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Flocs' morphology and stability in suspensions obtained in water treatment process

M. Banaś*
Department of Power Systems and Environmental Protection Devices,
AGH University of Science and Technology, Al. Mickiewicza 30, 30-059 Kraków, Poland
* Corresponding e-mail address: mbanas@agh.edu.pl

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Properties

ABSTRACT

Purpose: In this paper, an attempt was made to apply fractal geometry for the description of morphology of the particles found in the selected crude and coagulated suspensions derived from the water conditioning process. When morphology of such suspensions was determined, particular attention was paid to their stability and influence of hydrodynamic conditions on both morphology and stability of their particles.
Design/methodology/approach: Particle composition was examined using Low Angle Laser Light Scattering method and presented in volumetric particle size and parameters of log-normal distribution. Particles' shape and morphology was characterized by fractal dimension by means of light scattering method.
Findings: Fractal geometry is powerful tool for characterizing of non-grain suspension. Fractal dimension is very sensitive factor in terms to hydrodynamic and analytic conditions.
Practical implications: Determining the nature and properties of the suspensions treated undergoing coagulation and sedimentation process in settlers
Originality/value: Application of the fractal dimension to design and use of the sedimentation devices in order to characterize the properties of solid phase of suspensions.
Keywords: Particle composition of suspension; Water treatment; Fractal dimensions of floculated suspension; Sedimentation

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1. Introduction

The principal task of the municipal administration is to supply both individual consumers and institutions with good quality water. There are two main sources of drinking water: ground water and surface water. Ground water is extracted from an aquifer by the use of various types of wells or is taken directly from springs also. Surface water mostly originate from rainfalls and flowing rivers, but to some extent also from springs. Such water is collected directly from rivers or from the water-retaining dams.

In order to ensure water quality, regardless of the source, water have to be properly treated (conditioned to use as drinking water) in agreement with numerous regulations [1] or requirements given in an adequate legislative act. In Poland, this issue is within the competence of the Minister of Health. The Regulation of the Minister of Health with amendments to this act of April 20th, 2010 is a current legislative act in this subject [2]. However, sanitary requirements are as much important as practical aspects since water quality should not be a reason of any technical problems in the water distribution system like, for example, plugging of pipes.

Assuming that ground water causes less problems due to its infiltration (although, iron and manganese content must be adjusted); water taken from the surface intakes may contain far more biological, chemical and mechanical pollutants (solid colloidal and semi-colloidal phase). The presence of the fine-grain phase in water is expressed by higher both turbidimetric and nephelometric turbidity. Such a phase, which is responsible for a permanent increase in turbidity, is characterized by a great degree of granulation and contains particles of a size measured in individual micrometers, or in several tens or even hundreds of nanometers. These particles form a stable colloid phase, which is not settling.

The removal (reduction) of the phase, which is responsible for water turbidity, is necessary not only due to aesthetic reasons but also because of the fact that the surface of its particles is an excellent medium for proliferation of many microbes showing potentially (or real) harmful effects on human health.

The solid phase, which is mostly detach from the land surface by rainfalls, contains the minerals characteristic for the rivers' basin. These are chiefly different types of clays, silt, loesses and humus materials.

There is some number of techniques applied in the phase separation, which may be used to remove mechanical colloid pollutants, of which the core are coagulation and flocculation. These operations lead to the formation in their disperse phase of aggregates of colloid particles (primary particles) called fluffs or flocs. Such suspensions are then separated by means of the sedimentation; however, it is relatively slow process, which additionally requires bulky rectangular settling tanks, along with great build area. In this context, the application of the multiflux sedimentation is a very attractive method to support a classical process, since in this method settling tanks require lower build area due to the multiflux nature of their filling.

However, in order to design and exploit the devices for phases' separation by sedimentation (especially those multiflux) it is firstly necessary to gain a good knowledge of physicochemical properties of the settling suspension [3-6]. In this context, granulometic characteristics is principal since informs not only about quantities and size of particles in the suspension but also about their shape (morphology) or even their volumetric distribution [7].

Distribution of a statistical random variable is the most convenient method to characterize grain composition in the suspension, were grain composition is expressed as a distribution parameter. For this purpose, the log-normal distribution is the most popular; however, another distributions are also frequently used like the Rosin-Rammler cumulative distribution or the distribution based on Stacy's generalized gamma; the latter very interesting [8].

