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MEASUREMENT OF ACTIVATED SLUDGE PARTICLE DIAMETERS USING LASER DIFFRACTION METHOD

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Abstract: The paper presents a study on the possibility of using the laser diffraction method for measuring the size of the activated sludge particles. Susceptibility of the particles to mechanical disintegration, dependent on the programmed value of stirring intensity, was observed (stirring was caused by required dynamic flow of analysed suspension through the measurement unit). According to the conclusions presented in this paper, it may be assumed that the laser diffraction method can be applied for measurement of activated sludge particle diameters under the following conditions: 1) the size of activated sludge particles measured by the laser diffraction method is not a real value, but after standardisation of measurement conditions can be treated as a parameter describing the sludge; 2) the particle diameters of activated sludge should be stabilised before the measurement, *eg* by mixing in the measurement unit or by ultrasound waves application.

Keywords: laser diffraction method, activated sludge particles diameter

An activated sludge process efficiency is dependent on the properties of saprophytes and saprobes conglomerated in flocks [1-8]. Among the important properties of activated sludge you can enumerate *eg* metabolic activity [9, 10], ease of separation from treated sewage [9-11], susceptibility to densification and dewatering. These properties are notably related with the morphology of sludge flocks as well as with their diameters [9-13]. The species composition of an active sludge organisms may also inform about possible malfunction of wastewater treatment plant biological part [3, 5, 14, 15]. A safe use of dewatered sewage sludge, for example for agricultural application or recultivation of degraded areas, also depends on presence of heavy metals [16-18] and pathogenic organisms [19-21].

Particle diameter measurements can be conducted in many ways, and the most basic and the first to be applied were the microscope observation methods [11, 12, 22, 23]. Neis and Tiehm used, among others, the membrane filtration method. The examples of other

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methods which are reported in the literature are: change of electrical resistance, light blockage and field flow fractionations [24] or sedimentation [25]. Nowadays, an increasing number of reports bring information about the application of the laser diffraction method for flock diameter measurements [26].

Laser diffraction method is widely used in soil science and sedimentology [27-32]. Estimation of the particle size distribution using the laser diffraction method is based on the measurement of the laser light scattering on the measured particles. The less particle in the measuring system the bigger angle of scattered light [33, 34].

Biggs and Lant used laser diffraction for determination of effectiveness of disaggregation of flocs using sonication and then the flocculation dynamic of disaggregated activated sludge [35]. Similarly Nopens and co-workers used laser diffraction method for determination of flocculation dynamic [36]. Guellil with co-workers used laser diffractometer in measurements of transfer of organic matters between wastewater and activated sludge flocs [37]. Houghton used discussed method for determination of particle size analysis of digested sludge [38]. The laser diffraction was also used directly for measuring of activated sludge *flocs size distribution*, **FSD** [39, 40].

There is a big problem with study and comparison of results most of mentioned above papers. The authors carried out their investigation using different equipment and what is more important different measuring procedures. Unfortunately most of important information which are necessary to fully understand presented results are not included in the paper. For instance the information about the power of sonication is not sufficient because it characterises the source of ultrasounds - the energy deliver to the flocs depends first of all on the volume of the sample and next on the geometry of the measuring system. There is lack of the information of the pomp and stirrer speed during the measurement in the most cases - this parameters influence on the flocs disaggregation independently on the possible sonication. Moreover sometimes there is no information about the used theory (Fraunhofer or Mie theory is the algorithm which make possible recalculation of light intensity redistricted on the detectors on the particle size distribution). When authors used Mie theory it is not possible in some cases to find used by them the optical parameters of the sample (the Mie theory requires to define the refractive and absorption indexes of the measurement sample).

The aim of the study presented herein was to determine the effect of selected methodological aspects on the measurement of flocs (from activated sludge) size distribution using laser diffraction method.

Materials and methods

Method

The measurements were performed using laser diffractometer Mastersizer 2000, Malvern, UK, with two units Hydro MU and Hydro G. The measuring range for material analysed in liquid dispersion was 0.02÷2000 µm.

