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Method for the measurement of the stratification of concentrated suspensions

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ABSTRACT

A simple, fast and precise method for evaluation of precipitate formation kinetics and its amount in concentrated suspensions that are stored in closed containers after production has been developed. The method is based on the measurement of the mechanical pendulum period change over time. The principles of the development of this method are presented: theoretical fundamentals of measurement using this method have been analyzed; it was proved that the swing period and amplitude change rate are informative pendulum parameters; a pendulum variant was determined experimentally that allows to avoid the error introduced by the movement of suspension inside the container and reduces the requirements for the container mass uniformity; it was determined that in order to reduce the error it is appropriate to start the period duration measurement from the 5 period from the beginning of the swing; the dependence of the swing period on the mass of the precipitate was evaluated experimentally.

1. Introduction

Depending on particle size, mixtures obtained having dispersed solid particles in liquid (liquid medium) are referred to as colloids (particle size of 1–1000 nm) or suspensions (particles of >1000 nm) [1,2]. The range of solid particle size in mixtures used in practice is usually wide - from sizes typical of colloids to sizes characteristic of suspensions, and those mixtures are referred to as suspensions.

In accordance with colloid theory [3,4], colloidal particles contained in suspensions carry an electrical charge. Colloidal particles of the same chemical composition in the same colloidal system carry charges of the same sign (positive or negative), therefore they push each other and do not aggregate in clusters. However, due to certain reasons neutralizing repulsive charges (adding electrolyte solution, significant temperature rise accelerating Brownian motion, intense increase of particle concentration, etc. manifesting one by one or a few at a time), particles lose their charges and start clustering in aggregates (coagulating), because having lost their repulsive charges they no longer repel each other. Aggregates of colloidal particles adherent to each other having formed during coagulation get separated from liquids and settle in formation of sediments, if particles are hydrophobic, or, in case of hydrophilic particles, aggregate in clusters forming a three-dimensional structure with cavities, meanwhile molecules of a liquid medium fill the cavities, thus forming a very thick solid jelly-like mass called gel. Colloidal particles of metals, metal oxides and many other inorganic materials are hydrophobic. Aggregates having formed after their coagulation separate from a liquid medium and settle forming sediments, meanwhile colloidal particles of organic substances are hydrophilic colloids resulting in gel formation during their coagulation [4].

Suspension particles, which are larger than colloidal particles, are electrically neutral, and if no additional measures are taken, when suspended, they do not hold out for long, and, slower of faster, start to settle. The speed of particle sedimentation has been generally known to depend on their size, mass, viscosity of the medium, etc.

Suspensions, widely used in agriculture throughout the world, are so called highly concentrated fertilizer suspensions, which after dilution with water are used to fertilize plants. The range of solid particle size in these suspensions is wide – from sizes typical of colloids to sizes characteristic of suspensions. The stability of these suspensions (steadiness of dispersity of particles comprising them) over time after their preparation is a very important quality characteristic.

The main component of fertilizer suspensions are substances containing microelements necessary for plants. These substances usually are inorganic materials, typically salts dissociated into ions, which contain elements (Zn, Mn, Cu, Fe, Co, etc.) necessary for

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fertilizing plants. The concentration of these materials in the suspension is high and this causes their crystallization. The formed micro-crystals get suspended in a liquid medium. Since the concentration of substances is very high, micro-crystals tend to grow. If their growth is not stopped by any means, they grow to the size where they are no longer able to continue being suspended and start settling, thus forming sediments. To increase stability of suspensions and their operating efficiency are used special measures: surface active organic agents containing hydrophilic groups (surfactants), which suppress crystal growth and stabilize hydrophobic colloidal and larger particles, are adding during suspension production [5]; stable metal chelates are formed, usually using ethylenediaminetetraacetic acid (tetrasodium EDTA) for this purpose [6]; bentonite (montmorillonite clay, the main component of which is hydrated aluminosilicates) [7] and other clays containing hydrated aluminosilicates (such as attapulgite) [8] are added (up to 5%) in the suspension – particles of mentioned clays are very small (nanometer size), and they act as crystallization centers promoting the formation of a large number of fine crystallites of these salts in concentrated salt solution, which stay suspended in the concentrated solution; moreover, clay makes the suspension thicker, more viscous, thus suppressing particle sedimentation and stabilizing the suspension.