Particle shape, if deviates from spherical, is commonly determined by form factors in two ways: qualitatively - by classifying of particles into several groups of characteristic shape (e.g. in agreement with the BS 2955:1993 [9]; or quantitatively - by indicators of elongation, indicators of their oblate shape or by a Brockmann's dynamic form factor.

Non-spherical shape significantly complicates the description of the particle size since it is difficult to distinguish one geometric dimension describing the particle size. Therefore, instead of a grain diameter, an equivalent diameter is used, defined as a diameter of an equivalent sphere (or an equivalent circle illustrating the projected area of a particle), whose selected geometric features and behavior are identical with the discussed grain. Jarvis, in his paper, gives several definitions of the equivalent particle dimension [10].

In non-grainy suspensions, their particles formed due to coagulation (aggregation) of fine particles of the size of r_0 (so called primary particles) are 2-3 times higher in order of magnitude. Their complex structure causes that their size is usually expressed as the radius of gyration R_g, which describes the distribution of mass (of primary particles) around their gravity center.

The use of an equivalent diameter does not provide an opportunity to define precisely morphology of particles, their relative density, a degree of fulfilling of the floc's space or its hydrodynamics properties. Thus, the fractal geometry is more suitable than the Euclidean geometry to describe irregular particles, which often had an expanded three-dimensional structure. The fractal geometry was discovered and developed by Mandelbrot [11] and then used to describe fine particles by Lin [12]. This geometry gives the chance to characterize expanded, self-similar forms showing either three-dimensional, planar, or linear shape, or even loosely connected aggregates exhibiting some degree of arrangement recurring in a few scales of magnitude.

Fractal dimension is a crucial value describing a fractal object such as, for example, suspension particles. It is a proportionality coefficient in the power low, which describes various characteristic features of the particle geometry, number of particles, the length of a perimeter, their area or volume [13-15].

In suspension, their particles may be treated as n-dimensional mass fractal objects, were n is 3 or less frequently 2. The value of n-dimensional fractal dimension informs how the fractal object fulfils its n-dimensional space. N-dimensional fractal object takes a non-integer value, lower than n, that is the core of fractal geometry.

Fractal dimension of particles in suspension can be measured by means of various analytical techniques, for example, by a direct measurements of their geometric values or physical values and properties resulting from the fractal scaling. Such methods include the image analysis; the analysis of the settlement rate of the granules of different bulk density or different linear dimensions; the analysis of turbidity fluctuation; or the methods basing on laser light scattering.

The image analysis, when applied to establish fractal dimension of the 3D particles, has many disadvantages and restrictions, of which 2D nature of the images examined is the core since the method can only be applied to the objects of a fractal dimension lower than 2. The image capture of the fractal particles relies upon the selection of the privileged projecting surface. This causes that, the fractal particles show privileged orientation towards the flow direction due to a flow (understand as a flow of the sample examined or settling the fractal particles). This, in turn, causes that their geometric dimensions are overestimated. This technique alone strongly interferes in the structure of the object investigated. Moreover, the method is tedious and time-consuming since individual particles are analyze. Additionally, the results obtained are low-representative.

In view of the above, the techniques that use scattering (of laser, neutrons, or X-rays) for the determination of particle fractal dimension are very convenient, fast and free of the

aforementioned restrictions. Of these, a small-angle laser light scattering method (SALLS) is popular for the analysis of suspension particles of micrometric sizes [16,17].

Interactions between electromagnetic radiation and matter are a complex phenomenon that has been described by the Maxwell's theory. In practice, however, to find how the matter scatters incoming radiation, the methods of Reyleigh-Gans-Debye, Mie, and Fraunhoffer are used, which are based upon generalized scattering theories but not directly Maxwell's theory as too generalized one with complicated equations.

In order to simplify the phenomenon of light scattering on the particles in suspension, the monochromactic laser scattering is broadly used. Small particles scatter light at high angles whilst large particles scatter at low angles.

The principal rule is that scattering takes place on an individual scattering object, regardless of the other. Therefore, the value of monochromatic radiation scattering angle indicates the size of scattering particle and is expressed as the scattered vector defined as the difference between the incident vector and the scattered vector. The value of the scattered vector amplitude that was measured in the medium with n coefficient of light refraction is defined as

$$|Q| = \frac{4\pi}{\lambda} n \sin(\frac{\Theta}{2}) \tag{1}$$

where:

Q - is the scattered vector;

λ - is the wavelength of the scattered light; for MS 2000E λ=633 nm;

n - is the coefficient of light refraction in dispergant (n=1.33 for water);

Θ -is the angle of beam diffraction.