The laser diffraction method uses the effect of laser light dispersal (scatter) on the particles measured. The diffraction angle informs about the size of the particles. So, the pattern of light diffraction covering light detectors can be converted to the pattern of particle sizes measured. Two models are usually applied for particle size calculation - the Fraunhofer and the Mie theories [41]. The model choice is dependent on the studied particle sizes. According to the ISO 13320 standard, the Mie theory should be applied for particles with diameters below 50 μm, while for larger diameters the Fraunhofer theory can be used [42]. The researches' opinions given in the literature reports are divided in cases where there are particles of smaller and greater diameters in the mixture composition [43-45]. The Mie theory was applied in our calculations because individual bacteria and their colonies with particle diameter lower than 50 μm were a significant part of the studied suspension. According to the Mie theory the identification of optical parameters is necessary. The value of light refraction index for the continuous phase was set as for water (1.33). The values for dispersed phase were assumed as follows: index of refraction for light - 1.53, and absorption coefficient - 0.1. The assumed optical parameters can be treated as average when the measured mixture is heterogeneous [46].

Materials

Activated sludge sampled at a municipal *waste water treatment plant* (WWTP) with a capacity of approx. 80 000 m^3/day and employing C, N and P treatments served as the test material in the presented study. The sludge was sampled from the aeration chambers using 1.5 dm^3 containers.

The following methodological aspects were investigated:

- Retention time of samples before measurements. FSD was measured after 30 minutes from sampling (time necessary to bring the samples to laboratory) and after 3 hours after sampling. The aim of this stage of investigations was to show the sample stability in time.
- Sample preparation. Part of the samples was prepared using sonication and part measured without sonication. Sonication was used only in Hydro MU unit (after first series of measurements we decided that this unit is better for flocs measurements). Exposure duration of sonication covered the time of 4 minutes and the power of the applied ultrasound probe was 35 W (maximum power of ultrasound probe built in the device). The measurement in Hydro MU unit was carried in 800 cm^3 beaker which was filled in about ¾ of the maximum volume.
- Different units of the same device. In one unit (Hydro G) the stirrer is integrated with the pump and the operator has to adjust them together. In the second unit (Hydro MU) the speed of stirrer and pump were adjusted separately.
- Speeds of stirrer and pump. Because the measurement in based on the sample which is dynamically passing through the measuring cell the speed of stirrer and pump can influence on the measuring because of the disintegration of the flocs. Because of this we decided to not use the stirrer in the Hydro G unit (the stirrer and pump can be adjusted separately). The small weight of flocs and what follows easiness to homogenise the suspension was the additional justification for this decision.

The speeds of stirrers and pumps are presented in Table 1.

Table 1

The speeds of stirrers and pumps used for both units during the experint

Unit		**Speed [rpm]**				
Hydro G	Pump	1300	1600	1900	2200	2500
	Stirrer	0				
Hydro MU	Integrated stirrer and pump	1200	1500	1800	2100	2400

The lowest speed values for both units were determined experimentally as a minimal velocity value allowing the suspension to flow through the measuring cell. The 100 rpm difference in the lowest speeds was caused by the constant step of 300 rpm for both units and the different values of the lowest measuring rotational speed. The upper limit of stirring speed was applied to avoid the phenomenon of air bubbles induction to the measuring system - the air bubbles could be treated as the flocks.

Measuring procedure

Each measurement of particle size distribution was conducted as a series of 20 replications. A single measurement was adopted as 30,000 counts registered on the detectors during 30 seconds for red as well as for blue light. Hence, the duration of a single measurement was equal to one minute. The measurement replications were realized in a sequence, so the measurement time for one full studied series was equal to 20 minutes.

Mean values were not calculated for the results obtained for the 20 replications because it was assumed that intensive stirring and pumping might disintegrate the studied flocks. Changes in the medians and deciles (0.1 and 0.9) were observed as a measure of potential dynamics of changes in the successive measurements. The results obtained for the last, 20^{th}, measurement were used to compare the different manners of suspension treatment (various intensities of stirring and pumping during the measurements).

The volume of sludge used for the measurements was determined by *obscurance* - a parameter measured by the device during the dosing process. With respect to measurement reliability, the number of particles for which the *obscurance* parameter value covers the range of 10 to 20% is optimal [47]. Concentration of the studied suspension, obtained directly from the wastewater treatment plant, was too high, so the suspension was added to filtered water to obtain the recommended standard of the *obscurance* parameter. When because of overdosage of suspension to the measuring system obscuration was too high the procedure of dilution was used [48].

