# CHARACTERIZATION OF WASTEWATER SLUDGES END USER'S VIEW

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## 1. Summary

This paper gives an overview of the state of the art on the problem of sludge characterisation. The methods considered are related to sludge production, handling, thickening, dewatering, stabilisation, incineration, agricultural use and landfilling. Measured parameters and concepts are considered and the main limitations and drawbacks are discussed.

## 2. Introduction

Sludges from different sewage works can exhibit wide differences in their physical, chemical and biological characteristics, and there can also be seasonal variations in the sludge characteristics at a particular works. In practice, this means that the designer of a sludge treatment plant should initially obtain information about some specific properties of the sludge to be treated, and about any temporal variations that might occur in sludge properties, in order to produce the best overall process design specification.
It would certainly be unwise to assume that all sludges respond to a given treatment process in the same way. For, example, some sludges may not digest satisfactorily, while others may not dewater adequately even with the use of coagulants, so that the "blind" application of digestion or mechanical dewatering in such cases could lead to expensive process failures.
Characterisation tests to determine "digestibility" and "dewaterability" would prevent such a problem. Similarly, the plant operator often needs to undertake daily laboratory characterisation tests to enable him to identify the optimum operating conditions for the processes treating the plant sludge.

Similar remarks can be made as regards sludge disposal methods. If sludge will be disposed to tip for example, it is necessary to know several mechanical properties of sludge such as its shear strength, its compressibility etc. tests are therefore necessary and the availability of laboratory or pilot equipment to conduct them is equally important. Table 1 gives an overview of the main sludge characteristics related to processing and handling of sewage sludges.

**Table 1 : Characterization tests in relation to sludge treatment and disposal methods**

| Method of sludge treatment and disposal | Transportation | Sedimentation | Activated sludge | Stabilization aerobic | Anaerobic digestion | Chemical stabilazion | Thermal | Thickening by gravity | Flotation | Mechanical methods | Drying beds | Mechanical methods | Tipping, barging | Composting | Incineration | Agricultural use |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Temperature | | | | + | + | | | | | (+) | + | + | | | | |
| Density | + | | | | | | | | | | | | | | | |
| Dynamic viscosity | + | | | | | | | | | | | | | | | |
| Rheological properties | + | | | | | | | | | | + | + | | | | |
| Settling behaviour | (+) | + | | + | + | + | + | + | | | | | | | | |
| Sludge volume/sludge index | | + | + | | | | | + | | | | | | | | |
| Moisture content/total solids residue | | + | + | + | + | + | + | + | + | + | + | + | + | + | + | (+) |
| Volatile substance/fixed residue | | | (+) | + | + | + | + | | | | + | + | + | + | + | + |
| Organic carbon | | | (+) | (+) | (+) | (+) | (+) | | | | | | (+) | + | + | (+) |
| COD of liquid phase | | | | | + | | | | | | | | | | | |
| Digestibility | + | | | | + | | | | | | | | | | | |
| Digestion behaviour | | | | | + | | | | | | | | | | | |
| Digester gas composition | | | | | + | | | | | | | | | | | |
| pH value | + | + | | + | + | + | | | | | + | | + | | + | + |
| Acid consumption | | | | | + | | | | | | | | | | | |
| Volatile acids | | | | | + | | | | | | | | | | | |
| Fats and oils | | | | + | + | | | | | | | | + | | | (+) |
| Surfactants | | | | | + | | | | (+) | | | | + | | | (+) |
| Heavy metals | | | | | + | | | | | | | | + | + | | + |
| Other pollutants (cyanides, halogenated hydrocarbons etc.) | | + | | + | + | | | | | | | | + | (+) | | + |
| Nutrients (N, P) | | + | | + | | | | | | | | | | | | |
| Enzymatic activity | | (+) | | (+) | | | | | | | | | | | | |
| Conditionability | | | | | + | + | | | | | | + | | | | |
| Particle size distribution | | | | + | + | | + | | | | | + | | | | |
| Dewaterability | | | | + | + | + | + | | | | | + | | | | |
| Capillary suction time (CST) | | | | | | | | + | + | + | | + | | | | |
| Specific resistance to filtration | | | | | | | | + | + | + | | + | | | | |
| Compressibility | | | | | | | | | | | | + | | | | |
| Centrifugability | | | | | | | | | | | | + | | | | |
| Calorific value | | | | (+) | (+) | | (+) | | | | | | | | + | |
| Heating by water | | | | | | | | | | | | | | + | | |
| Leachability | | | | | | | | | | | | | | + | | |
| Microbiological | | | | | | | + | | | | | | | + | | + |

In the framework of COST 681, WP1 was particularly concerned with the development of improved tests (1) to determine key physical and chemical characteristics of sewage sludge. Altogether 28 different parameters were identified and published in a compendium of methods (6,7).