The written above show that fertilizer suspensions usually are very complex mixtures. Determining the stability of such mixtures in application of well-known methods is impossible for the following reasons: the method for determining colloid stability based on the measurement of zeta potential is not suitable for evaluating the stability of the entire suspension as such, because the majority of particles comprising the suspension are larger than colloidal particles and have no charge, while this method is used solely for evaluating the stability of particles carrying charge. Exact evaluation of the speed of particle sedimentation in polydispersed mixtures using a method of mathematical calculations is complex [9] or even impossible, because the size of particles and the state of the entire system may gradually change due to above-mentioned physical-chemical processes occurring in the suspension over time. Thus currently there is no method, the application of which would allow to exactly evaluate the stability of complex mixtures, such as fertilizer suspensions. However, evaluating the stability of such widely used in practice mixtures, after production usually stored in closed containers, is absolutely necessary, because physical-chemical processes leading to the sedimentation of particles comprising the suspension and the stratification of the suspension, are usually irreversible, and therefore the stratified suspension becomes faulty and unsuitable for use. When stirring stratified suspension, the sediment layer and other layers of suspension may intermix, however, having stopped stirring, the suspension quickly stratifies because of a very rapid sedimentation of particles due to irreversible changes, such as coagulation, crystal growth, aggregation and etc., having occurred as a result of previously occurred physical-chemical processes.

The aim of this work is to resolve of the above described problem of the assessment of the stability of complex mixtures, including very important for practice concentrated fertilizer suspensions – to create a method for the determination of the concentrated suspensions stratification and estimation of the amount of the precipitate in the concentrated suspensions which after production are stored in closed containers.

2. Precipitate evaluation mode selection

When selecting the precipitate evaluation mode that could be used to develop the method for evaluation of sediment formation and its amount in concentrated suspensions, the research was based on differences in widely known physical properties of freshly made suspension and precipitate, such as density, X-ray penetration, ultrasound propagation velocity, and specific heat, as well as the shift of the center of gravity of container in which the suspension is being stored, caused by the formation of precipitate.

2.1. X-ray radiation penetration evaluation method

The denser the material is, the poorer is its X-ray permeability. As the sediment density is greater than of the suspension above it, this method is suitable for the measurement of precipitate density. However, it is very expensive and application of sensitive X-ray equipment in industry is limited.

2.2. Ultrasound propagation velocity measurement method

The ultrasound propagation velocity and attenuation depends on the density and composition of material. However, the ultrasound propagation velocity measurement is problematic due to the precision of the base (distance between emitter and receiver) and impact of the temperature, and therefore it is not suitable to evaluate the precipitate.

2.3. Mechanical impedance method

When an indenter with an acceleration transducer hits materials of different density, the pulse shapes obtained during the impact are also different. But the pulse shape depends on the construction of the vessel as well. In this work, materials of different density, the sediment and the suspension above it, is being stored in vessels of different constructions, and therefore this method is unsuitable for the evaluation of precipitate.

2.4. Specific heat measurement method

Specific heat of materials of different density and composition is different. The specific heat of sediment differs from the specific heat of suspension located above the sediment. This can be illustrated by the following experiment: after keeping the tank with suspension at a constant temperature for about 1 h, and then relocating it into a room in which the ambient temperature differs from the temperature at which the suspension was maintained previously by 10–20 °C, thermal imagers show visible stratification inside the tank.

All the latter three methods mentioned herein cannot be used to evaluate the mass of the sediment.

2.5. Weighing method

Sediments accumulate at the bottom of the vertically-installed containers. The gravity centers of equal containers with and without the precipitate will differ. We put the container containing the suspension without precipitate inclined at 90° on the lever scales, so that the instrument is in equilibrium. Afterwards and in the same way, we place the container that contains suspension with precipitate in the same position. As the center of gravity in this case will be shifted toward the bottom, we will have a torque proportional to the amount of sediment. However, when the tank is tilted, sediment changes its position in the container. Therefore, the determination of its mass becomes approximate.

2.6. Pendulum method

The swing period of the physical pendulum depends on the distance between the axis and the center of gravity of an swinging

body, as well as its moment of inertia with respect to an axis passing through its center of gravity parallel to the axis of oscillation [10]. If we alternatively put uniform containers with and without the precipitate on the pad (base) of the pendulum, their swing periods will be different – the swing period of the container with the sediment will increase due to the lower center of gravity. Since the period – time interval – as a physical magnitude can be measured potentially with the greatest accuracy, this method was chosen for the further examination.

3. Investigation of the pendulum principle suitability for development of the method for evaluation of precipitate formation and its amount in concentrated suspensions

3.1. Pendulum measurement system

In case of unstratified suspensions (i.e. containing no precipitate), the density is uniform throughout the entire volume. When the suspension becomes stratified, the density is lowest at the top of the container, and highest at the bottom. Thus, the suspension stratification changes the position of the center of gravity of the container. The stratification-related shift of the center of gravity constitutes the essence of the precipitate evaluation using the pendulum method. The pendulum measurement system is shown in Fig. 1. Magnetic field transducer 7 is used for the precision measurement of the swing period, which is triggered when the container 6 passes the equilibrium position. Eddy current converter 1 with an amplifier 2 serves for the purpose of measuring the motion of pendulum 5. The controller 3 performs digitization and period measurement functions. The control and data processing takes place on a computer 4.