The primary goal of our studies was to determine the influence of measurement conditions on the obtained results of studied particle diameters. Thus, the assessment of suspension stability during the examinations was required - it had to be checked whether the diameters of particles (especially flocks) were variable during the measurement. To assess the potential changes in particle sizes, the results obtained for samples just provided from the WWTP and for those sampled after the completion of all analyses were compared for the minimal Hydro MU settings (1200 rpm for pump and stirrer). The time between those two measurements was about 2.5 hr.

Results and discussion

In view of the fact that the microscopic measurement method of flock sizes used to be commonly applied, the following should be considered before the interpretation of the presented results:

Microscope-measured flock diameters are identified in real time in the microscope field of view and attain values of 100÷800 μm (and more) [9, 11, 12]. Microscopic measurements are realized beneath the covering glass, in a volume lower in the vertical dimension than the diameters of the measured structures. So, the measured flocks may be pressed and the obtained results may be overestimated relative to the real values.

Meanwhile, in a majority of actually conducted measurements based on the laser diffraction method, the obtained medians of flock diameters are covered by the range of 100÷150 µm and 0.9 deciles do not exceed the value of 250 µm. The medians in the results presented below were in the range of 60 to 80 µm. These values may be underestimated relative to the real values of flock diameters because the suspension was intensively stirred during the measurements, which could cause fragmentation of flocks.

The microscopic method enables precise identification of studied objects [2]. The observer can interpret not only the sizes of the measured flocks but also their morphology [9-13]. Such a possibility is unavailable in the case of the laser diffraction method. So, activated sludge flocks, bacteria and their conglomerates, protozoa, metazoa and others are the measured objects in this method. The presence of particles different than flocks may be the source of uncertainty in laser diffraction method measurements. However, considering the aim of the research and the possible changes of activated sludge particle diameters caused by measurement conditions (stirring and pumping), the presence of the other elements in the system does not limit the research scope because the dynamics of changes, if it occurs, is apparent by changes in the medians and deciles.

Stability of flock suspension during the experiment

The distributions of flock sizes obtained for the suspension provided directly to the laboratory, about 30 minutes after sampling (the 1st measurement), and for the same suspension but held at the laboratory for about 2.5-3 hours since sampling (the last measurement), are presented in Figure 1. The unit Hydro MU at 1200 rpm was used in these measurements. The 20th replication means that the presented measurement was the last repetition in the series of 20. Values of medians and two deciles of these distributions waves are presented in Table 2.

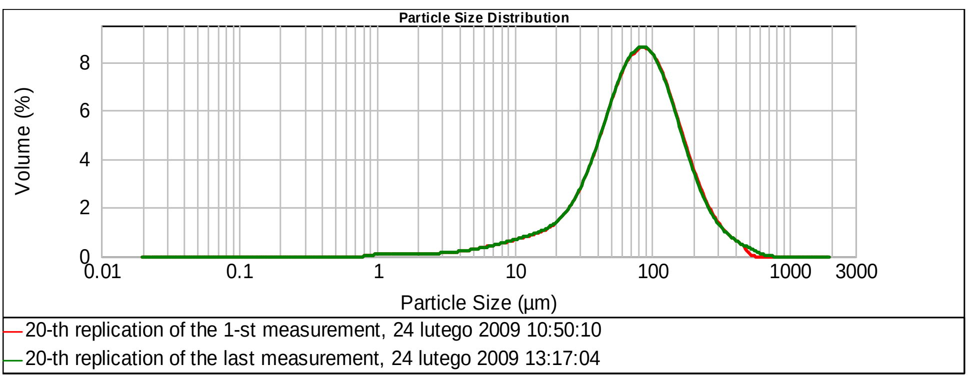

Fig. 1. Distribution of measured flock. Explanations: *the 1st measurement* means that measurements were conducted directly after sample delivery to the laboratory. *The last measurement* means that the measurement was conducted as the last in a series of 20; the time between the measurements was about 2.5 hr; both measurements were conducted on the Hydro MU units at 1200 rpm; *the 20th replication* means that the measurement was registered as the last, 20th replication in the series

Table 2

Median and decile values for distributions presented in Figure 1

	20th replication of 1st measurement	20th replication of last measurement
Median [μm]	78.862	78.492
d(0.1) [μm]	24.766	24.524
d(0.9) [μm]	191.549	191.035

According to the data presented in Figure 1 and Table 2, it can be assumed that the studied suspension was stable during the conducted measurements - the processes occurring inside the container with suspension sample were not reflected in any significant changes of flock sizes.

