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Determination of density and classification of post-coagulation sediment

Abstract

The coagulation process is one of the water and wastewater treatment processes. It results in the precipitation of sediment. After the coagulation process, the density of the sediment has a significant impact on the sedimentation rate of particles. These particles have different shapes, sizes and structures, and they settle in a different way. The particle density has an impact on the settling rate. The structure of particles results in two types of pores. Micropores are characteristic features of the sediment, but macropores should not be considered while determining the density. In this paper sediment samples were prepared and the density was determined using the gravimetric method by means of the press. The number of press strokes should not destroy the microstructure of sediment. The authors determined the degree of sample compression to minimize errors while determining the density. The volume of samples was obtained using X-ray tomography and image processing. The correct determination of the sample volume depends on the correct number of the threshold. Image processing was performed using LabVIEW software and Vision module. The paper presents the statistic classification of post-coagulation particles and the results of tests.

Keywords: sedimentation, coagulation, sediment density.

1. Introduction

The need for drinking water of high quality is increasing as the non-polluted water sources are continuously decreasing. Coagulation is a process commonly used in the treatment of surface water, groundwater and wastewater. In general, coagulation removes dirt and other particles suspended in water and colloids. Alum and other chemicals are added to water to form tiny, sticky particles called ‘flocs’, which attract dirt particles. Under the increasing weight of flocs, they become heavy enough to sink to the bottom during the sedimentation process. Aluminum sulfate (alum), ferrous sulfate, ferric chloride and ferric chlorosulfate are commonly used as coagulants [1, 2]. 32% of Asia, 75% of Europe, 29% of South America, 51% of North America, and 15% of Australia use groundwater for drinking. Iron concentrations in 87% of groundwater resources exceed the permissible rate of hygiene [3, 4]. Iron compounds lead to the precipitation in technical devices, which often results in the reduction of their efficiency. Trivalent forms of iron present in water increases turbidity and give it an unpleasant metallic smell, taste and irony brownish-yellow color. Such deposits can result in the growth of ferruginous bacteria, which in turn can result in the corrosion of equipment.

Iron compounds usually occur in the form of colloidal solutions. The removal of this type of pollution is possible by means of the coagulation process. The colloidal particles have electrical charges, which have to be neutralized in order to destabilize them, and combine them into larger clusters (agglomerates). Depending on the type of the medium, the dose and the type of the coagulant, and the physical and chemical conditions of the process, flocs have different structures and ability to agglomerate. Flocs formed after the coagulation process have different structures and, as a result, they are characterized by different sensitivity to sedimentation and filtration [5].

Depending on the nature and number of particles, the following types of settling are distinguished: settling of granular particles and settling of floc particles. Due to hydraulic conditions, we distinguish free settling, hindered settling and zone settling. Free settling occurs when the number of particles is small. In such a case, each particle settles separately without affecting adjacent particles or changing physical properties. It usually occurs in case of granular particles. Hindered settling takes place in case of significant concentrations of particles. Such a phenomenon most frequently occurs during the settling of post-coagulation suspensions in case of highly concentrated floc suspension. During this type of settling, vortices are created that eject smaller particles upwards. Zone settling occurs when the content of floc suspension reaches a limit value or the concentration of the non-floc suspension is very high. Then, apart from the sedimentation process, the consolidation (densification) of the sediment takes place at the bottom of the settling tank.

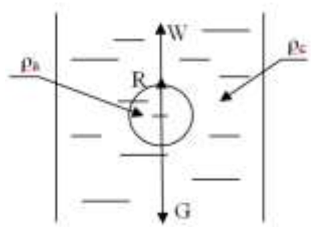

Fig. 1. Sedimentation of particles in the gravity field [6]

During the sedimentation process, flocs form larger agglomerates - this is called flocculation. The density of the sediment is similar to the density of water, which is the result of a very high mass fraction of water in the sediment usually amounting to 95-99%. In case of gravitational settling, the following forces affect the particles: buoyant force *W*, gravity force *G* and drag force *R*, and stop the settling of particles. In addition, there are forces related to the rotation of particles around their own axis and the intermolecular displacement. The gravity force is closely related to the density of settling particles ρ_a and liquid density ρ_c (Fig. 1). This parameter is important for the sedimentation process. Therefore, an attempt was made to form the density pattern of post-coagulation sediment particles based on the study carried out using X-ray tomography.

