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GRANULOMETRIC ANALYSIS TO ESTIMATE INFLUENCE OF ANIONIC SURFACTANT ON ACTIVATED SLUDGE STRUCTURE

ZASTOSOWANIE ANALIZY GRANULOMETRYCZNEJ DO OKREŚLENIA WPŁYWU SURFAKTANTU NA STRUKTURĘ OSADU CZYNNEGO

Abstract: The work concerns the evaluation of the possibility of using laser diffraction analyzer to determine changes in the distribution of sludge floc size in the presence of anionic surfactant. As a model of surfactant linear alkylbenzene sulphonate (LAS) was used. Different concentrations: 10, 100 and 200 mg/dm^3 of LAS was applied. The granulometric analysis showed that the pure activated sludge was composed of particles with a size of 1 μm to 2 mm. The largest and fastest changes in the distribution of sludge floc size after the addition of a surfactant at a concentration of 100 mg/dm^3 were observed. The flocs with a diameter above 0.8 mm disappear and the average diameter over volume of particles decrease from 0.29 to 0.19 mm. Doubling the dose of surfactant causes the flocs aggregation. The sludge flocs capability for break-up and aggregation is not proportional to the concentration of LAS. Addition of LAS to the sludge results in decrease of the sludge volume index. This study showed that granulometric measurement may help to estimate an impact of chemical substances contained in wastewater on activated sludge.

Keywords: laser diffraction method, activated sludge, floc size distribution, anionic surfactant

Introduction

Ability to assess the size of activated sludge flocs is one of the important and useful elements of the biological wastewater treatment process analysis. Knowledge of the floc

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size can indicate possible malfunction of wastewater plant. There is a number of technological parameters that influence size of activated sludge flocs such as sludge age, loading as well as aeration method. In well functioning activated sludge bacteria are present mostly in the form of large and medium size flocs. The morphology of activated sludge is also influenced by the composition of the influent of wastewater, particularly the presence of toxins that can cause the formation of small and incoherent flocs [1, 2]. High concentrations of surfactants in surface waters have a negative impact on the environment: hinder the penetration of oxygen into natural waters therefore limiting the development of living organisms and inhibiting self-purification processes [3, 4], are very susceptible to adsorption on sediments [5, 6] and bioaccumulate in living organisms. The presence of synthetic surfactants in the wastewater flowing into the mechanical-biological treatment plants can contribute to serious disturbances in the biological wastewater purification processes [2].

Anionic surfactants concentrations in municipal wastewater generally do not exceed 25 mg/dm^3, but even at such low concentrations can have negative effects on biological wastewater treatment, due to the morphological changes of activated sludge [7]. Concentrations of anionic SPC in industrial wastewater reach values of 300 mg/dm^3 [8, 9]. Such high concentrations of surfactant inhibit the nitrification process [10] changes the structure of activated sludge and its enzymatic activity [2], and thus affect the performance of wastewater treatment.

For the determination of flocs size, microscopic analysis is mostly used. In the works based on digital technology a number of microscopic images of activated sludge has been analyzed [7, 11]. The basic parameter in image analysis is the mean projected area of floc. This technique also allows the assessment of floc morphological features, floc size and the designation of the floc circularity index. Despite numerous advantages, this technique is time consuming, obtaining a representative sample requires a large number of measurements, and microscopic image interpretation can be very difficult. In the case where the object of interest are only particle size rather than their morphological features, measurement technique based on the laser light diffraction, can be used [12–14]. This method allows to obtain analytical results in a very short time.

The operation principle of diffraction analyzer is based on laser light diffraction phenomenon, also called low angle laser light scattering (LALLS) [15]. The laser light is scattered by the suspended particles, and the angle of refraction is inversely proportional to particle size. Although the size of floc measured by this method is not a real value, but may be treated as a parameter describing the examined sample of sludge [13]. Determination of the size of sludge flocs occurs indirectly by calculating its volume. This reduces the error associated with the ambiguity of this assessment, which dimension is measured. By Wilen et al [16] and Bushell [17], this method is valid in the case of particles characterized by small values of particle refractive index and a loose structure.