These parameters are listed in table 2. Less than half of these methods are standard methods, about one third are tentative methods only and 12 methods are still at the exploratory stage. Thus there is some way to go before a full suite of standardised and harmonised methods of testing sludges is available to the design engineer. In the following discussion parameters will be grouped according to their relation to the sludge treatment processes or disposal method.

## 3. Sludge production

Within a particular sewage treatment works sludge production will be influenced by the volume of incoming wastewater and its quality. The latter will be influenced by the type of activities and the district served. Within the treatment plant sludge is produced by primary sedimentation, by biological treatment processes and by chemical processes, (2), (5).

Sludge production by biological treatment processes will depend on the organic load of the plant but also on the type of the process used. For example extended aeration processes give very low sludge production rates, due to auto-oxidation by bacteria. Similarly chemical precipitates depend on the dose and type of the coagulant added as well as the volume of the influent. Therefore it is very difficult to arrive at sludge production values in terms of per capita or g per DS of incoming sewage. There is therefore a need for these parameters to be accurately determined in each plant.

A protocol for the measurement of sludge production at sewage treatment plants has been proposed by Bruce (4). Both mass and volume of sludge must be measured. On line flow meters using mostly magnetic methods have been used successfully for measuring sludge volumes. Solid concentration can also be measured on-line using optical analysers or ultrasonic attenuation techniques.

**Table 2 List of parameters included in the Compendium of Methods for the of sewage sludges in relation to the design and operation of sludge treatment processes**

| **Gravimetric** | **Stabilisation** |
|---|---|
| 1. Total solids (percent dry matter) | 17. Anaerobic digestibility |
| 2. Volatile solids (loss on ignition) | 18. Aerobic digestibility |
| 3. Dissolved solids | 19. Volatile fatty acids |
| 4. Suspended solids | 20. Bicarbonate alkalinity |
| | 21. Digester gas composition |
| **Consolidation** | |
| | **Stability** |
| 5. Column tests and centrifuge tests | |
| | 22. Stability index |
| | 23. Odour intensity |
| **Conditioning and dewatering** | |
| 6. Conditionability (coagulant demand) | **Incineration** |
| 7. Effect of shear | |
| 8. Specific resistance to filtration | 24. Calorific value |
| 9. Suction time (Buchner funnel) | |
| 10.MFT test | **Handling and disposal** |
| 11.Capillary suction time (CST) | |
| 12.Compressibility coefficient | 25. Rheological properties |
| 13.Ultimate cake solids content | 26. Physical state |
| 14.Particle mobility | |
| 15.Filter leaf yield | **Other parameters** |
| 16.Centrifugability | |
| | 27.Particle size distribution |
| | 28. Density |

## 4. Thickening

Thickening is a process aiming at reducing sludge volumes up to 4 times. Both undigested and digested sludges are subjected to thickening but probably, the most cost effective single process in sludge treatment is gravity thickening of raw sludge since volume reduction has such a profound effect on downstream processing costs, and obviously on transport. A number of tests have been used to estimate thickening performance.
These are listed in table 3.

SVI and SSVI have found widespread use particularly in the control of the activated sludge process. These methods are based on column batch experimental results. The diameter of the column must be large enough to prevent wall effects and other artefacts. Low speed stirring used in the second method limits these problems.
Dimensioning of thickeners is often made by using the flux method which calculates the maximum possible flux of suspended matter through the most critical section of the thickener (26) (38).

A more sophisticated design procedure for sludge consolidation tests was described by Hoyland and Day (11). For any particular sludge, tests are performed using a small centrifuge and a pilot-scale thickener to evaluate three basic parameters (compression coefficient, compression index and resistance to consolidation) which characterises the sludge consolidation properties. Predictions made by the model indicate that, depending on the size of the tank and the resistance to consolidation, there is an optimum height of tank which maximises the concentration of solids in the consolidated sludge.
This research has lead to a dramatic increase in the use of consolidation in the UK and to a lesser extent elsewhere. Similar tests using a low speed centrifuge have been described by Colin and co-workers (36, 37).

**Table 3 : Thickening tests**

| **Parameter** | **Measurement** |
|---|---|
| Sludge Volume Index (SVI) | Sludge volume/suspended solids (ml/g) |
| Stirred Sludge Volume Index (SSVI) | SVI in a slowly stirred graduated cylinder (ml/g) |
| Low speed centrifugation | Sludge blanket settling velocity in a field of 10-100 G |
| Ultimate cake solids content | Dry solids concentration after settling or centrifugation. |

## 5. Dewatering

Sludge dewatering is one of the main unit processes in sludge treatment as it minimises sludge volumes for final disposal. In some countries i.e. Germany, authorities require a high solids percentage, over 35%, if sludge is to be disposed of by controlled tipping. Sludge dewatering can be achieved by filtration or centrifugation. Prior to dewatering sludge is always conditioned.

The parameters related to conditioning are measurements of dewaterability in relation to the amount of coagulants. The most important tests related to conditioning and dewatering are listed in table 4.