The general formula for settling velocity is presented below.

$$v_\infty = \sqrt{\frac{4}{3}\frac{gd(\rho_r - \rho_c)}{\rho_c c_x}} \quad (1)$$

$\rho_r -$ particle density, $\rho_C -$ fluid density, $c_x -$ drag coefficient

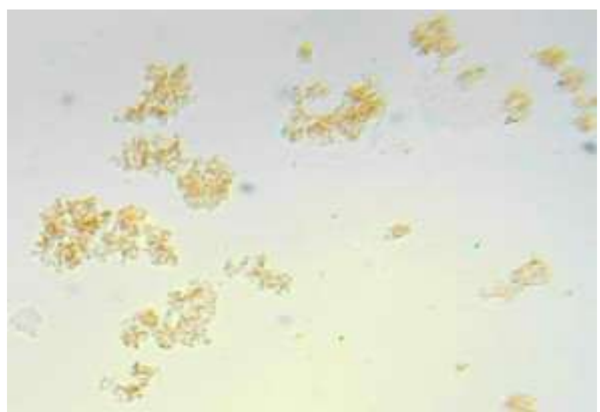

Fig. 2. Various form of the post-coagulation sediment

Due to the fact that the resistance coefficient c_x depends on both the shape and the density of particles, there are some difficulties in determining it. The determination of this coefficient is significantly hindered due to different forms of flocs as a result of the coagulation process (Fig. 2). Sediment structures formed during this process are diverse in terms of their shapes and degree of density. Taking the above into consideration, it can be observed that the modeling of this process is extremely difficult. It is possible to group flocs, taking their characteristics into account, and then formulate general equations describing the movement of particles belonging to a given group. However, to properly describe the settling velocity of particles, it is required to know the density of the material of which a floc is composed. This material has a closed microporous structure, which is a characteristic feature of a given sediment.

This paper presents the classification of flocs. It was carried out in order to determine the statistical resistance coefficient value for particular groups of particles, which will allow for the modeling of a complex sediment suspension.

2. Classification of post-coagulation sediment

The settling of the post-coagulation sediment is disturbed. Figure 2 clearly shows a very irregular form of flocs. Water-filled spaces (micro and macropores) make it difficult to determine the volume and the density of particles. Each particle of the sediment contains micropores which are a characteristic feature of every material and macropores which should not be taken into account in the determination of the density. Due to the above-mentioned reasons, the determination of the density for the post-coagulation sediment is not easy. The theoretical description of the actual movement of polydisperse suspension is extremely difficult. Attempts were made to describe the movement of particles based on the motion of a single particle or a regular arrangement of particles. The results were not relevant to the real path of sinking particles [7].

The classification of flocs formed from the post-coagulation sediment was carried out for the purposes of the research. Due to technological reasons, the settling time of the sediment is the most important parameter. Therefore, the classification proposed by the authors mainly focuses on the separation of groups of flocs according to their characteristic features affecting the settling velocity.

While examining the images, the authors decided to classify individual particles statistically. Flocs were divided into three characteristic groups. The classification takes into account the structure of a floc in terms of mass concentration (presence of macropores) and its shape. Flocs were divided into: compact particles, particles having a distinct mass concentration nucleus around which sediment crystallization networks are formed (flocks without mass concentration) and particles having a porous sponge structure (porous conglomerates).

The classification does not take into account all the characteristics. In the future, it will be used for equations describing the settling process. According to it, only the most important parameters of particles were considered. The occurrence and the size of pores in a single floc were assumed to be the criterion of division.

Fig. 3. Compact structure of flocs

Therefore, the flocs presented above (Fig. 3) are flocs with compact structure containing a large number of pores. These particles are characterized by the compact structure and relatively regular shape of a floc. Pores occurring in it are a natural feature of this type of suspensions. Settling of such particles can be described using the Stokes' law [7]. Due to various shapes, it is necessary to determine an appropriate resistance coefficient experimentally.

The second group consists of particles that have nucleus with concentrated mass surrounded by a number of sediment crystallization arms (Fig. 4). In case of such particles, the weight of a nucleus mainly affects the settling velocity. However, irregular crystallization arms change the direction in which particles settle. A non-linear trajectory significantly extends both settling path and time. The mathematical description of such particles requires much more complex mathematical models.

Fig. 4. Flocks without mass concentration

The third group includes porous conglomerates consisting of the types of particles present in a given suspension, combined into clusters. Their structure resembles a sponge, they are very unstable and they can easily change their shape while settling. Fig. 5 presents the mass that is not concentrated in one place. The structure developed a porous structure (like a sponge). In addition, it was observed that the porous structure of flocs enables them to combine into larger conglomerates. According to the phenomenon of falling conglomerates, this type of structure is classified as a separate group in the classification of particles. The occurrence of a distinct porous structure will result in an additional flow of liquid through open pores. Flow phenomena will be very similar to flows through porous deposits.

