Measurement 106 (2017) 101–108

Contents lists available at ScienceDirect

Measurement

journal homepage: www.elsevier.com/locate/measurement

Method for the measurement of the stratification of concentrated suspensions

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ARTICLE INFO

Article history:
Received 26 September 2016
Received in revised form 19 April 2017
Accepted 22 April 2017
Available online 24 April 2017

Keywords:
Concentrated suspensions
Precipitate formation kinetics
Precipitate amount measurement
Mechanical pendulum
Evaluation of stratification

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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http://dx.doi.org/10.1016/j.measurement.2017.04.036
0263-2241/

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