are irreversibly connected, while preserving their orientation. A dimer can connect with other dimers or a singular molecule. The result of a computational simulation performed in the 3D space, Meakin et al. [31] found a value for such aggregation mechanism to be D = 1.75.

The photographic method applied for aggregate examination allowed the acquisition of data on particle size distribution in sludge. Fig. 5 shows the percentage of aggregates in sludge depending on their actual dimension. The analysed objects had a diameter of 80 to 980 µm for wastewater I under treatment, for which the average sedimentation rate was 0.10–0.73 mm/s; the aggregate size dispersion was smaller in wastewater II (80 to 400 µm), for which the purification degree was better, and the sedimentation rate was 0.05–0.19 mm/s. Small aggregates, with small dimensions, account for the majority of the sludge. It is a positive feature due to better filling of space with the solid-phase and lower hydration of the sludge [32].

Studying aggregate properties can have practical significance in many important processes of chemical engineering, for example, in sludge filtration. Information on the particle size is useful when choosing a method of purification and designing and operating devices used to eliminate suspensions and sludge from water or wastewater [33].

4. Summary and conclusions

Electrocoagulation is a complex wastewater treatment process that is dependent on numerous parameters and phenomena. One such parameter is the load of the output wastewater. Finding the fractal size D of the created aggregates of wastewater sludge is a way to quantitatively describe the irregularities or to define the degree of irregularities of the object. It can also point to the mechanism of aggregation.

In the experiment, it was found that electrocoagulation performed in a recirculatory system with the use of aluminium electrodes at a current density of 4.5 mA/cm² and pH correction to the value of 6.0 resulted in 63% COD removal, with an initial COD_o of 780 mg/L and nearly complete elimination of colour, turbidity, and suspensions from wastewater. The optimal duration of electrocoagulation was found to be 120 min. Lower treatment results were achieved for wastewater with an initial COD_o of 1,260 mg/L, where 58% COD removal was obtained and 90% removal of color, turbidity, and suspension. For this load, the optimal time of EC was found to be 180 min. The effectiveness of impurities removal largely depends on the initial impurities load in the wastewater.

Studying the post-coagulation sludge, it was noted that the sludge flocs obtained in the process of recirculatory electrocoagulation possess the same morphological qualities. Each subunit of a given object has the same qualities of the aggregates/flocs. Aggregate self-similarity suggests their fractal nature and therefore aggregates of sludge obtained by electrocoagulation treatment can be described by fractal geometry with the fractal dimension.

The structure and properties of post-coagulation sludge have been influenced by the load of the output wastewater. The sludge that was created during the treatment of wastewater of COD_o 1,260 mg/L was characterized by the fractal size of $D = 1.60$, a diameter of 80 to 980 μm, and an average sedimentation rate of 0.10–0.73 mm/s. However, the sludge created in the treatment of wastewater of COD_o 740 mg/L has a higher fractal size of $D = 1.80$, a diameter of 80 to 400 μm, and an average sedimentation rate of 0.05–0.19 mm/s. The lower load wastewater has created the sludge of a denser structure (higher D), more susceptible to dehydration with a higher part of smaller aggregates. This guaranteed that a larger part of post-coagulation sludge was in a solid state. In the high-load wastewater treatment, more open-structure sludge flocs were formed with a more pleated structure (lower D), more resistant to dehydration.

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