Sometimes, the Q value, fundamental in describing the phenomenon of beam scattering on solid phase in suspensions, is defined as the wave number of the scattered vector and is adversely proportional to the size of scattering structures.

On the fractal-like structures formed from the smaller elements, the intensity of the scattered radiation depends upon both properties describing interaction between radiation and primary particles (so called "form factor - $P(Q)$); and the manner of 3D distribution of primary particles inside the fluffy structure (so called "structure factor - $S(Q)$)

$$I(Q) = I(0) \cdot P(Q) \cdot S(Q) \tag{2}$$

where:

Q -is the amplitude of the scattered vector;

I(0) - the intensity of radiation under zero angle;

P(Q) -is the form factor;

S(Q) - is the structure factor.

For low Q values, the $P(Q)$ value is nearly constant. On the other hand, the $S(Q)$ value is stable for high Q values. Hence, with respect to the various ranges of Q values intensity of scattering depends on both these factors considered separately. Therefore, when analyzing $I(Q)$ as a function of a Q (that means reversed dimensions of the scattering structures - given in a log scale), three ranges may be distinguished [16]:

- Guiner's - which refers to large structures, usually flocs (weakly correlated with the size of primary particles)
- Fractal - scattering by fractal aggregates,
- Porod's - scattering by primary particles of suspensions.

The fractal dimension of aggregates in the suspension examined may be easily found from the slope of the line plot within the fractal range. Fractal range exists for the Q: $1/R_g < Q < 1/r_0$, where bordering values are reciprocals of a primary particle radius and mean-square value of flocks radius of gyration.

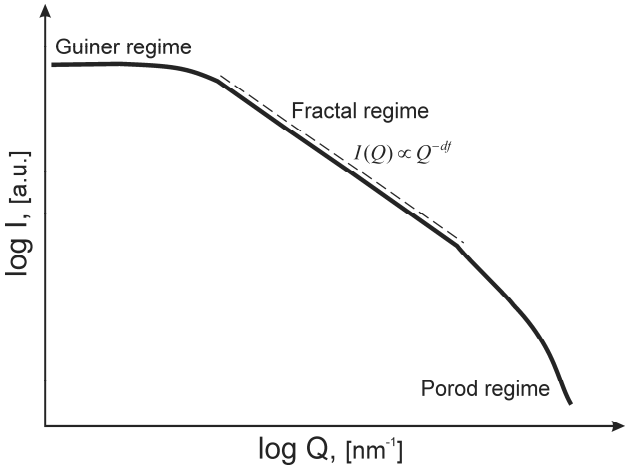

Fig. 1. Intensity of the scattered radiation as a function of Q

The methods used for light scattering, including a small-angle laser light scattering method (SALLS), make it easy to find fractal dimension of suspensions' particles in an non-invasive manner (without serious deformations arising e.g. from oblate projection during the images' analysis or from the privileged particle's orientation during the process of image capture), assuring *in situ* analyses without interference in the running process.

2. Laboratory experiments

2.1. Aim

In this research work an attempt was made to use fractal geometry to describe grain composition and particle morphology of suspensions' solid phase, analyzing both "crude" and coagulated suspensions occurring in the process of water conditioning. During granulometric measurements, particular attention was paid to the problem of stability of the aggregates formed by particles of the solid phase as well as to the influence of hydrodynamic conditions on their composition and morphology. The above was done taking into account the

application of sedimentation to water clarification during its purification.

2.2. Malvern MasterSizer MS 2000E

Grain analyses were conducted by means of a Malvern MasterSizer 2000E, which is used for evaluation of the floc size distribution within the range 0.1-1000 μm. The principle of this method is based on the physical phenomenon of multi-angle laser light scattering (MALLS). The MS2000E analyzer was equipped with a helium-neon laser at the wavelength λ=632.8 nm and a 44 radiation' sensors at the angles from 0.01 to 40.6 degrees, of which the sensors from 1 to 33 were small-angle ones. Depending on the model of disperger (the dispersing unit applied along with an adequate measuring cell), measurements might have been carried out in the gas phase as well as in liquids. The analyzer (equipped with the special dispersing unit for small volumes) allows also measurements of a very small volume of expensive or toxic liquids. In this experiment, a Hydro MU dispersing unit has been used - for the measurements in large volume liquids (up to 1 dm^3), additionally equipped with a rotor pump working at rotational speed within the range 800-4000 rpm. Moreover, it is also possible to uniform the sample by the use of an ultra-wave disinegrator combined with a disperger. In this experiment three pumps were used: a rotor pump integrated with a disperger; a peristaltic Ismatec MCP-process pump (connected both before and after the measuring cell of analyser); and a syringe pump. The detail analyser description is given in the producer's technical guide [18].