Effect of stirring intensity on flock sizes

Changes in particle sizes in the measurement unit for the device settings applied (constant rpm) were observed in the successive measurement replications. Examples of the results obtained for the median (d(0.5)) for the application of the Hydro MU with stirrer and pump speed of 1200 rpm are presented in Figure 2.

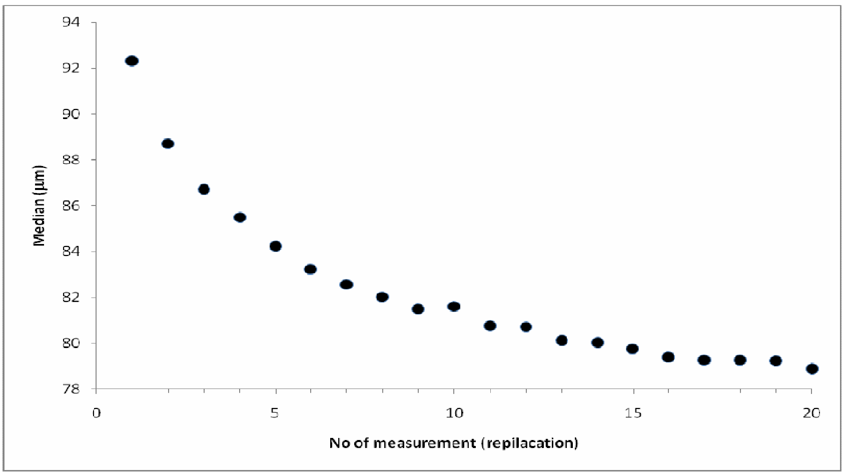

Fig. 2. Change in the dynamics of distribution medians for flock size during the successive measurements. The measurements were conducted on the Hydro MU at 1200 rpm

Practically the same relations as those presented in Figure 2 were observed for all other apparatus settings. The dynamics of changes was different, but in all cases the values of the medians stabilized at 10^{th}-18^{th} measurement replication. One should remember that the measurement time in the present study was equal to one minute (at applied earlier number of simple counts on the detectors). In this case, stabilisation of measurement conditions in the successive replications was achieved after 10 to 20 minutes. Thus, for the needs of further analysis it was assumed that the results obtained for the 20^{th} replication were stable and representative. However, for a different time of single measurement (different number of simple counts on the detectors) this time might be different and therefore the stabilisation time of measurement results should be determined separately for every case.

The absolute difference between the deciles of the first and the 20th measurement was adopted as a measure of particle size change during the time of a series of measurements.

Data presented in Table 3 show that decay dynamics reflected by the difference for the successive settings, practically for all cases (except the difference for decile d(0.1) for the Hydro MU unit), was the highest for the lowest value of stirring intensity. That result, at cursory analysis, seems to be surprising because one might expect that the higher the intensity of stirring the higher flock fragmentation is achieved. However, the least differences obtained at the most intensive stirring and pumping between decile values for the first and the 20th replication might be caused by high intensity of flock break-up during the first measurement, at the beginning of the experiment. Regardless of the reasons causing changes in the differences described in Table 3 it seems that in the case of suspension stirring intensity, pump rotor of 1300÷1500 rpm is the most suitable choice.

