European developments in standardisation of sludge physical parameters

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Abstract Characterisation is an important step in sludge management as it allows sludge properties to be evaluated and behaviour predicted. This is well recognised by the European Union (EU) countries which consider necessary the development of standardised sludge characterisation methods and procedures because objective and transparent regulations allow sludge management to be properly performed, legal requirements correctly fulfilled, and public confidence built. To this end, the European Committee for Standardization (CEN) established the Technical Committee 308 (TC308) whose scope is the production of Standards for sludge characterisation, and of Guides of good practice. In this field, physical properties are of great importance as they allow the prediction of sludge behaviour when handled and submitted to almost all treatment and utilisation/disposal operations. Activity of CEN/TC308 is developed in three Working Groups (WG) and several Task Groups (TG); in particular, TG3 of WG1 deals with physical parameters. In this paper developments regarding Standards for evaluation of capillary suction time, specific resistance to filtration, compressibility, settleability, thickenability, and calorific value, and Technical Reports dealing with a procedure for laboratory chemical conditioning and with sludge consistency (flowability/solidity) are briefly outlined. The results of relevant inter-laboratory tests for the validation of above standards are summarised.

Keywords Characterisation; physical parameters; sludge; standard methods; validation

Introduction
The need for a regular and environmentally safe utilisation and disposal of sludge deriving from the treatment for drinking water, and of municipal and industrial wastewaters, is well recognised by the European Union (EU) countries. A number of requirements, such as guide and/or limit values, are contained in the European legislation on this subject, but unlikely methods for the determination of the respective parameters are often not available or described, so the development of standardised characterisation methods and procedures for sludge becomes necessary because objective, transparent and easy to be controlled procedures would allow (i) sludge management to be properly performed, (ii) legal requirements correctly fulfilled, (iii) comparison and consistency of application done and (iv) stakeholder and public confidence built.

To this end, the European Committee for Standardization (CEN) established the Technical Committee 308 (TC308) whose main task is the production of standard methods for the chemical, biological and physical characterisation of sludge, and of guidelines for good management practice. The main objectives and strategic directions of TC308 are to (Spinosa, 2001):

• elaborate documents (standards or technical reports) on terminology, methods of analysis and characterisation, good practice for different methods of sludge use and disposal, and operational practices for preparing sludge, in order to harmonise the technical language, methods and practices for sludge across Europe;
enable compliance with legislation through the application of consistent analytical methods;

promote and enable sustainable development through good practice for the conservation of organic matter and completion of nutrient cycles;

contribute to improvements in public and environmental health and food safety through promoting and disseminating good practice;

support and contribute to the production and revision of European Directives relevant to sludge;

coordinate with and build on the work carried out by other Environmental CEN/TCs;

support European stakeholders in sludge management (legislators, public and private companies, control agencies, etc.);

give orientation to producers and users on how to meet legislation requirements in relation to the area of growing interest, such as safety, health, environment protection, etc.;

give a larger diffusion to the standards, thus favouring the global market.

The scope of CEN/TC308 includes sewage sludge and all other sludge types potentially having similar environmental effects. CEN/TC308 also cooperates with other programmes, such as ISO/TC190 “Soil quality”, CEN/TC223 “Soil improvers and growing media”, and CEN/TC292 “Characterisation of wastes”.

Work has been organised in three working groups:

- the first (WG1) dealing with the standardisation of methods for determining sludge parameters; work is developed into five task groups specifically dealing with heavy metals (TG1), nutrients (TG2), physical parameters (TG3), micropollutants (TG4) and hygienic parameters (TG5);

- the second (WG2) with the preparation of guidelines of good practice for the different options of sludge use and disposal;

- the third (WG3) with the preparation of documents on the current and future needs in sludge management, and with the development of guidance to preserve and extend utilisation and disposal routes.

Status of CEN/TC 308 work is reported in AFNOR (2005f). Standards and technical reports already published are listed in Table 1.

**Evaluation of physical properties**

The evaluation of physical properties is of great importance as their knowledge allows the prediction of sludge behaviour when handled and submitted to almost all treatment, storage and utilisation/disposal operations. It is also to put in evidence that fundamentals of procedures adopted in these characterisation methods are basically known and accepted everywhere, but each laboratory or analysis textbook follows and/or proposes different equipment size, accessories and procedures, so results obtained in different places are correct by themselves, but are difficult to compare because they are obtained in different, not standardised, conditions; in addition, statistical analysis of errors is often not available. As an example, the procedures reported in Vesilind (1974) and CNR-IRSA (1984) can be mentioned.

