SLOVENSKI STANDARD
SIST-TP CEN/TR 16394:2015

01-februar-2015

Karakterizacija blata - Protokol za pripravo sintetičnih suspenzij

Characterization of sludges - Protocol for preparing synthetic suspensions

Charakterisierung von Schlämmen - Protokoll zur Herstellung synthetischer Suspensionen

Caractérisation des boues - Protocole de préparation de suspensions synthétiques

Ta slovenski standard je istoveten z: CEN/TR 16394:2014

ICS:

13.030.20 Tekoči odpadki. Blato Liquid wastes. Sludge

SIST-TP CEN/TR 16394:2015 en,fr,de

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

CEN/TR 16394

November 2014

ICS 13.030.20

English Version

Characterization of sludges - Protocol for preparing synthetic suspensions

Caractérisation des boues - Protocole de préparation de suspensions synthétiques

Charakterisierung von Schlämmen - Protokoll zur Herstellung synthetischer Suspensionen

This Technical Report was approved by CEN on 18 August 2014. It has been drawn up by the Technical Committee CEN/TC 308.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. CEN/TR 16394:2014 E

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Foreword

This document (CEN/TR 16394:2014) has been prepared by Technical Committee CEN/TC 308 “Characterization of sludges”, the secretariat of which is held by AFNOR.

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Introduction

In order to carry out lab-scale tests of suspensions, and interlaboratory validation trials for standardized methods, it is necessary to have samples with constant characteristics available. When dried suspension samples cannot be used and testing requires fresh sludge samples, problems arise because:

a) most suspension characteristics change over time, which makes them unfit for storage;

b) some preservation practices (e.g. freezing) are not applicable, or their effects unknown;

c) some suspension characteristics are strongly affected by handling;

d) transporting sludge samples requires special precautions and authorization.

This means that fresh suspension samples cannot be used to guarantee results reliability and reproducibility. As a consequence, fresh suspension samples should be examined by laboratories very close to wastewater or waterworks plants and analysed as soon as possible, minimizing their manipulation. As a result, the circulation of fresh suspension samples to laboratories in several countries is analytically not feasible. Interlaboratory analysis on these suspensions should be carried out through circulation of analysts, close to the place where samples are collected according to the Modified Round Robin tests procedure (CEN/TR 15252).

A valid alternative is the creation and testing of synthetic suspensions.

Synthetic suspensions are prepared on-site on the basis of a defined recipe and specified ingredients. This will also allow the comparison of results obtained from different places, at different times.

This Technical Report establishes methods for preparing synthetic suspensions, both inorganic and organic, in repeatable and quantified conditions, able to describe the behaviour of a real suspension with regard to specific parameters, and suitable for circulation as samples in interlaboratory trials for validation of standards when fresh suspensions cannot be used. It represents the state of the art of the available knowledge on synthetic suspensions.

1 Scope

This Technical Report deals with methodologies for preparing synthetic suspensions. Synthetic suspensions can be used for:

a) evaluating or testing new devices or techniques for suspension treatment;

b) studying the influence of different compounds on suspension behaviour with regard to specific parameters, e.g. settleability, dewaterability, physical consistency, etc.

The chemical, physical and biological characteristics of suspensions are subjected to changes as soon as they are collected. Guidance exists on the sampling and handling techniques (see ISO 5667-12 and EN ISO 5667-13), and on the preservation and storage procedures (EN ISO 5667-15) that help minimize changes in the composition. This is mainly achieved by suppressing chemical and/or biological activity and by avoiding contamination.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12832:1999, *Characterization of sludges - Utilization and disposal of sludges - Vocabulary*

EN ISO 17353, *Water quality - Determination of selected organotin compounds - Gas chromatographic method (ISO 17353)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12832:1999 apply.

4 Preparation of inorganic synthetic suspensions

4.1 General

The procedure specified in this Technical Report refers to the preparation of inorganic synthetic suspensions to simulate the behaviour of real inorganic suspensions. Inorganic suspensions produced by waterworks plants exhibit wide variations in their physical and chemical properties due to the raw water differences and the design and operation of each individual treatment plant. Many different products could be used for preparation of such suspensions depending on the properties to be evaluated.

Quartz sand, calcite and kaolin are the three most commonly used minerals in large industrial sectors. Hence, a combination of these three minerals is perfectly suited to simulate the behaviour of real inorganic suspensions. Role and characteristics of such materials have been discussed in [6] and [7].

4.2 Materials

Primary particle properties, such as particle shape, size distribution, specific surface area, density, surface structure, dry bed porosities, as well as their dispersion properties, for example solid concentration, or pH, have a strong influence on the physical properties of inorganic suspensions (e.g. compressibility, settleability, specific resistance), which have to be known and defined for a standardized synthetic suspension.

Table 1 gives an example of the main characteristics of kaolin, calcite, and quartz sand [6].