Table 3

Absolute values of differences between decile value of the first and the twentieth measurement for different settings of pumping intensity (different rotor speeds)

Rotor speed [rpm]	Hydro G			Rotor and stirrer speed [rpm]	Hydro MU		
	$\lvert Rd(0.1) \rvert^*$	$\lvert Rd(0.5) \rvert^*$	$\lvert Rd(0.9) \rvert^*$		$\lvert Rd(0.1) \rvert^*$	$\lvert Rd(0.5) \rvert^*$	$\lvert Rd(0.9) \rvert^*$
1000	7.4	17.1	25.1	1200	6.3	13.4	18.7
1300	5.3	9.1	20.0	1500	6.7	13.3	13.7
1600	5.4	10.5	15.7	1800	6.0	11.6	13.3
1900	4.3	6.9	10.6	2100	5.5	10.2	14.6
2200	5.0	10.0	20.9	2400	5.9	10.9	16.8
Mean value	5.49	10.72	14.48		6.08	11.89	15.41
Standard deviation	1.16	3.84	9.73		0.42	1.43	2.27
Coefficient of variation *k*	21.15	35.77	67.24		6.85	12.03	14.73

* Absolute values of differences between deciles values of the first and the twentieth measurement

Considering the mean values for particular deciles, statistically there was no difference ($P = 0.05$) between values obtained with the two unit used. So, it may be concluded that the stirrer operation did not influence the flock decay. However, analysis of coefficients of variability for both units (Table 3) shows that significantly lower values were obtained for the Hydro MU (integrated operation of pomp and stirrer). The differences in the design and in the possibility of separate rotor and stirrer speed control for both units lead to the conclusion that better values (less variable) for the evaluation of flock size were obtained with the use of Hydro MU.

Comparison of separate decile values for the 20th replication in relation to pump rotor speed may be assumed as an alternative assessment manner of the effect of stirring intensity on flock diameters. Examples of results for median changes (d(0.5)) are presented in Figure 3 and values for the rest of the analysed deciles are presented in Table 4.

The Hydro MU and Hydro G units have different design and different settings scope [49]. This results in different lowest rotational velocities of pump rotor causing movement of flocks in the measurement unit. So, it was decided that not the same speed settings but an identical step in rotor speed increments between successive measurements, in relation to the minimal values, was the point of reference in our research. The minimal value of rotational velocity setting was 1200 and 1000 rpm for Hydro MU and Hydro G, respectively.

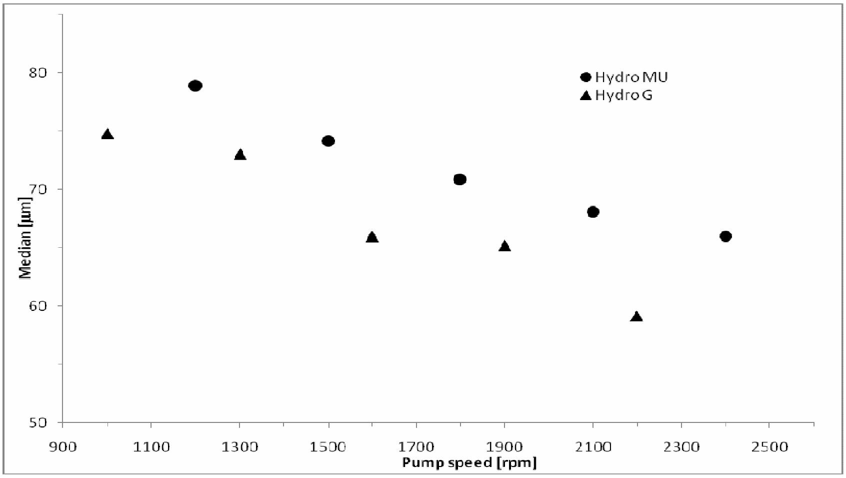

Fig. 3. Change in the dynamics of distribution medians for flock size during the 20th replication in dependence on the mixing intensity. Explanations: the mixing intensity was presented in *revolutions per minute* (rpm). In the Hydro MU unit the pump was integrated with the stirrer, so the presented rotational speed values relate also to the speed of stirring. In the case of the Hydro G the stirrer was switched off

Table 4

Decile values for both units, obtained at different settings of stirring and pumping intensity of suspension through the measurement device. Values achieved for the 20th measurement/replication