Standardisation activity in this area is covered by TG3 of WG1 whose work programme includes the development of “Standards” for capillary suction time (CST), specific resistance to filtration (SRF), compressibility (CPR), settleability (STL), thickenability (TCK), and calorific value (CAL), and “Technical Reports” on a “Procedure for laboratory chemical conditioning” (LAB), and on evaluation of “Consistency (flowability/solidity)” (CON).
To become an official CEN document, three approval steps must be passed: “Internal enquiry”, “Six-months enquiry”, and “Formal vote”. The validation through inter-laboratory trials is also required for documents to be published as a standard. However, for some parameters, the circulation of sample may involve characteristics change, thus avoiding a reliable comparison of results. That is particularly true for some physical parameters requiring the analysis of fresh sludge samples with the consequence that many problems occur because:

- the sludge physical characteristics change with time of storage, also considering that any preservation practice (e.g. freezing) makes things worse;
- the sludge physical characteristics are strongly affected by transport and handling;
- fresh sludge requires particular precautions and authorisation for transport by ordinary delivering systems.

As a consequence, fresh sludge samples need to be examined by laboratories close to wastewater or waterworks plants and analysed as soon as possible, minimising their manipulation; therefore, the circulation of fresh sludge samples to laboratories in several places and in different countries is not possible. A valid alternative could consist in the examination of synthetic sludge samples to be on-site prepared on the base of a defined recipe and ingredients, but a better option is carrying out validation tests through a circulation of analysts, and not a circulation of samples, thus allowing operators from the participating laboratories to meet in a common location, close to the place where samples are collected. To this end, a validation procedure for physical parameters has been defined by TG3, and successfully applied for validation of methods on CST, SRF, CPR, STL and TCK. This procedure is currently under CEN enquiry (AFNOR, 2005e).

### Results and discussion

At the time of writing, the validation of three physical parameters standards (CST, STL and TCK) has been carried out and relevant documents submitted to formal...
Standards

Capillary suction time (CST). CST is a fast and simple way to evaluate the sludge dewaterability by filtration. The measurement of CST should also make possible the evaluation, although only qualitatively, of sludge dewaterability by centrifugation through measurement of floc strength (AFNOR, 2005a). The document does include five informative annexes dealing, respectively, with description of apparatus, relations between CST and the specific resistance to filtration (SRF), relations between CST and solids content, variation of viscosity with temperature, and the results of validation trials.

The validation trials resulted in a repeatability standard deviation ranging from 3.6% for activated/thickened sewage sludge to 4.4% for activated sewage sludge, to 3.4% for waterworks sludge, and to 5.3% for digested sewage sludge. The mean value was 4.9%. The reproducibility standard deviation ranged from 5.6% for activated/thickened sewage sludge to 9.9% for activated sewage sludge, to 4.4% for waterworks sludge and to 14.0% for digested sewage sludge, with a mean value of 11.3%.

Results of validation evidenced the influence of which side of the filter paper is placed upwards, so the method procedure was amended by imposing that the filter paper shall always be placed with the same side up.

Settleability (STL). The STL determination is used for calculating the rate of sludge settling and the sludge volume index, and for evaluating the performance of settling tanks. The sludge volume index is calculated by measuring the sludge volume after 30 min settling in a 1,000 mL graduated cylinder (AFNOR, 2005b).

The repeatability standard deviation ranged from 0.2% for digested sewage sludge to 1.2% for waterworks sludge, and to 3.0% for activated sewage sludge. The mean value was 2.2%. The calculation for the reproducibility standard deviation gave results of 0.3% for digested sewage sludge, 2.2% for waterworks sludge and 5.1% for activated sewage sludge, with a mean value of 3.8%.

Thickenability (TCK). The laboratory determination of TCK, i.e. the further concentration of suspended (undissolved) sludge solids in settling under gravity, is negatively affected by bridging, wall effects and particle size effects. Ideally, this parameter should be measured in large-diameter columns having the same depth as the prototype thickener, but the above effects can be satisfactorily overcome by introducing a low-speed stirrer in a cylinder; this also helps to reduce the effect of the shallow depth. Thickenability is therefore determined by the proportion of the volume occupied by the sludge within 30 min after settling under gentle stirring in standard apparatus and under standard conditions (AFNOR, 2005c). The document does contain two informative annexes, the first describing characteristics of the stirred cylinder used for tests, and the second the results of validation trials.