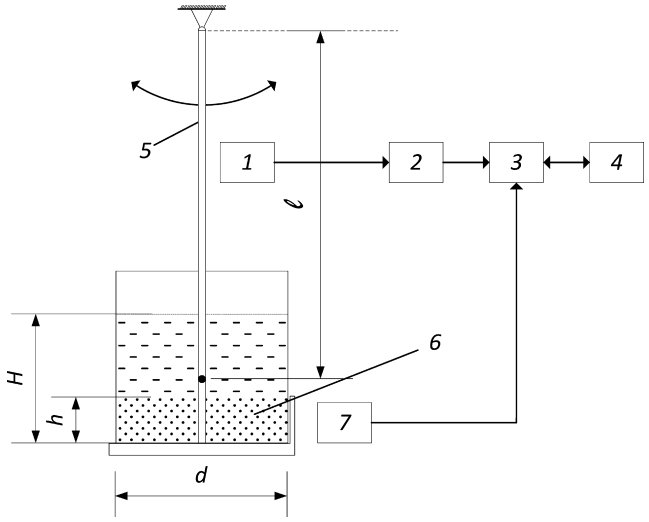

Fig. 1. Pendulum measurement system: 1 – eddy current converter, 2 – amplifier, 3 – controller, 4 – computer, 5 – pendulum, 6 – object, 7 – magnetic field transducer.

The principle of measurement using the pendulum method is shown in Fig. 2. The measurement model was developed assuming that the container a contains suspension in which the sediment layer of height h has formed, and the particles comprising the sediment are uniformly distributed; the container b contains liquid (dispersive medium), which is homogeneous; container c_1 contains particles of uniformly distributed substance(s), i.e. dispersive phase, which when dispersed in the liquid (dispersive medium) leads to the formation of suspension; container c_2 contains uniformly distributed precipitate resulting after the settlement of particles from the container c_1. The mass of the liquid in the container b is m_1, the mass of the particles of the substance from the containers c_1 and c_2 is m_2.

The swing period T is the main information-carrying parameter, which is determined from [10].

$$T = 2\pi\sqrt{\frac{I}{mgl}}, \tag{1}$$

here I – moment of inertia,

$$I = ml^2 + I_0, \tag{2}$$

here m – total mass of the container,

$$m = m1 + m2. \tag{3}$$

I_0 – moment of inertia with respect to an axis parallel to the swing axis and passing through the center of gravity of the container:

$$I_0 = (m1+m2)\left(\frac{m1\cdot R + m2\cdot r}{m1+m2}\right)^2 + m1\left[\left(\frac{m1\cdot R + m2\cdot r}{m1+m2} - R\right)^2 + \frac{1}{12}(H^2+d^2)\right] + m2\left[\left(\frac{m1\cdot R + m2\cdot r}{m1+m2} - r\right)^2 + + \frac{1}{12}(h^2+d^2)\right]. \tag{4}$$

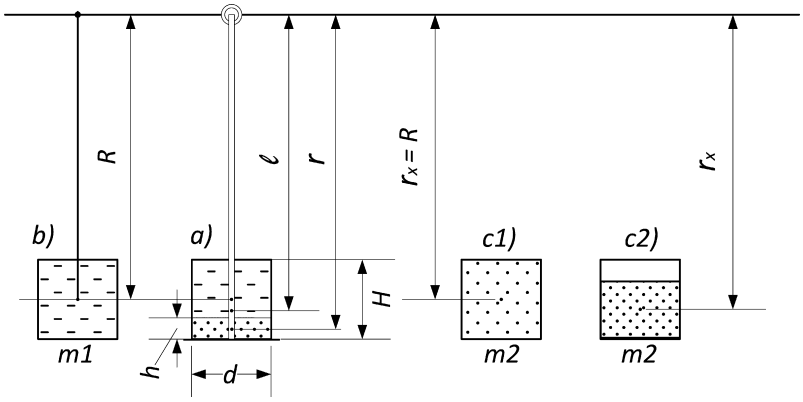

Fig. 2. The measurement principle using the pendulum method: a – container containing suspension in which the sediment layer has formed; b – container containing homogeneous liquid (dispersive medium) of mass m_1; c_1 – container containing particles of uniformly distributed substance(s), i.e. dispersive phase, of mass m_2; c_2 – precipitate formed after the settlement of particles from the container c_1. H and d – height and width of container a; h – height of the sediment layer formed in container a; R – distance to the gravity center of container a before stratification of the suspension; l – distance to the gravity center of container a after the stratification (precipitate formation); r – distance to the gravity center of the precipitate.