2.3. Experimental material

The experimental material was the clay suspension collected from the Raba catchment basin. Such clay is commonly found in the river basins, being a component of in-coming water in the Water Conditioning Plants. The presence of clay in water, particularly in the periods of larger rainfalls or snow thawing, leads to higher turbidity of the water undergoing later process of conditioning;. The turbidity removal, usually performed by coagulation combined with sedimentation and filtration, is therefore the fundamental technological operation in water purification.

From the material collected, a suspension was prepared by suspending 2 kg of fresh clay in 5 dm^3 of water from which larger fractions (mineral and biological) were removed by passing the material through a sieve with 100 μm holes. The raw suspension (coded as GTD) was a base for preparing the initial suspension (GTDB) by dispersing 50cm^3 of GTD up to 1dm^3.

The GTDB suspension was analyzed for the concentration of solid fraction by means of a filtering method. In this case, the detected value was S_0=12.29 g/dm^3. The GTDB suspension was the initial material for further investigations.

Sedimentation was investigated in terms of the density of solid fraction measured by a helium pycnometer (MicroMeritics - AccuPyc 1330). Measurements were carried at 27°C in five replications to avoid stochastic errors. The value determined for the solid phase was ρ=2650 kg/m^3 at 0.0012 kg/m^3 standard deviation.

3. Results and discussion

3.1. Granulation and morphology of raw suspension

At the beginning, the raw suspension has been analyzed, assuming that, in the water treatment processes, such suspension perfectly reflects composition of the in-coming river water.

The beaker of a dispersion unit was filled with 990 cm^3 of demineralized and degassed water and 10 cm^3 of the sample containing GTDB; the suspension achieved had 11.24% obscuration (weakening of the laser beam passage). In such a sample, the concentration of solid phase was S_r=0.1366 g/dm^3 and turbidity 130 NTU (measured by a PC-Compact turbidimeter). The sample, uniformly dispersed in a beaker of a dispersion unit, was then delivered to a MasterSizer2000E instrument, using an original Hydro MU dispersion unit equipped with a rotor pump, working at rotational speed of 2000 rpm. Measurements were done in 25 replications and finally a mean value was defined to minimize stochastic errors.

Figure 2 illustrates the result obtained as a volumetric distribution of particle size in raw suspension. The specific values determined for the particle characteristics were: d_{50}=16.27 μm; d_{10}=2.93 μm; and d_{90} =45.05 μm.

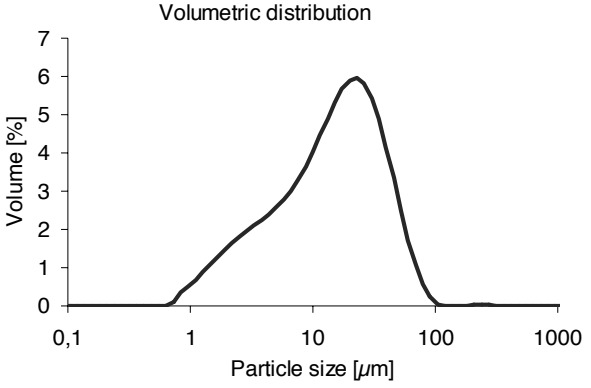

Fig. 2. Characteristics of the raw sample particles

In order to obtain the results comparable to the literature, the assumption has been made that the actual particle size distribution is similar in shape to the log-normal distribution [12], so the parameters were also established for such a description. The results of these measurements, expressed as the cumulative distribution function ("size below") were then calculated by means of the Author's programme (based on a regression method) in order to establish the log-norm distribution parameters.

These were: m=2.44 (11.4 μm for medium grain size); σ=0.93; and R=0.991 (regression coefficient).

For the characteristics of the particle morphology in suspension, its fractal dimension was also found The intensity of scattered radiation, recorded on the subsequent 44 sensors of the MS2000E analyser, was expressed as:

$$\log I(Q) = a \cdot \log Q + b \tag{3}$$

The wavenumber of the scattered vector was calculated using tabularized values provided by a producer of the analyzer (Malvern 2007). Basing on this plot, the established fractal dimension was d_f=1.59±0.03 with R^2=0.995 (Fig. 3).