Rotor speed [rpm]	Hydro G			Hydro MU			
	d **(0.1)**	***d*** **(0.5)**	***d*** **(0.9)**	**Rotor and stirrer speed [rpm]**	***d*** **(0.1)**	***d*** **(0.5)**	***d*** **(0.9)**
1000	24.1	74.9	201.1	1200	24.8	78.9	190.5
1300	21.5	73.0	201.1	1500	22.2	74.1	184.5
1600	18.3	66.0	179.0	1800	20.6	70.8	181.4
1900	16.8	65.2	180.8	2100	18.9	68.0	174.9
2200	14.6	59.2	164.4	2400	17.7	66.0	172.2

Data presented in Table 4 show that the values of diameter deciles decreased linearly along with increase in the stirring intensity for the consecutive settings. Practically in all the cases (apart from one case) the values of determination coefficient for the analysed curves exceeded the value of 0.9.

Effects of ultrasounds on flock sizes

The distinct decrease in the values of flock diameter medians to 62.55÷61.65 µm at 1200 rpm and to 52.32÷46.55 µm at 2400 rpm was caused by treating the samples with ultrasounds for 4 minutes before the experiment.

It appears that the conditions under which the measurements were conducted influenced the results obtained. The measured structures were prone to mechanical fragmentation. The application of ultrasounds clearly breaks up the flocks of activated sludge. Similarly, the pumping and stirring speeds had an influence on the flock diameters; the median values decreased along with increase in those speeds. However, it is not precluded that the stirrer speed had a positive effect on the sludge flocculation - the second series, with the stirrer turned off was characterized by lower values of the median.

Summary and conclusions

Based on the conducted experiment with the implementation of activated sludge particle diameters measurement using laser diffraction method it is possible to make the following conclusions:

- Sample holding for 3 hours before the measurements did not influence the measured values of flock diameters.
- Flocks of activated sludge are prone to fragmentation caused by the applied method of measurement.
- The measurement results of activated sludge flocks obtained with the laser diffraction method were dependent on the pump rotor and stirrer speed to a high degree. Flocks of larger diameters were fragmented more intensively than smaller ones.
- Median of measured diameters decreased along with increase of rotor speed as well as with the time of measurement - stabilisation of measurement results was observed after 10÷15 minutes from the start of the experiment.
- The application of the Hydro MU units to the Mastersizer 2000 apparatus resulted in more stable results than in the case of the Hydro G application for which measurements were characterised by higher dispersion.
- Ultrasound treatment of samples before measurements with the laser diffraction method significantly affected flock sizes and result stability - the results were stable from the first measurements.

Based on the conclusions presented above, the laser diffraction method can be applied for measurements of activated sludge flock diameters under the following conditions:

- The size of activated sludge particles measured with the laser diffraction method is not a real value but after standardisation of measurements results may be treated as a parameter describing the examined sample of sludge.
- The diameters of activated sludge particles should be stabilised before the measurements.

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POMIARY ŚREDNICY CZĄSTEK OSADU CZYNNEGO ZA POMOCĄ METODY DYFRAKCJI LASEROWEJ

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Abstrakt: Praca dotyczy oceny możliwości wykorzystania dyfrakcji laserowej do badania rozmiarów kłaczków osadu czynnego. W czasie pomiarów zaobserwowano podatność analizowanych cząstek na zniszczenie mechaniczne zależną od zaprogramowanej intensywności mieszania (mieszanie i przepompowywanie jest wymagane w celu wymuszenia przepływu analizowanych zawiesin przez układ pomiarowy). Na podstawie wniosków opracowanych na podstawie prowadzonych badań można stwierdzić, iż metoda dyfrakcji laserowej może być stosowana do pomiarów rozmiarów kłaczków osadu czynnego pod następującymi warunkami: rozmiary kłaczków oraz pozostałych elementów osadu czynnego uzyskane w czasie pomiarów nie mogą być traktowane jako wartość bezpośrednia opisująca rozmiary kłaczka, lecz po standaryzacji warunków pomiarowych może być traktowana jako ilościowy parametr opisujący właściwości osadu czynnego. Przed pomiarem metodą dyfrakcji laserowej osad czynny powinien być uprzednio stabilizowany na przykład za pomocą mieszania w układzie pomiarowym bądź też za pomocą ultradźwięków.

Słowa kluczowe: metoda dyfrakcji laserowej, średnica cząstek osadu czynnego