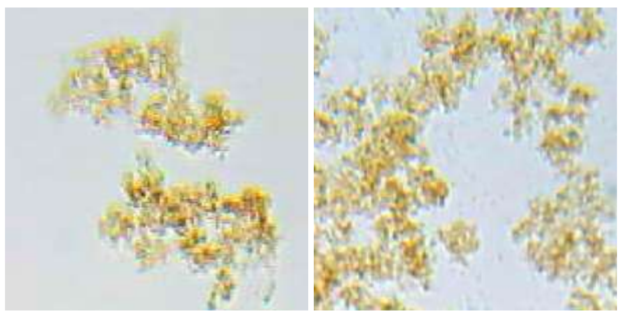

Fig. 5. Flocs of porous conglomerates

This issue is so complicated that researchers have not developed any universal methods for the modeling of the sedimentation process yet. To date no sufficiently accurate models describing hindered settling and zone settling that are characteristic of a floc suspension have been developed. Therefore, it is appropriate to search for measurement methods allowing for the determination of basic parameters of a suspension. The results of such measurements may be used to develop new mathematical models.

3. Methodology of density determination

The paper presents the description of the measurement method used to determine the density of the post-coagulation sediment using X-ray tomography. The method consists in the determination of the density by means of the gravimetric method. For this purpose, the post-coagulation precipitate was pressed into cylindrical samples with a diameter of 2.5 mm. Samples were weighed on the laboratory scales with a measurement accuracy of 0.0001g. The volume of the sample was determined on the basis of the reconstructed three-dimensional images from the X-ray tomography. The density was determined according to the formula:

$$\rho = m/V, \tag{2}$$

where: ρ- density, kg/m^3, m - mass, kg, V - volume, m^3.

The dried post-coagulation sediment was subjected to the pressing process on the press (Fig. 6). The press is composed of a cylindrical sleeve in which there are two squeeze rollers between which the material is pressed. The pressing process was carried out by lifting the hammer of 1 kg to a constant height of 5 cm, then dropping it. The compression was adjusted by means of the number of strokes of the hammer.

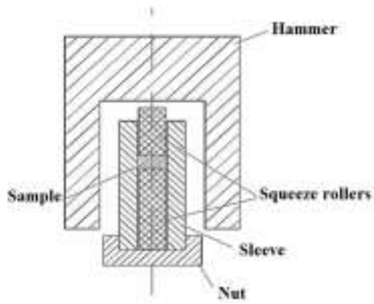

Fig. 6. Scheme of the press impact

The pressing of samples should eliminate macropores, but it should not affect the microporous structure of the matter. It is important to determine the number of hammer strokes. In order to precisely determine the sample volume, X-ray microtomography was used. The X-ray microtomograph gave a series of cross-section images for each sample. The images from the microtomograph were shown in grayscale, where light areas represent the precipitate and dark areas represent air in the macropores of the sample.

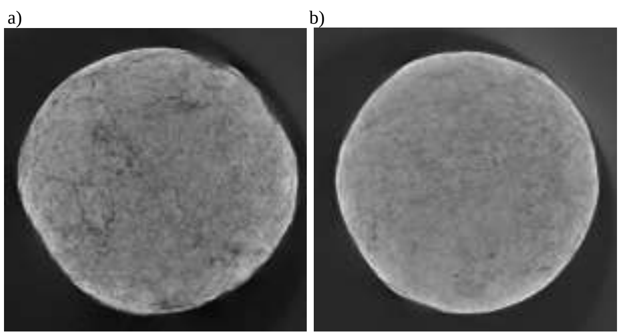

Fig. 7. Cross section of the sample for a) 1 stroke of the press, b) 8 strokes of the press

The volume of the sample was determined by means of the sum of the volume of stored solids obtained from cross-section images with a thickness of one pixel. To determine which pixel represents the sediment, the image processing was carried out. This process was conducted using the LabVIEW Vision module. The application reads the selected folder of images. Pixels representing the sediment were isolated for subsequent scans. The first step was to determine the threshold of the gray level. In the next step, the artifacts were removed, and the number of pixels representing the sediment was determined. The volume of a single section was calculated (one pixel of thickness) on the basis of this scale. Then, the density was calculated for each sample [9]. In case of the sample subjected to only one stroke of the press, macropores are visible, which results in the incorrect determination of the volume (Fig. 7a). In case of eight strokes, the size and the number of macropores were significantly reduced, but it was not possible to completely eliminate them (Fig. 7b). The determination of the volume of a small number of macropores can be corrected, and measurement errors are not significant. However, the proper threshold in the thresholding of gray shades has to be chosen.