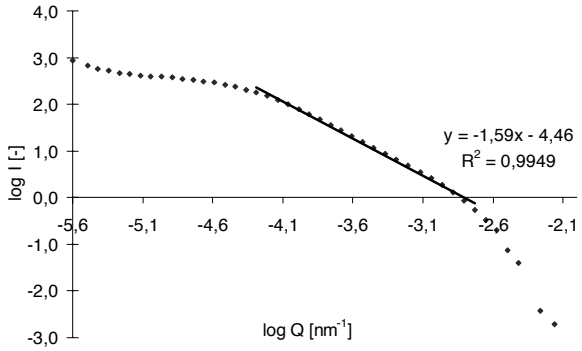

Fig. 3. Determination of the fractal dimension of the raw sample

The information obtained on the particle structure of the raw suspension suggest that its particles are already composed not from the primary particles but from the aggregates formed by a larger number of particles that, in turn, indicates on their complex inner structure. Hence, to prove the above, the raw suspension having undergone ultrasonic treatment of the defined exposure time on the suspension's particles, was examined in terms of its particle characterization. Particle characteristics of the suspension examined was conducted in fifteen successive series according to the following cycle: a 2-minute exposure to ultrasounds, then a measurement of particle composition.

To simplify the analysis, the results achieved were transferred into the parameters of log-normal particle size distribution and had been presented as the relationship between the parameters of such distribution and total exposure to ultrasounds (Fig. 4).

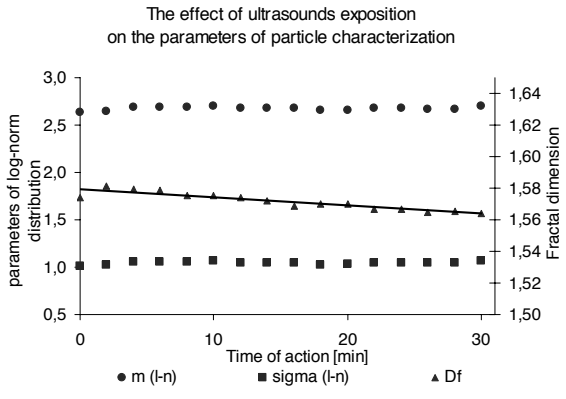

Fig. 4. The effect of ultrasounds on particle composition in the raw sample

Figure 4 indicates that even 30-minute exposure to ultrasound do not lead to the geometrical changes in the particle size. However, a slight decline in fractal dimension was observed due to the disruption of the internal structure of compact aggregates; although, such structure rearrangement was only slight, with the changes in fractal dimension being of 1% initial value in the examined time of exposure to ultrasound.

3.2. Coagulated sample - sample delivery by a rotor pump with a rotational speed of 1200 rpm

The subsequent analyses were done for the coagulated suspension delivered to the analyzer by the use of various dispersion units.

For the coagulation process, the PAX 16 coagulant has been selected as the one usually used in the municipal water treatment since its application does not have a detrimental effect on the water sensory indicators, for example its colour. Basing on the producer's recommendation, a coagulant portion of 80 ppm has been established for the initial sample of turbidity 130NTU.

The beaker of a dispersion unit was filled with 980 cm^3 of demineralized and degassed water, 10 cm^3 of the uniformly dispersed GTDB suspension, and 8 cm^3 of the coagulant in a concentration of 10 g/dm^3.

The sample was stirred for 1 min with a rotor stirrer at 2000 rpm. Afterwards, when the speed was reduced to the minimal value of 1200 rpm, which still enabled the sample to be pumped into the analyzer, the measurements were done in 46 replications, averaging the results recorded to avoid stochastic errors.

Figure 5 shows volumetric distribution of the particle size.