Distance l is determined from

$$l = \frac{m1 \cdot R + m2 \cdot r}{m1 + m2}. \quad (5)$$

The swing period is found using formulas (1)–(5). It should be noted that the friction and air resistance are not taken into account here.

We shall consider characteristic cases.

3.1.1. Long pendulum, $l \gg H$

We assume $H = h$, i.e. homogeneous filler.

Additionally, $I_0 \ll \mathrm{ml}^2$, and $l = R$, and $m_1 = m_2$; $m_1 + m_2 = m$.

Then the swing period

$$T_1 = 2\pi\sqrt{\frac{R}{g}}. \quad (6)$$

Another case, when the precipitate is contained at the bottom only. Then $h \ll H$, $r = R + \frac{H}{2} - \frac{h}{2}$ and

$$l = R + \frac{1}{4}(H - h); \quad l \approx R + \frac{1}{4}H. \quad (7)$$

The swing period

$$T_1' = 2\pi\sqrt{\frac{R + H/4}{g}}. \quad (8)$$

The relative change – increase – of the period after the precipitate settles to the bottom, results from (6) and (8):

$$\frac{\Delta T}{T_1} = \frac{H}{8R}. \quad (9)$$

3.1.2. Short pendulum, $l = H$

We assume $H = h$, i.e. homogeneous filler; $H = d$, $m_1 = m_2$, $m_1 + m_2 = m$.

The swing period is determined using (1). The moment of inertia

$$I = mH^2 + \frac{m}{12}(H^2 + d^2). \quad (10)$$

Using (1) and (10) we obtain

$$T_2 = 2\pi\sqrt{\frac{7H}{6g}}. \quad (11)$$

When $h \ll H$ and all the other conditions remain the same, from (2) and (4) we have:

$$I = \frac{7}{4}mH^2. \quad (12)$$

and

$$T_2' = 2\pi\sqrt{\frac{7H}{4g}}. \quad (13)$$

The relative change (increase) of the period is found from (11) and (13):

$$\frac{\Delta T_2}{T_2} = \frac{T_2' - T_2}{T_2} = \sqrt{\frac{3}{2}} - 1;$$

$$\frac{\Delta T}{T} = 0.225. \quad (14)$$

In the examined cases we did not consider the motion of the material contained in the container during the swing. It is clear that the influence of the motion will be greater for short pendulum case. As follows from (9) and (14), increased sensitivity to the amount of precipitate is provided by short pendulum. Therefore, there must be an optimal length of the pendulum. In a real measurement system we assume $l = (2 \div 3)H$.

The sediment viscosity has significant influence on swing period T. Therefore in order to reduce the influence of motion of material contained in the container, occurring during the measurement, the data acquisition and measurement can be started only from the fifth swing period. When satisfying this requirement, we achieve that the measurement is started after the sediment inside the container stops to splash and starts to move in tact with the container, smoothly on its bottom. The experimental observation of the change of the swing period, starting from the first period, showed that this choice is sufficient to reduce to the required size the component of the random error of the precision measurement of sediment amount. The equation 6 does not consider the movement of material, as well as friction and aerodynamic impact on the duration of the period. The systematic influence of these factors and the shape of the container are eliminated by calibration.

3.2. Calibration of the pendulum measurement system

The length of the pendulum is 425 mm. A steel imitator with dimensions (length, width, height, mm) 186/65/48 mm, and weight $m_0 = 4500$ g, was used for calibration. It was positioned at the center of the pendulum base so that its long axis was directed to the axis of swing. Initial deflection angle of the pendulum was 15°. The pendulum swing period T was measured and the measurement uncertainty was evaluated [11]. In total 10 measurements were completed without removing the imitator, and the following data was calculated: the average period $T_0 = 1211424.2$ μs, the period root-mean-square deviation $S_x = 11.36$ μs, and the expanded relative uncertainty of the period measurement $U_{T_0} = 1.88 \cdot 10^{-5}$. The example of the swing signal is presented in Fig. 3.

Tests were also conducted by placing additional sediment-imitating weights of 200 g, 400 g, and 800 g, on the base of the pendulum.

The weight of respective mass is put in place, and the pendulum swing period (which we will denote as T) is re-measured. The result of the research is shown in Fig. 4. As it can be seen from the test result, the change of the period almost linearly depends on the added extra weight (determination coefficient reaches 0.995).