Table 1 — Characteristics of materials composing inorganic synthetic suspension

	Kaolin	Calcite	Quartz sand
Average particle size (µm)	5	10	20
Crystal structure	monoclinic	rhombohedric	tetragonal
Specific surface area (m^2/g)	9	3	2
Dry bed porosity (tapped-poured)	0,6 to 0,7	0,5 to 0,6	0,4 to 0,5
Density (g/cm^3)	2,60	2,70	2,65
SiO_2 (%)	46	2	99
Al_2O_3 (%)	39	0,4	0,2
Fe_2O_3 (%)	0,2	< 0,1	0,3
CaO (%)	1	55	0,1
MgO (%)	0,1	2	0,1
K_2O (%)	< 0,1	< 0,1	< 0,1
Na_2O (%)	0,2	1	0,1
Loss on ignition (%)	13	39	< 0,1
Compressibility range	0,7 to 1,1	0,2 to 0,3	0,1 to 0,2

Only binary mixtures can be used for evaluation of specific parameters. For example, mixtures of kaolin (75 % to 90 %) and quartz sand were used for rheological measurements [8]. Suppliers of polymers prefer using mixtures of kaolin (30 g/l to 100 g/l) and calcium chloride (0,5 g/l to 1,65 g/l) to prepare inorganic synthetic suspensions which easily flocculate both with anionic and cationic polymers.

For ternary mixtures, comparison tests were performed on a 30 g/l industrial suspension sampled at a granulate extraction plant.

Reasonably good compliance in terms of settleability, capillary suction time (CST), specific resistance, compressibility and suspension dryness (after compression) was achieved using a synthetic suspension consisting of 90 % kaolin, 5 % quartz sand and 5 % calcite [9].

4.3 Modalities of preparation

Ingredients are kaolin, quartz sand and/or calcite which can be mixed in different proportions to be fixed for the development of a standard inorganic suspension.

The suspensions have to be prepared from mineral powders before the start of each set of experiments in order to avoid modification of surface properties of powders. All samples shall be prepared in the same way because the preparation has a significant influence on the final state of the suspension (degree of dispersity, rheological behaviour).

A good preparation of inorganic synthetic suspension usually requires:

— fine particles and narrow particle size distribution to ensure homogeneity;

— a defined proportion of ingredients, whose respective weights should be adjusted to prepare the suspension of chosen concentration;

— a defined quality (e.g. pH, turbidity, conductivity, alkalinity, hardness) of water for suspension preparation as defined for example in EN ISO 17353;

— a description of mixing conditions between powder and water. Mixing with a magnetic stirrer at low speed (250 r/min) for 30 min is generally sufficient to achieve uniform dispersion;

— additives (HCl, HNO_3) to adjust pH ≈ 7 to achieve the highest possible suspension stability;

— additives (KCl, KNO_3) to adjust ionic strength to prevent possible interference by ions, released by the surface of the clay.

5 Preparation of organic synthetic suspensions

5.1 General

5.1.1 General

The preparation of organic synthetic suspensions able to reproduce a biological real sewage suspension is a more complex matter than conventional particulate systems. Sewage suspensions, whichever the treatment, consist mainly of water (95 % to 98 %), but also contain a high number of organic and mineral matters, such as colloids, flocs, fibres, micro-organisms, particles with size varying from 1 mm to 1 µm, polymers (proteins, sugars, fats) and ions. Organic matter represents 50 % to 90 % of total solid matter and has a very complex composition (mixture of bacteria, cellular wastes and particles). Further, a biological suspension is a complex system for which the structural stability results from the combination of various interactions; particle charge density or hydrophobicity play important roles and cannot be summarized by only one compound [10].

Carbohydrates and proteins were found to be the major extracellular polymeric substances components, but lipids, nucleic acids and humic compounds were also reported. Calcium ions participate in the inter-polymer binding, forming a very complex bio-flocculated structure [11]. Moreover, micro-organisms interact with organic matter leading to a composition change in the suspension over time (Figure A.1) [12].

Synthetic suspensions can be prepared by direct formulation or by concentration of synthetic waters.

Preparation of organic synthetic suspensions by direct formulation

5.1.2 Materials

An organic synthetic suspension shall be a colloidal system of appropriate porosity, compressibility, visco-elasticity and sticking ability. It shall also contain proteins, lipids and polysaccharides, microorganisms and ions.

Some publications ([13], [14] and [15]) proposed a synthetic suspension composed with polystyrene latex, alginate and calcium ions. The polystyrene latex particles (0,05 % mass fraction) of a size (0,5 µm) similar to bacteria are used to simulate individual bacteria, while the polysaccharide alginate (100 mg/l) is used to simulate microbial extracellular polymers, and calcium (20 mol/l) acts as bridging cations. The stable and well-defined nature of a synthetic suspension makes it a useful, non-complex analogue for the physical and chemical properties of an activated bio-flocculated suspension, by adjusting calcium concentrations in both synthetic and activated suspensions according to settling, dewatering characteristics and their response to a cationic conditioner.

Nevertheless, synthetic suspensions show a noticeable, quantitative difference in flocculation behaviour due to the absence of a filamentous backbone structure, often present in activated suspension.

Authors ([16], [17] and [18]) improved this formulation by adding cellulose fibres and yeast to simulate the filamentous micro-organisms found in activated suspension. They proposed a synthetic suspension with 2 different solid concentrations as 30 g/l and 10 g/l. Alginate (10 %), microcrystalline cellulose (61,5 %), fresh yeast (27 %), dehydrated calcium chloride (1,5 %) and potassium chloride was used. These ingredients and amounts were established to represent the properties of a particulate, partially flocculated gel including the response to addition of a common polyelectrolytic flocculant.