In considering the spherical particles and for small angles of refraction, this phenomenon can be described by analogy to the diffraction of light through the slit. The split light intensity I (θ) describes the following relationship:

$$I(\theta)=\frac{1}{\theta}\int_{0}^{\infty} r^{2}n(r)J_{1}^{2}(k\cdot r\cdot\theta)dr \qquad (1)$$

where: θ – angle of refraction of light on particles,
r – equivalent particle diameter,
$n(r)$ – particle size distribution,
$k = 2\pi/\lambda$, λ – laser's wave length,
J_1 – Bessels function of the first kind.

Measurement of the intensity of light split by multiple-element type detectors allows, after the transformation of equation (1), to determine particle distribution function $n(r)$. Mathematical description of this phenomenon are provided by McCave and Syvitski [18]. More information on the principles of diffraction analyzer can be found, among various papers, in the publication of De Boer et al [19].

In this work the laser diffraction analyzer was used to determine the granulometric composition of activated sludge from SBR reactor of wastewater treatment plant (WWTP) and to determine what changes occur and are influenced by the presence of an anionic surfactant. Study was carried out for different concentrations of LAS, typical of both, municipal and industrial, wastewater

Materials and methods

Materials

– The activated sludge used in experiments was taken from municipal wastewater treatment plant in Swarzewo. The biological unit of this treatment plant is anaerobic/aerobic sequencing bath reactor (SBR) with biological nutrients removal. The plant treats about 10.000 m^3 of wastewater daily (97 % domestic sewage). The composition of wastewater is generally constant with mean anionic surfactant concentration about 7 mg/dm^3. Suspended solids of the sludge samples were 5.6 g of dry mass per liter.

– The activated sludge for experiments was taken directly from the aeration chamber of SBR during the sedimentation phase, collected in 5 dm^3 containers and immediately transported from WWTP to laboratory.

– The control sample ("background") for all experimental series was oxygenated activated sludge without surfactant.

– In each series of measurements to 1 dm^3 of activated sludge, surfactant at concentrations of: 10 mg/dm^3 ($Serie_{LAS10}$), 100 mg/dm^3 ($Serie_{LAS100}$) or 200 mg/dm^3 ($Serie_{LAS200}$) was added. This corresponds to surfactant loads of: 1.8 mg/g d.m, 17.9 mg/g d.m. and 35.7 mg/g d.m. respectively, for the abovementioned series. LAS concentration range was chosen based on previous studies [20].

– Linear sodium dodecylbenzene sulphonate (LAS) from Sigma Chemical company Ltd has been used as an anionic surfactant.

– Before the granulometric measurement, for each series of test, the sludge sample was diluted (1/5).

Methods

In order to determine the effect of LAS to size change of the activated sludge flocs, its granulometric composition has been studied. Effect of surfactant addition to floc size change was measured over time, *ie* 5, 15, 30 and 45 minutes after the start of testing. Each measurement was conducted as a series of 15 replications.

The measurements were performed using laser diffractometer Mastersizer 2000 (Malvern Instruments Ltd), with unit Hydro 2000MU (integrated stirrer and pump). Particle size range measured by the instrument, through the use of red and blue laser beams, is in the range from 0.0002 mm to 2.0 mm. Analysis of the results is based on Mie theory and Fraunhofer theory [19]. The results of sludge flocs size analysis are presented as histograms, which allowed to evaluate the distribution of "grain" of the analyzed activated sludge.

The Mastersizer software generates a basic floc size distribution parameters (D[4,3], d(0.1); d(0.5); d(0.9)) In order to describe the mean particle size, the volume-weighted average diameter, D[4,3] was used. This parameter is calculated as:

$$\mathrm{D}[4,3] = \frac{\sum_{i=1}^{n} d_i^4}{\sum_{i=1}^{n} d_i^3} \quad (2)$$

where: d_i – the diameter of the particle with size i.

The influence of the steering on the fragmentation of sludge flocs was examined for the "background" sample. The 1 dm^3 of diluted activated sludge, without surfactant, at temperature 20 ^{o}C, was stirred for 5 min at a speed of pump 900 RPM. This velocity ensured good mixing of sludge without floc breakdown. No changes in particle of sludge distribution was noted. All other measurements were carried out at the same conditions

For each test series ("background", Serie$_{LAS10}$, Serie$_{LAS100}$ and Serie$_{LAS200}$) sludge volume index (SVI) was measured according to standard methods [21] in a 1 dm^3 settling cylinder. SVI is the volume of 1 g of the total suspended solids after 30 min of settling. This parameter is correlated with compressibility of sludge.