A series of cross-sections of the samples obtained thanks to the X-ray images was studied. The representative images are shown in Figure 7. The images from the scanner are shown in a grayscale, where light areas represent the precipitate, and the dark area is the air in the macropores of the sample. After one stroke of the press (Fig.7a), there were a large number of macropores in the sample, whereas after eight strokes (Fig. 7b), the number of macropores significantly decreased. However, obtaining a single structure will lead to the destruction of the microporous structure, which is a characteristic parameter of solids.

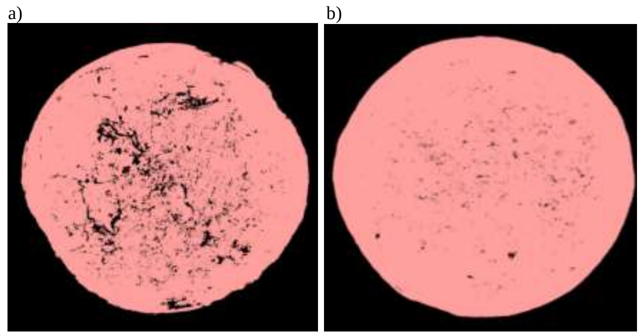

Fig. 8. Cross-section of the sample after a) 1 stroke of the press, b) 8 strokes of the press

The volume of the sample was determined by adding the volume of the solid component obtained from the images of cross-sections with a thickness of one pixel. To determine which pixel represents settlements, the image analysis process was carried out. The process of image analysis was conducted using the LabVIEW Vision module (Fig. 8) [10].

The determination of the proper number of threshold values was very important. Figure 9 shows the relation between the volume and the threshold amount. If the threshold amount is within the range from 55 to 105, the value of the indicated volume does not change. It proves that the measurement result is insensitive to the change in the threshold number in that range. For further tests, 81 was assumed as the threshold number for which the volume is equal to the mean value of 5.9 mm^3.

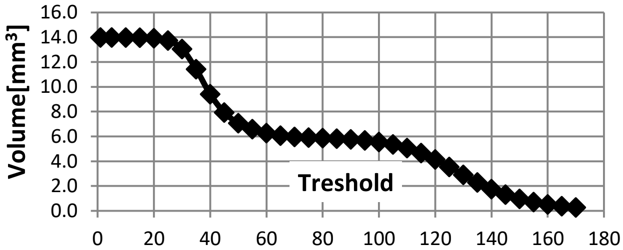

Fig. 9. Graph presenting the relation between the sample volume and the threshold amount

4. Methodology of density determination

Studies were carried out using model water which was prepared by mixing appropriate portions of distilled water with salt doses presented in Table 1 with the use of a magnetic stirrer. For the repeatable measurement, model water was prepared. The water was prepared by dissolving salt composed of: 24 g/dm^3 of sodium chloride NaCl, 12 g/dm^3 sodium sulfate Na_2SO_4 and 0.05 g/dm^3 iron (II) sulfate $FeSO_4$. For the purposes of the precipitation of dissolved iron to form a sediment, pre-hydrolyzed aluminum coagulant called Flokor 1.2A was used. It was decided to use 60 g/m^3 as a dose of the coagulant [2, 8]. In the first step, after the coagulation process, the precipitate was separated from the water by means of filtration on a cellulose filter, with a pore size equal to Φ 45μm. At the second stage, the precipitate was dried using a moisture analyzer at a constant temperature of 105°C to eliminate water from micropores.

Tab. 1. Doses of salt in model water

NaCl	Na_2SO_4	$FeSO_4$
[g/dm^3]	[g/dm^3]	[g/dm^3]
24	12	0.05

Figure 10 shows the results of densities calculated for different numbers of strokes of the hammer. Six and more strokes of the press, provided a relatively small number of macropores in the sample without destroying its microporous structure.

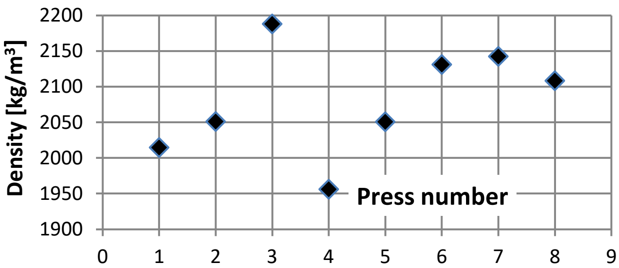

Fig. 10. Graph presenting the relation between the sediment density and the number of strokes of the hammer

5. Summary

The presented measurement method is used to precisely determine the density of porous materials containing both macro and micropores. This method can be used to determine density patterns of porous materials. Examples of the determination of the post-coagulation sediment density are an introduction to further studies and the modification of the developed method.

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