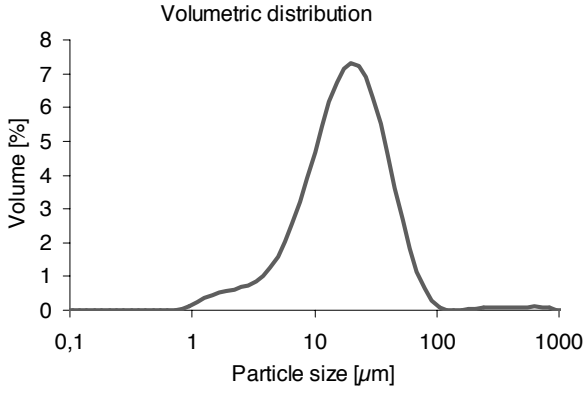

Fig. 5. Particle characteristics of the coagulated sample - stirring, a rotor pump of 1200 rpm

The typical values for the particle characterization were d_{50}=19.61 μm; d_{10}=6.17 μm; d_{90}=47.69 μm. The results obtained were exported to the text file as the cumulative distribution. Then, the following values of parameters for the log-normal distribution of particle size were established: m=2.91, σ=1.029, with R=0.984 (the mean particle size of 18.37 μm).

Identically as it was done for a raw sample, the fractal dimension was found from the slope of the line plot defined as in point 3.1. The fractal dimension obtained was d_f=1.82±0.02, R^2=0.997 (Fig. 6).

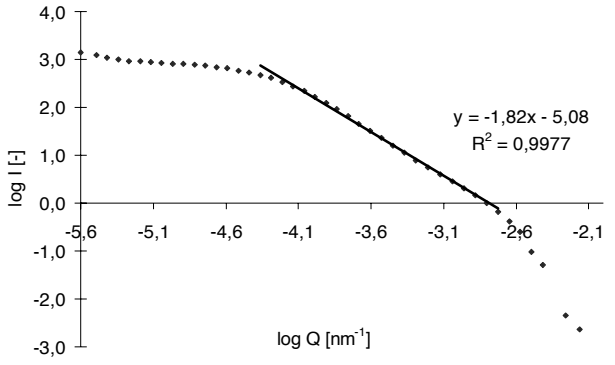

Fig. 6. Determination of the fractal dimension for the coagulated sample - stirring, a rotor pump at 1200 rpm

3.3. Coagulated sample - sample delivery through a peristaltic pump

The rotary pump employed previously has relatively high rotation that may disrupt the forming flocs. Therefore, this time, for the identical coagulation conditions, measurements were carried out using a peristaltic pump as a device transporting suspension to a measuring cell of an analyzer. The suspension was prepared according to the point no 2 (see above). After quick stirring at 2000 rpm for 1 min., a stirrer was switched off and a measurement started by pumping the flocculated suspension to the analyzer by means of a peristaltic pump at rotational speed of 50 rpm. The result is expressed as a mean value of the 20 measurements. The dependence between the particle size distribution and volume illustrates Figure 7 presents volumetric distribution of particles.

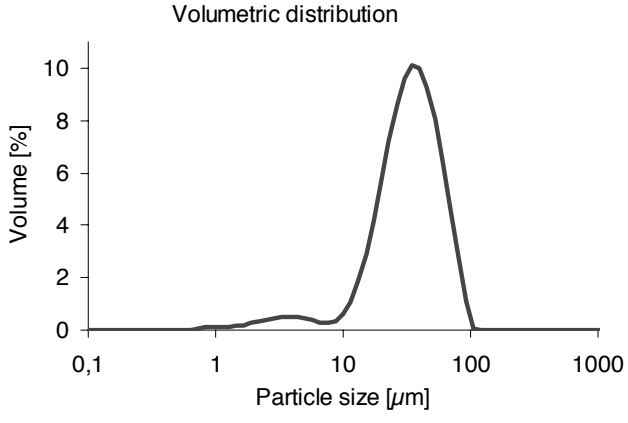

Fig. 7. Particle characteristics of the coagulated sample - pressing by peristaltic pump

The characteristic values are as follows: d_{50}=36.02 μm; d_{10}=15.95 μm; d_{90}=67.42 μm. The averaged result of the measurements was a base for determining the parameters of log-normal particle size distribution. These were: m=3.44, σ=1.078, R=0.952 (the mean value of particle size: 31.19 μm).

As it was earlier done, the fractal dimension of the flocs formed under such coagulation and measurement conditions has been determined. The fractal dimension was d_f=2.01±0.02 with R^2=0.996 (Fig. 8).