Results and discussion

After the addition of LAS at concentrations of 10, 100 and 200 mg/dm^3 for each sample of activated sludge differences in the composition and size of the flocs were identified. These differences depended on the concentration of added surfactant. A wide range of particle sizes is defined as the percentage of particles (v) of diameter (d_i) represented by a distribution function $F(d_i)$ (Fig. 1–4). Studies have shown that activated sludge flocs taken from the SBR reactor (forming the background for further research), significantly differed in size. The sludge consisted of both, a particle size of about 1 μm and larger with substitute diameter equal to 2 mm. Granulometric analysis also showed that the flocs exceeding 0.25 mm accounted for half of all flocs in the sample volume. The distribution of large flocs size in the range from 0.7 mm to 2 mm was only about 6.0 % of all particles volume. No change in the sludge flocs size over time was observed for activated sludge without the surfactant addition, indicating good reproducibility of the test method used.

After the addition of LAS at a concentration of 10.0 mg/dm^3 (Serie$_{LAS10}$) data analysis showed a significant curves reversal trend $F(d_i)$ in the direction of the smaller size of the activated sludge floc. The reason for this phenomenon was the slow and steady fractionation of sludge flocs taking effect during 30 minutes. Already after 5 min of adding the surfactant particles with sizes larger than 0.2 mm began to undergo fractionation. In addition, no flocs with size greater than 1.0 mm were identified (Fig. 1).

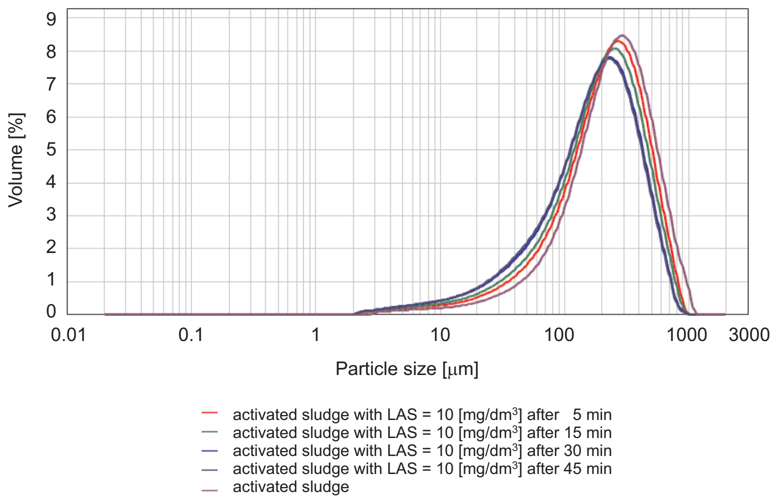

Fig. 1. Particle size distribution of activated sludge for background and Serie$_{LAS10}$ after 5, 15, 30 and 45 minutes

After a longer time (30 minutes after the start of the tests) the floc size decrease rate observed in the sludge slowed down. After 45 minutes the percentage of floc size in the range from 0.7 mm to 2 mm compared with activated sludge forming the background, was almost six times lower and was only 1.0 %. While the share of floc with a size to 0.25 mm increased and was 67 % of all particles in the sample.

The significant and rapid fractionation of sludge flocs was observed after the addition of a surfactant with ten times higher concentrations (Serie$_{LAS100}$). In contrast to the Serie$_{LAS10}$ floc fractionation did not occur gradually but stabilized after 5 minutes of the surfactant impact (Fig. 2).

The percentage of particles larger than 0.7 mm decreased from 6 % to 0.5 % and the percentage of the particles with a diameter of 0.25 mm increased from 50 % to 73 %. The percentage of the average-size flocs, with the best properties of the sedimentation