The precipitate mass measurement resolution threshold:

$$\Delta m = \frac{2 \cdot S_x}{T - T_0} \cdot m. \quad (15)$$

Here T_0 is the initial swing period, and T is the swing period after adding the weight of $m = 800$ g.

$$\Delta m = \frac{22.72}{9422.9} \cdot 800 = 1.94 \approx 2 \text{ g}.$$

4. Evaluation of precipitate formed in concentrated fertilizer suspensions

For the precipitate investigation, we selected two containers filled with a concentrated fertilizer suspension, containing manganese, zinc, and copper-based substances and suspension stabilizers. The suspension in the container No. 1 was freshly prepared, while the container No. 2 contained suspension made 4 years ago. The volume of each container was 5 L, the mass was 9 kg.

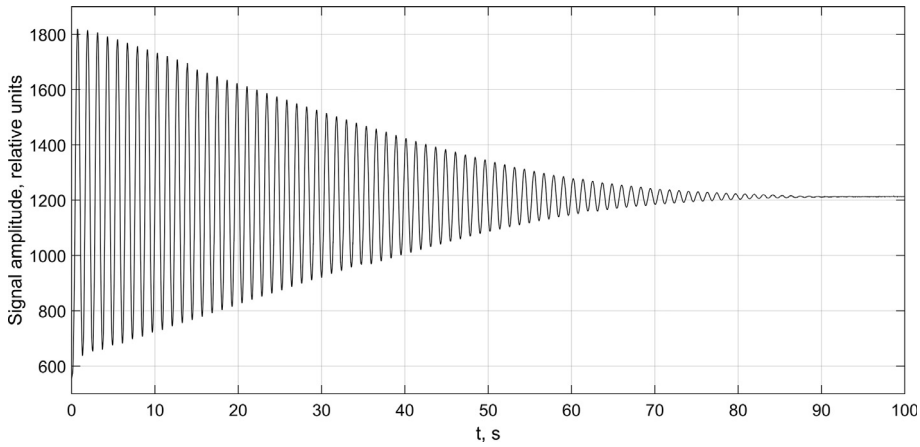

Fig. 3. Example of the swing signal.

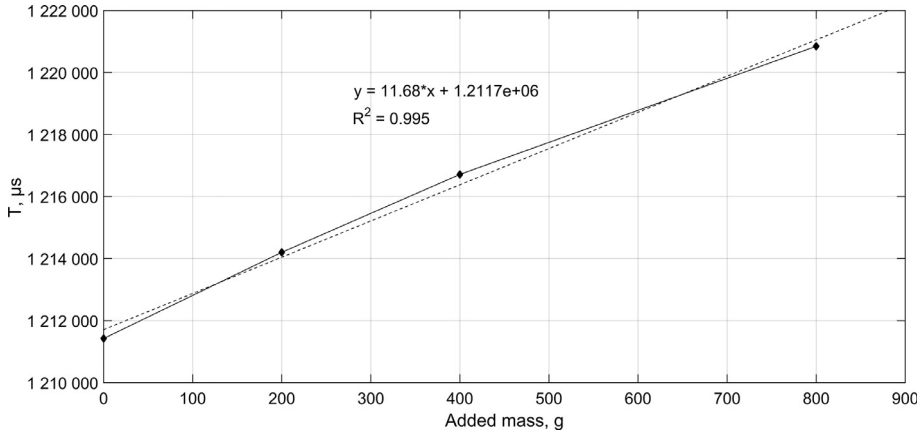

Fig. 4. Change of the period obtained by adding extra weights which imitate the precipitate.

Instrument *CILAS 1090 Liquid* was used to measure the particle size. Fig. 5 shows the particle size measurement results of the freshly prepared suspension (container No. 1), and Fig. 6 shows corresponding results for the particles of the sediment formed in the suspension produced 4 years ago.

The data presented in Fig. 5 shows that the major part of particles contained in the freshly made suspension had sizes ranging from 1 μm to 10 μm.

The sediment particle size measurement data shown in Fig. 6 (container No. 2) indicate that the sediment particle size prevailed in the range of 100 μm.

When comparing data from Figs. 5 and 6, it can be noted that the precipitate-forming particles are substantially larger than in the fresh suspension.

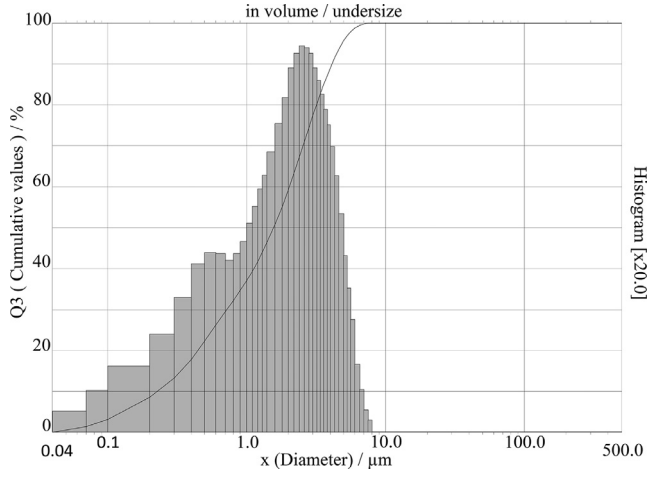

Fig. 5. Freshly made suspension particle size measurement data.