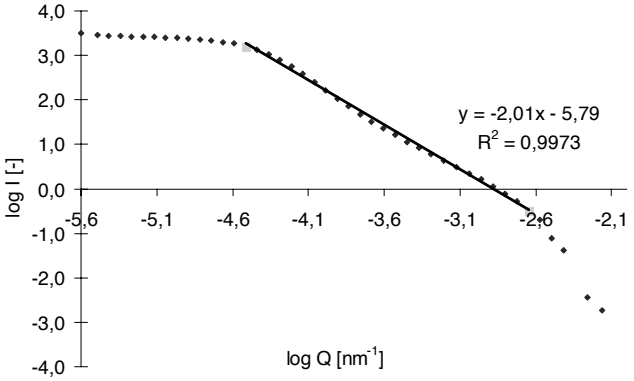

Fig. 8. Establishing of fractal dimension for the coagulated sample - pressing by peristaltic pump

3.4. Coagulated sample - sample delivery through a syringe pump

It has been observed that during the transportation a sample of the coagulated suspension into the analyser by a peristaltic pump, a regular intensive pulsating pressure changes were noted in the suspension due to irregular work of such a pump that in turn could lead to the disruption of the forming flocs. Hence, measurements were made using a medical syringe of 100 cm^3 volume as a sample delivering device. A sample of the suspension, prepared in agreement with the procedure aforementioned, was stirred quickly and then pressed to the measuring cell of the analyser by means of the syringe. Next, the measurements were conducted, during which the suspension was not pressed and therefore its particles could freely settle. For this reason, a series of 25 successive measurements has been done, finally averaging the result.

Figure 9 shows the volumetric distribution of particle size.

In this case, the characteristic particle values were: d_{50}=79.1 μm; d_{10}=27.62 μm; and d_{90}=162.54 μm. The results from volumetric characteristics of particles have been transferred into the values of parameters of log-normal particle size distribution. The values obtained were: m=4.19, σ=1.09, with R=0.961 (the mean particle size: 66.02 μm).

The flocs, formed in the aforementioned conditions of coagulation and the mechanism of sample delivery into the analyzer, had fractal dimension of d_f=2.09±0.03, with R^2=0.997 (Fig. 10).

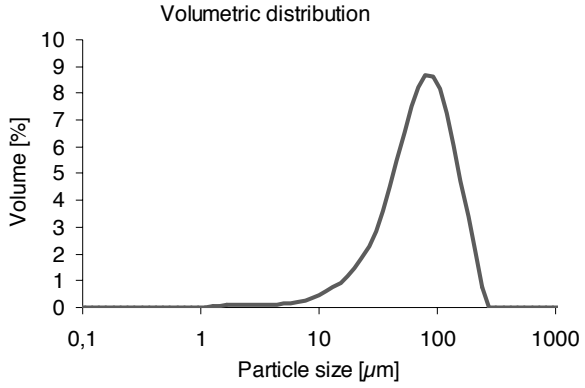

Fig. 9. Particle characteristics of the coagulated sample - sample delivery with a syringe

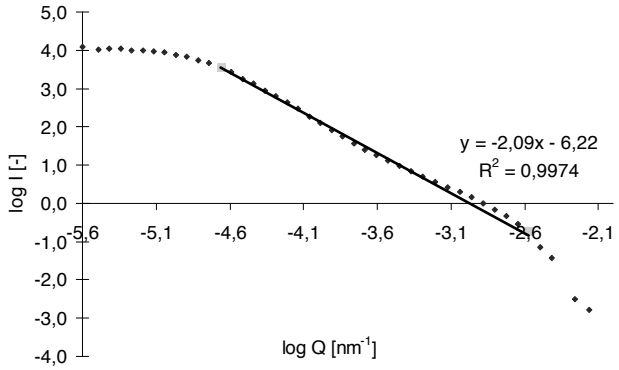

Fig. 10. Establishing of fractal dimension for the coagulated sample - delivery with a syringe

3.5. Coagulated sample - peristaltic pump, suction

In all previously described mechanisms of the suspension delivery to the analyser, the suspension examined was pumped to the measuring cell. The flocs of the suspension flowing through the dispersion unit were disrupted by its working part, by the blades of the rotor pump, pumping cylinders of the peristaltic pump, or finally by a piston of a syringe. Therefore, in order to minimize the influence of the working section of a dispersion unit, rearrangement has been done in the location of this unit against a measuring cell of an analyser. The latter has been put in front of the pump. As a result, prior to the contact with the working section of a pump, flocs were flowing through a measuring cell to the suction section of the pump. In this experiment, the suspension was transported by a peristaltic pump.