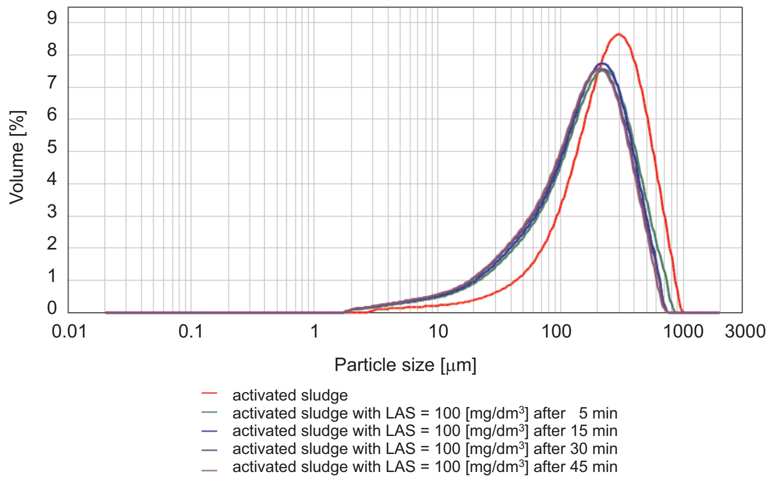

Fig. 2. Particle size distribution of activated sludge for background and Serie$_{LAS100}$ after 5, 15, 30 and 45 minutes

ability, was reduced by only 4.5 %, compared to their content in the sludge without surfactant.

Further doubling of the surfactant dose (Serie$_{LAS200}$) does not result in sludge breakage. After the addition of LAS at a concentration of 200 mg/dm^3 quite different results were observed. With such a large dose of LAS curve $F(d_i)$ "moved", after 5 minutes, in the direction of larger size flocs and were created flocs reaching the size of 2.8 mm, thus resulting in size increase beyond the size attained by activated sludge flocs without surfactant (Fig. 3). The reason for this phenomenon was probably due to the

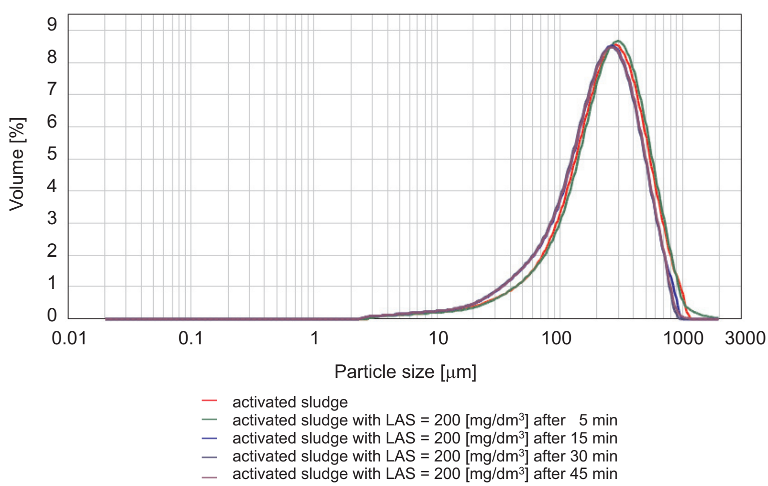

Fig. 3. Particle size distribution of activated sludge for background and Serie$_{LAS200}$ after 5, 15, 30 and 45 minutes

sludge flocs combining into larger aggregates. Over time, part of large aggregates underwent break-up, therefore number of small floc size 0.02–0.2 mm slightly increased and this kind of particle distribution was observed in measurements at 30 and 45 min.

The results of the study showed that for all the concentrations of added surfactant, the size distribution of the activated sludge flocs after 30 min is stable. Figure 4 shows the distribution function $F(d_i)$ for activated sludge (background) and all test series after stabilization of the changes taking place under the influence of surfactant. The displacement of the curve $F(d_i)$ in the direction of the smaller particle size was observed. Dominant particle size decreased from 0.3 mm for "background" to about 0.2 mm for Serie_{LAS100}.

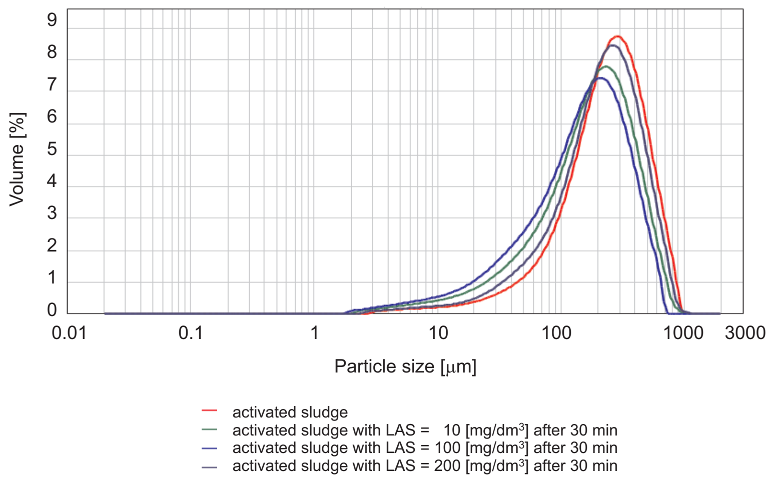

Fig. 4. Particle size distribution of activated sludge for background and series of sludge with LAS, after 30 minutes

Detailed values of volume-weighted average diameter and diameter deciles, obtained after 30 min of measurement are presented in Table 1.