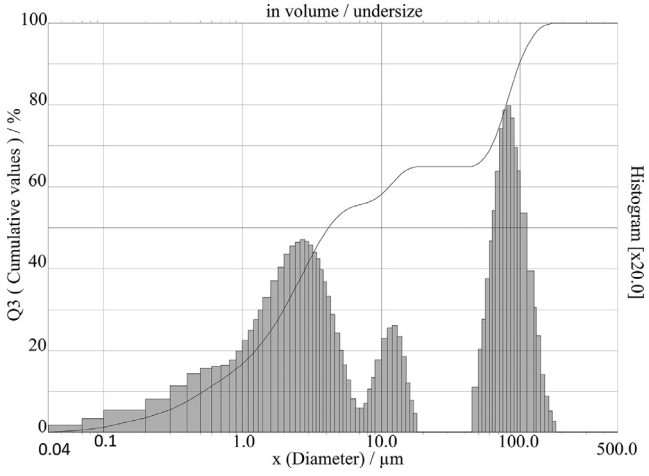

Fig. 6. Particle size measurement data for the precipitate from the suspension produced 4 years ago.

The particle chemical composition was determined by performing and X-ray diffraction analysis. Freshly made suspension and sediment were dried at the temperature of 110 °C, and the analysis was carried out using the *DRON – 7* diffractometer (Cu Kα radiation).

Analysis has shown that diffractograms of freshly made suspension and of precipitate are identical, i.e. chemical composition is identical in both cases. One of the identical diffractograms is presented in Fig. 7.

We used our developed pendulum-based measurement system to evaluate the suspension stratification and to determine the amount of precipitate.

In order to optimize the measurement process, i.e. to achieve sufficient accuracy with the minimum duration, an swing research has been carried out when loading the pendulum using the steel imitator (more detailed description in Section 3.2), container No. 1 with freshly made suspension, and container No. 2 with suspension produced 4 years ago (with precipitate). The change of swing amplitude over time is shown in Fig. 8.

As might be expected, the swing amplitude has the slowest decline in the case of imitator, and the fastest – in the case of fresh fertilizer suspension. In the first case the friction and air resistance leads to decline, and in the second – the suspension movement in the container. When there is a large amount of sediment it has approached to the first variant and the rate of the decline is an intermediate. Fig. 8 shows that an evaluation of the amplitude decrease in aforementioned three threshold cases indicate the possible measurement period of 20 s interval. When the swing period is about 1 s, 20 swings are performed in this interval.

The swing amplitude change also can be considered as an informative parameter, although it requires a long measurement time.

It is possible to set the beginning of the measurement and the number of measured periods in a programmable way. The measurement system calibration is performed using the container No. 1 filled with fresh suspension and a weight of a known mass. Calibration with a real container is advisable as in this way it is possible to assess its geometry and the suspension motion inside the container. During calibration, we evaluate the uncertainty of precipitate mass measurement. After completing 10 measurements, the expanded measurement uncertainty calculated according to [11] for the 5-L, 9 kg container was 22 g. When measuring containers No. 2 (filled with 4-years old suspension) and No. 3 (filled with 1-year old suspension), their swing periods were compared against the period of container No. 1 and then converted to the mass of precipitate. The measurement results are visualized on computer. The software "window" is shown in Fig. 9. The measurement system calibration reflects the left "window" side (Fig. 9). The middle section shows the signal of swing and change of period, and the right – the result of the measurement. In this case, corresponding to container No. 2. The measurement results are listed in Table 1.

All containers were identical and equally filled. We see that the greatest amount of precipitate is in the container No. 2, in which there is the suspension stored for the longest time.