In this case, only the reproducibility standard deviation was calculated because only one measurement was performed by each operator. Deviation ranged from 0.5% for digested sewage sludge to 1.0% for waterworks sludge, to 1.1% for activated/thickened sewage sludge, and to 1.8% for activated sewage sludge, with a mean value of 1.2%.
The results of validation trials are summarised in Table 2. In all cases, the repeatability and reproducibility standard deviations gave good results, around few percent units, except for CST where, as expected, higher values up to 14% were obtained.

Specific resistance to filtration (SRF). SRF is a parameter which indicates the suitability of sludge to be dewatered by means of a filtration process. The value of the specific resistance to filtration has great importance in dewatering processes as it can be useful for estimating the performance of full-scale filtering devices, mainly pressure filters, and comparing dewaterability characteristics of sludges produced in different plants. Specific resistance measurements can also give indications on both the optimal type and dosage of conditioner to be used. Three filtration procedures are described: under vacuum, under air pressure, under pressure with piston. Relative apparatuses are described in an informative annex.

Validation trials using three different sludge types, each tested at a pressure of 50, 150 and 300 kPa, were carried out in March 2005 and results are now in evaluation.

Compressibility (CPR). The above results obtained at different pressures will also allow the evaluation of CPR parameter which is complementary to that of SRF, and whose scope is to evaluate the best range of pressure to be adopted for filtration.

Calorific value (CAL). At the time of writing, this method is under CEN enquiry. The proposed method, which is not suitable for S content >1%, consists of burning the sludge sample in a bomb calorimeter calibrated by combustion of certified benzoic acid. The generated heat increases the temperature of the water which surrounds the bomb. From the temperature change, the generated heat can be determined using a calibration curve defining the amount of energy required to cause a unit change in the calorimeter temperature. The result obtained is the gross calorific value of the sample at constant volume and at the reference temperature of 25°C with all the water of the combustion products as liquid water. The gross calorific value is calculated taking into account the secondary chemical reactions and the eventual thermal losses. The net calorific value of sludge is obtained by calculation from the gross calorific value, based on the sample hydrogen content. The main difference between the gross and net calorific values is related to the physical state of water in the reaction products.

Technical reports

Procedure for laboratory chemical conditioning (LAB). This report gives a standardised procedure for the conditioning operation when selecting a conditioning product at laboratory scale considering that (i) the laboratory assessment of sludge dewaterability is sensitive to the operating procedure adopted for the conditioning step, and (ii) no generalised ranking of products in order of effectiveness can be given since the ranking changes with the sludge type, dosage of conditioning agent, degree of shearing and dewatering device. The test may give information about the energy needed for the procedure as far as determination of the mixing power is available.

Table 2 Validation trials results

<table>
<thead>
<tr>
<th>Method</th>
<th>Repeatability standard deviation s_r (%)</th>
<th>Reproducibility standard deviation s_R (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capillary suction time (CST)</td>
<td>3.4 – 5.3 (mean 4.9)</td>
<td>4.4 – 14.0 (11.3)</td>
</tr>
<tr>
<td>Settleability (STL)</td>
<td>0.2 – 3.0 (2.2)</td>
<td>0.3 – 5.1 (3.8)</td>
</tr>
<tr>
<td>Thickenability (TCK)</td>
<td>n.a.</td>
<td>0.5 – 1.8 (1.2)</td>
</tr>
</tbody>
</table>
Three informative annexes are included in the document, the first with some notes for dosage of coagulant aids, the others giving, respectively, an example for mixing device for chemical conditioning procedure and an example of table summarising the operating conditions and the results of the characterisation tests.

Physical consistency (CON). Task Group 3 (TG3) of CEN/TC308/WG1 defined three physical states for sludge (DIN, 2001):

- liquid: sludge flowing under the effect of gravity or pressure below a certain threshold;
- paste-like: sludge capable of continuous flow under the effect of pressure above a certain threshold and having a shear resistance below a certain threshold;
- solid: sludge having a shear resistance above a certain threshold.

This firstly involves the necessity to set up methods to measure values in the range of the boundary area between liquid and paste-like behaviours (limit of flowability) and that between solid and paste-like (limit of solidity). Further, the thixotropic behaviour of solid materials (from “the solid to the liquid state and vice-versa”) must be evaluated, together with the piling behaviour referred both to “compaction and physical stability”. Also, CEN/TC292/WG2, in the method EN 12457 for the characterisation of waste, included in Annex B (Informative) the description of a test for determining whether waste is in the liquid state or not.