Table 1

Deciles and median for values obtained for background and series of sludge with surfactant after 30min of measurement

Serie	Parametr [µm]			
	D[4,3]	d(0.1)	d(0.5)	d(0.9)
"Background"	293.84	72.35	248.52	579.88
Serie_{LAS10}	215.92	40.06	184.28	438.83
Serie_{LAS100}	189.78	31.14	159.81	394.02
Serie_{LAS200}	250.06	57.27	217.69	490.26

Important role in the binding of microorganisms in compact aggregates are extracellular polymeric substances (EPS), which are a kind of a matrix in which microorganisms are embedded [22, 23]. EPS matrix is not uniform, it can be divided into two fractions which differ greatly in composition and properties. The tightly bound EPS (TB-EPS) is strongly associated with the fraction of microorganisms, containing significant quantities of trivalent and divalent metal ions (Fe^{3+}, Al^{3+}), and is responsible for maintaining a compact structure of floc. This bond is formed by the influence of the negatively charged surface of the TB-EPS of positively charged cations. The loosely bound EPS (LB-EPS), is a fraction of the surrounding floc, more from mobile EPS TB [24]. Significant amounts of this fraction can weaken the bond between the microbial cells and contribute to the deterioration of floc cohesion [25]. Surfactants, due to their properties may lead to biopolymers leaching from the EPS matrix to the supernatant [26]. Used in the studies anionic surfactant LAS, due to its electric charge can also interact with the cations present in the TB-EPS, weakening the structure of the flocs. The presence of a net negative surface charge on floc surfaces may create repulsive electrostatic interactions. These processes may be the cause of the observed, in granulometric analysis, increase of floc fractionation.

Floc aggregation observed in the case of LAS concentration of 200 mg/dm^3 indicates different effects of LAS at so high concentrations. It is known that floc aggregation increases with the content of proteins and nucleic acids in the EPS [23, 27]. Such a high LAS load can lead to cell lysis, which facilitates the release of biopolymers from bacterial cells (disintegration of part of bacterial cells).

It has been reported that there is no simple relationship between the floc size and its compressibility [12, 23]. A comparison between SVI and floc size measurements is presented in Fig. 5.

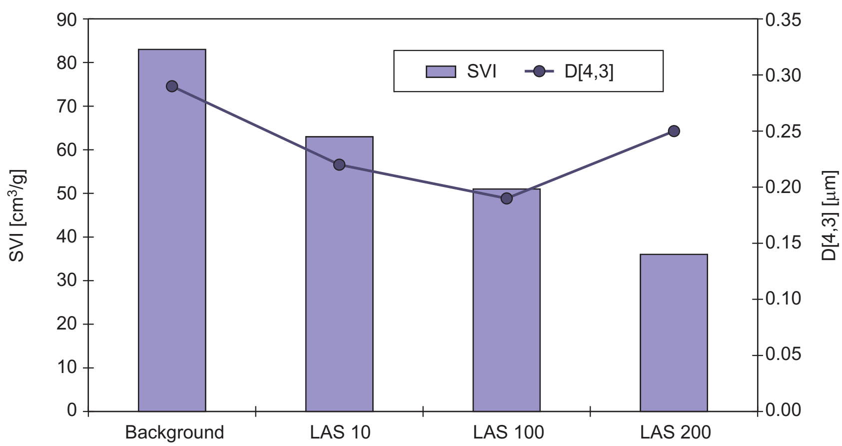

Fig. 5. SVI and D[4,3] evolution with increase of LAS concentration

For all series the sludge volume index value decreases with increase of LAS concentration. This trend is not disturbed even by the increase of floc size resulting with their aggregation ($Serie_{LAS200}$). The reason for this phenomenon may be an increase (in the presence of surfactant) in sphericity and smoothness of the floc borderline, described in the work of [2, 7]. Due to their round morphology flocs could compact efficiently, giving a low SVI [23]