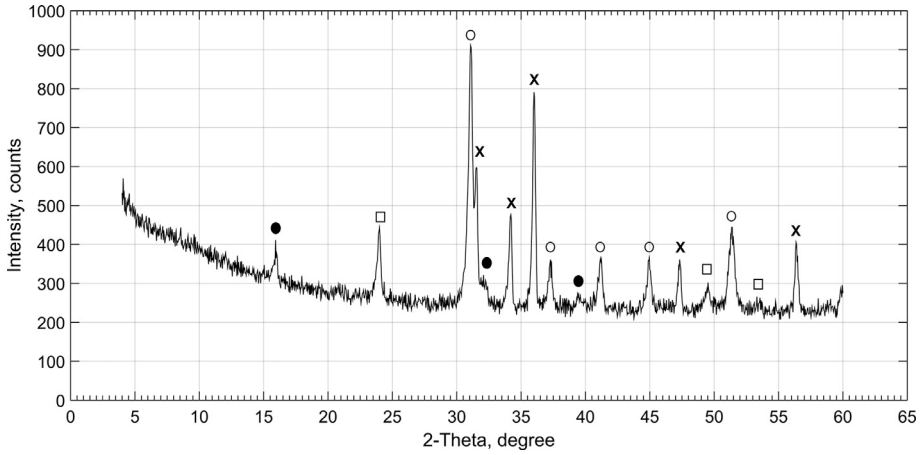

Fig. 7. Diffractogram of precipitate. □ – $CaAl_2Si_2O_8$, ○ – $MnCO_3$, × – ZnO, ● – $CuCO_3 \cdot Cu(OH)_2$.

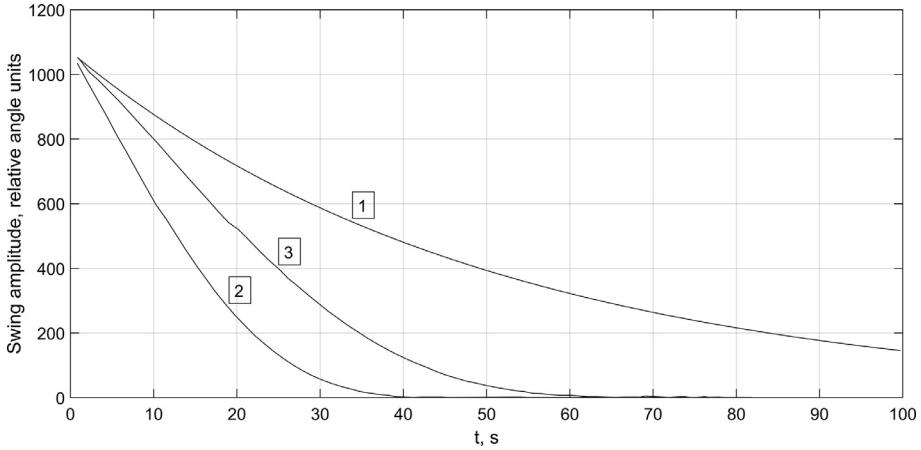

Fig. 8. Change of swing amplitude over time, when the pendulum is loaded with: 1 – imitator; 2 – container No. 1; 3 – container No. 2.