Although the methods to be developed are partly known and used in other technology fields, e.g. soil mechanics, materials for construction works (concrete, suspensions), etc., widely accepted methodologies for the evaluation of the above properties, able to give comparable and reliable results, are not available yet. It therefore follows that it is necessary to define simple and reliable measurement procedures to be applied in the field, together with those to be used as reference in laboratory. Standardisation procedures for the material examination will consist of:

- sampling, transport, preservation, storage;
- pre-treatment;
- measurement and evaluation of results.

The CEN Technical Report “Physical Consistency” is derived from two desk studies on physical properties, the first (Report 21) dealing with flowability (Battistoni et al., 2003), the second (Report 22) with “Solidity, thixotropic behaviour and piling behaviour” (Wichmann et al., 2003), both developed within the framework of the horizontal project.

The horizontal project has the objective to develop harmonised European standards in the fields of sludge, bio-waste and soil to facilitate regulation of these major streams in the multiple decisions related to different uses and disposal governed by EU Directives. The horizontal project includes the Work Package 7 “Mechanical properties” including the subject of physical consistency, because it is recognised that this property is very important for the characterisation of sludge, since it affects almost all treatment, utilisation and disposal operations, such as storage, pumping, transportation, handling, land-spreading, dewatering, drying and landfilling. The importance of the physical consistency is also true for the characterisation of bio-waste and soil. Handling and the utilisation of many other materials, such as cement and asphalt, are strictly dependent on their physical consistency. The needs for control of operations and material characteristics are described.

Preliminarily, a wide search for existing standards and methods to be possibly used or adapted for utilisation in the field of consistency evaluation is described. It resulted that more than 250 standards and non-standardised methods are potentially applicable to consistency evaluation. Then, on the basis of the selected list of standards and
non-standardised methods for further consideration, the methods for the determination of flowability, solidity, thixotropic behaviour and piling behaviour of sludge, bio-waste and soil have been divided into several groups, according to the instruments used for measuring:

- **flowability**: capillary viscometer, penetrometer, rotational viscometer and flow apparatus;
- **solidity**: shearing apparatus, vane testing apparatus and penetrometer;
- **thixotropic behaviour**: a combination of methods should be investigated for the determination of the solidity such as penetration, etc., and an energy-input in terms of flow apparatus to simulate the shear stress;
- **piling behaviour**: slump test apparatus, compacting apparatus, cubic piling box (CPB) and turned box.

For each group, the feasibility at lab-scale or field-scale was evaluated. The recommended methods for flowability resulted in being the coaxial cylinder viscometer at laboratory scale, and flow cone, magnesium penetration cone and extrusion tube viscometer at field scale.

The recommended methods for solidity resulted in being the laboratory vane shear apparatus and vicat needle for use as laboratory reference, and the pocket penetrometer for use on the field. Generally speaking, the penetrometers could be used for both laboratory and field tests. For determination of the thixotropic behaviour the penetrometer, coupled to an energy-input system, e.g. a vibrating table or a hammer, is also a suitable instrument. For measuring the piling behaviour, the cubic piling box (CPB) and the oedometer are recommended methods, the first to be possibly used at both laboratory and field level, while the oedometer only at laboratory scale. All proposed methods now need to be tested and optimised to adapt design and sizing to the materials: this is the programme of future experimental activity.

**Conclusions**

Standardised characterisation methods and procedures for sludge management allow (i) correct fulfillment of legal requirements, (ii) comparison and consistency of application, (iii) improvement of stakeholder and public confidence, and (iv) prediction of the behaviour of sludge when handled and submitted to most management operations.

Work in the course by CEN/TC308, in particular the Task Group 3 of Working Group 1, for the development of standards and technical reports for physical parameters, is intended to avoid problems following the impossibility of comparing results obtained in different places and not standardised conditions, also including the evaluation of methods reliability.

Standardised methods for the evaluation of capillary suction time, settleability and thickenability have been validated and relevant documents submitted to formal vote, together with one technical report (LAB). Standards for specific resistance to filtration and compressibility determination have been submitted to inter-laboratory trials for validation, but results are still under evaluation. The standard on calorific value determination and the technical report on physical consistency have been drafted and are under internal enquiry procedure.

**References**


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