An important factor for sedimentation is the presence of dispersed gas bubbles in the intensive aerated activated sludge, which may contribute to the floating of the sludge. The floc sedimentation may be hindered by air bubbles. Gas bubbles contained in the liquid with surfactant are not stabilized. Since gas density is less than the density of the liquid, the buoyancy force causes the escape of gas bubbles from the liquid in the de-aeration process. In an aqueous solution of a surfactant gas bubbles are stabilized through the creation of a thin layer of adsorption of surfactant molecules at the border of the liquid-phase air. As a result, the thermodynamic description of the interface of the follicle is changed and therefore it's interaction with the liquid and sludge [28]. Lowering the surface tension of a mixture of water and aerated activated sludge reduces air bubbles, has an effect on the speed of lifting, the contact time of the phases, and the interfacial surface area [29, 30]. Moreover, LAS substantially adsorbs on the surface of the floc [31], changing the interfacial surface and its interaction with the liquid. The result of these changes may be an increase in sedimentation rate and better compressibility of the floc in the sludge with a surfactant.

Summary and conclusions

Laser diffraction method is a modern method that can be used to determine the particle size distribution of activated sludge, as well as track the changes taking place in the sludge due to the changing conditions of wastewater treatment. It allows provisionally estimate an impacts of chemical substances contained in waste water on activated sludge. On the basis of studies of activated sludge subjected to the impact of anionic surfactant can be concluded as follows:

– LAS concentrations less than 200 mg/dm^3 cause fragmentation of sludge flocs. Surfactant concentrations higher than this value leads to their aggregation.

– The largest and fastest changes in the distribution of sludge floc size were observed after the addition of a surfactant at a concentration of 100 mg/dm^3

– The method allows to estimate the time after which added chemical compound affects the sludge floc size. It also allows you to assess whether it is a one-time change, or act in time.

– The compressibility of investigated samples of sludge is the result of many factors and it is difficult to clearly determine which factors are decisive. These result (after the addition of LAS to the environment) in decreasing of the volume-weight index of activated sludge in the presence of surfactant. This disproportion deepens with increasing surfactant concentration

It should also be noted that the granulometric size distribution is only a tool for determining the particle size. Changes in floc sizes is not a reliable indicator of the sludge condition, or its enzymatic activity. Further complementary research must be done in order to determine the impact of LAS on activated sludge and its wastewater functions.

It would be desirable to examine in the future whether, at a constant flow of wastewater with LAS, the fragmentation of the floc sludge changes its characteristics permanently, or the elimination of LAS allows the sludge to return to the original characteristics.

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ZASTOSOWANIE ANALIZY GRANULOMETRYCZNEJ DO OKREŚLENIA WPŁYWU SURFAKTANTU NA STRUKTURĘ OSADU CZYNNEGO

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Abstrakt: Praca dotyczy oceny możliwości wykorzystania analizatora dyfrakcji laserowej do określenia zmian w dystrybucji rozmiarów kłaczków osadu czynnego w obecności surfaktantu anionowego. Jako model surfaktantu, zastosowano liniowy alkilobenzenosulfonian sodu (LAS). W przeprowadzonych badaniach użyto różnych stężeń wyjściowych surfaktantu: 10, 100 i 200 mg/dm^3. Analiza granulometryczna osadu wykazała, że rozmiar cząstek osadu mieści się w zakresie od 1 μm do 2 mm. Największe i najszybsze zmiany w rozkładzie wielkości kłaczków osadu zaobserwowano po dodaniu surfaktantu o stężeniu 100 mg/dm^3. Zanikły kłaczki o średnicy powyżej 0,8 mm, a średnia średnica kłaczków zmalała z 0,29 do 0,19 mm. Podwojenie dawki surfaktantu powoduje agregację kłaczków. Stopień rozdrobnienia bądź aglomeracji kłaczków pod wpływem LAS nie jest wprost proporcjonalny do stężenia surfaktantu. Dodatek LAS do osadu czynnego skutkuje spadkiem indeksu wagowo-objętościowego Przeprowadzone badania wykazały, że analiza granulometryczna może być stosowana jako metoda ułatwiająca oszacowanie wpływu związków chemicznych zawartych w ściekach na osad czynny.

Słowa kluczowe: metoda dyfrakcji laserowej, osad czynny, rozkład rozmiaru kłaczków, surfaktant