A sample was prepared in accordance with the previously described procedure. After coagulation of the suspension followed by quick stirring, the sample was sucked into the analyser by means of a peristaltic pump. The measurement were carried out in eighteen replications and the results were finally averaged.

Figure 11 illustrates the volumetric distribution of particle size in the flocculated suspension.

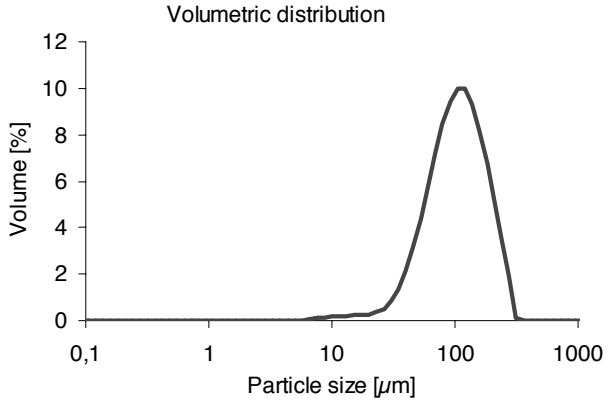

Fig. 11. Particle characteristics of the coagulated sample - suction with peristaltic pump

The characteristic values of the particle characteristics are as follows: d_{50}=112.07 μm; d_{10}=52.81 μm; d_{90}=211.8 μm.

Parameters of the log-normal particle size distribution were estimated basing upon the averaged results of the measurements and were: m=4.64, σ=0.754, and R=0.977 (the mean grain size: 103.54 μm).

The fractal dimension determined for the above conditions of coagulation and dispersion was d_f=2.50±0.03, with R^2=0.997 (Fig. 12).

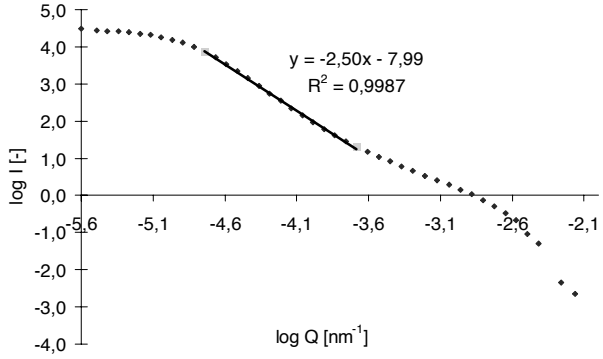

Fig. 12. Establishing of fractal dimension for the coagulated sample - suction with peristaltic pump

4. Conclusions

The results obtained lead to the following conclusions:

- The examined suspension (the exemplary raw material in the Water Conditioning Plants) is a suspension of very fine grains. Its fraction, of a size below 16 μm, is half the volumetric share of particles.
- Particles forming such a suspension (and their possible conglomerates) are relatively stable: the suspension does not

show significant changes in their granulometric characteristics due to ultrasonic treatment, even during a long exposure to ultrasounds.

- Particles of the raw suspension show the expanded structure of a fractal dimension 1.59 that indicate the domination of chain-alike structures.
- Long-lasting ultrasonic treatment of the raw suspension leads to a slight decrease in the fractal dimension, probably due to breaking the chain-like structures of such suspension.
- Coagulation of this suspension with polyvinyl chloride (PAX 16) has a positive effect; the d_{50} indicator visibly increases from 16μm up to even 112 μm that is accompanied by a distinct decrease of fine particles (flat plot of the density curve in the part referring to fine particles).
- The suspension, which has been coagulated, exhibits a clear fractal structure - its fractal dimension is markedly higher compared to the raw suspension.
- Coagulation of this suspension leads to the formation of unstable flocs, which can be easily disrupted due to hydro-dynamic power like, for example, stirring.
- Different hydro-dynamic conditions found in a coagulator, a dispersing unit and in a measuring cell lead to the formation of flocs of various sizes and different structures that describes fractal dimension varying from 1.82 to 1.99, 2.02, or even 2.42.
- The choice of a stirring method and device (necessary for the sample averaging but also to prevent the process of sedimentation during the measurement) is a key factor for size and shape of the flocs obtained. A rotary pump, applied in an original dispersion unit of a diffractometer (MS2000E), is therefore not useful for this purpose. The application of peristaltic and syringe pumps gave as a result the flocs of a mutually similar structure. Therefore, in the experiments on particle characterization of the aforementioned suspensions, this type of pumps (peristaltic or syringe) may be applied interchangeably.

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