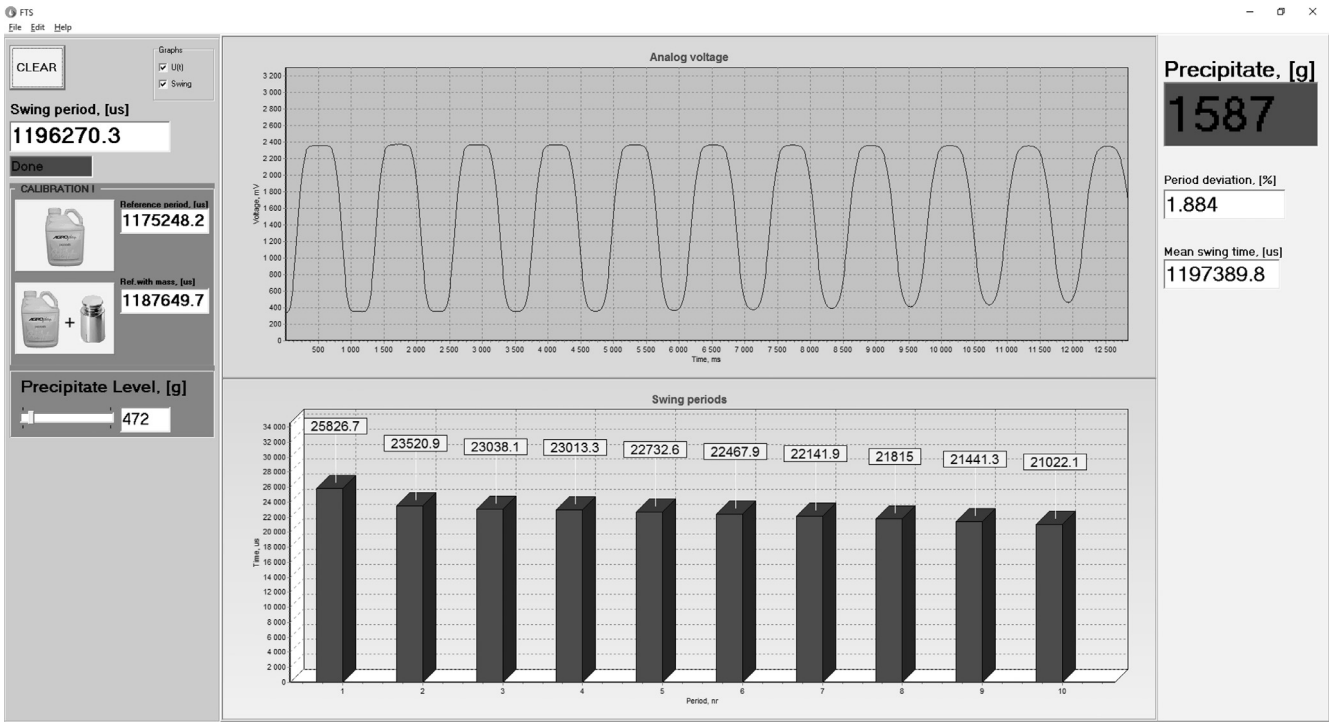

Fig. 9. Example of the measurement system software "window".

Table 1
Measurement results.

Container No.	Swing period, μs	Mass of precipitate, g
1	1175248.2	0
2	1197389.8	1587 ± 22
3	1186794.5	828 ± 22

Further presented experimental results show that the precipitate identification and their amount measurement system can be used to assess the dynamics of the particle settling process.

We investigated the particle sedimentation dynamics with the formation of precipitate, using suspensions of the same composition but stored for different periods of time after production, in which when they are shaken all the particles (even those which had settled before shaking) are distributed in entire volume of suspension. Suspension stored for a long time after production (4 years) proved to be unsuitable for this experiment, because when shaking only a part of its sediment rose up from the bottom and spread in the suspension volume, and the most part of the sediment remained on the bottom. Therefore, further we explored just two suspensions – freshly made and stored after production for 1 year. The experiments were performed with two containers, filled with suspensions, respectively, with freshly made and stored after production for 1 year. The mass of suspension in each container 9 kg.

Data presented in Fig. 10 evidently show that the precipitate identification and their amount measurement system can be used to assess the dynamics of the particle settling process because its measurement results allow to evaluate the difference of the particle settling process dynamics in the stabile (freshly made)

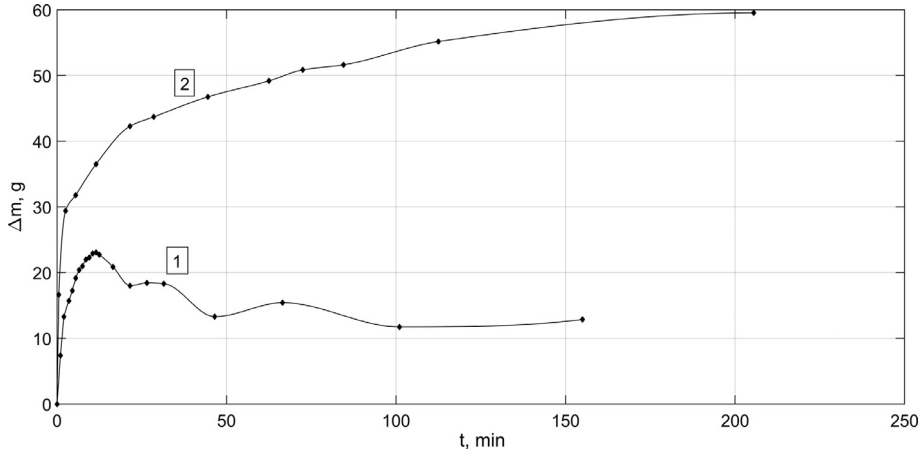

Fig. 10. Suspension sedimentation dynamics: 1 – fresh suspension; 2 – suspension produced 1 year ago.

and in the less stabile (stored for 1 year) suspensions. It is seen in Fig. 10 as well that the sensitivity of the measurement is high – the measurement results show (curve 1) approximately 10 g of predisposed to precipitation particles in the freshly made suspension the mass of which, as was mentioned above, is 9 kg.

5. Conclusions

1. Using a method of mechanical pendulum, a simple, fast and precise method for evaluation of precipitate formation dynamics and its amount in the concentrated suspensions, stored after production in closed containers, was created based on the measurement of the mechanical pendulum amplitude change over time.
2. It was determined that the swing period dependence on the mass of precipitate can be considered as linear. The expanded measurement system calibration uncertainty, when calibrating using 4.5 kg mass imitator, was 2 g, while expanded uncertainty of precipitate mass measurement in a real 9 kg mass container was 22 g. The measurement time not exceed 10 s.
3. The validity of this method was approved after conducting measurements of the precipitate amount and the stability of the concentrated suspension of agricultural fertilizer, with the peak of its particle size distribution at 2 μm (when freshly produced) and the particle size distribution maximum of the sediment formed in it at 90 μm after storing the suspension for 4 years.

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