Filter leaf testing relates directly to vacuum filters and is quite uncommon in Europe apart from France.

The first two parameters r and CST have found widespread use and acceptance and are by far the most commonly quoted indicators of dewaterability, both measured under standardised conditions (1), (20), (39), (21), (22).

An innovative procedure for automatic measurement of specific resistance has recently been reported by Springer (19). Complementary to specific resistance to filtration are the compressibility coefficient and the shear strength tests.

Methods for predicting belt press performance have been proposed by several authors. Heide and Kampfe (8) described a new procedure for characterisation of the dewatering behaviour of sludges in belt presses, for selecting suitable polyelectrolyte coagulants and for optimising the use of selected polyelectrolytes. The procedure evolved from laboratory scale and full scale evaluations and comprises a combination of conditioning at laboratory scale and the application of a single dewatering test - the modified filtration test (MFT) - which gives an indication of dewaterability as well as the attainable dry solids content of the cake. Verification of the procedure was demonstrated at more than 10 sewage works where aerobically digested sludges were dewatered by belt presses.

Another common method of dewatering is centrifugation but laboratory assessment of sludge "centrifugability" has long proved difficult. Spinosa and Mininni (9) concluded that centrifugability characterisation is very difficult as it is practically impossible to reproduce in the laboratory the conditions actually existing in an industrial centrifuge. In industrial centrifugation three properties are important: settleability, scrollability and floc strength.

Unfortunately no standard methods are available in which the above methods, are considered as a whole. Two methods can be used to evaluate these properties and to select the best type and dosage of polyelectrolyte. In the first method a bench centrifuge is used and the SS concentration is measured after centrifugation, while scrollability is determined by a penetrometer test on the cake collected in the tube. Optimal polymer dosage can be determined and cake concentration and solids recovery can be predicted. However, the procedure is difficult to apply to activated sludges because of their poor consistency.

The other, more promising, method is based on floc strength measurements (based on the COST 68 standard procedure (1) and these seem to provide a reliable means of determining optimal conditioner dose.

Despite the availability of these tests many designers would prefer to carry out full scale trials to evaluate centrifuge performance.

One of the successful new developments in methods for assessing sludge "conditionability" and optimum conditioner dose was described by Cambell and Crescuolo (10), (45) and it has lead to the design of an automatic on-line control system for the dosing of polyelectrolytes.
The basis of the method is the continuous measurement of the rheological properties of sludge using a co-axial viscometer interfaced with a computer to analyse the rheogram and ultimately to proceed a feed-back control loop. Using a standard test methodology of this type, various sludges tested showed a distinct peak in the rheogram at a particular polymer dose and this enabled the optimum dosage to be identified.

## 6. Stabilisation

The object of all stabilization processes are to make sludge less offensive. The first definition of aerobically stabilised sludge proposed by COST - 68 (1) was a sludge where the odour intensity Index (OII) does not exceed 11 at any time prior to 14 days of storage at 20 °C unless the odour can be classified as a typical soil odour. However odour is a highly individual perception and its measurement requires a panel of test persons and a rather laborious test procedure. Odour measurements have therefore found little practical application in sludge treatment.

The ability to assess the anaerobic digestibility of sludges in the laboratory is important for design engineers and plant personnel since such information is useful in predicting gas production and volatile solids reduction. It is also very important to carry out laboratory tests before embarking on the design of digestion facilities because sludges vary in their amenability to digestion. Occasionally, sludge from a particular works will not digest satisfactorily and if initial tests had not been made before a full-scale plant was installed this could represent an abortive capital investment.

**Table 4 Dewatering tests**

| Parameter | Measurement |
|---|---|
| Specific resistance to filtration (r) | Rate of solids buildup in a filter cake |
| Capillary suction time (CST) | Rate of liquid withdrawal from a sludge by capillary suction |

| Parameter | Measurement |
|---|---|
| Compressibility | Rate of increase in r with increasing pressure in filtration |
| Filter leaf yield | Sludge solids captured per unit area in a vacuum filter |
| Shear strength | Rate of change in r or CST with turbulent strength on sludge particles |
| Filter belt press yield | • Rate of gravity water drainage from sludge on filter bed<br>• Rate and extend of water removal from sludge subjected to compression subsequent to drainage |

There are various laboratory digestion tests described in the literature and itemised in the COST Compendium Methods (6). Stuckey (15) introduced the 'Biochemical methane potentials' (BMP) test which involves incubation of test and control samples in 250 ml reagent bottles stoppered with rubber serum caps. Gas production during incubation is monitored using a wet syringe and the samples of gas can also be analysed for methane content. Such tests allow routine monitoring of sludge biodegradability and also reveal the presence of any components toxic to methanogenesis.

The method proposed by the UK Department of the Environment (40) is based on the comparison of gas production of the sample sludge and that of a reference sludge (generally a raw non-toxic sludge with total solids content higher than 4%). The seed sludge is obtained by a laboratory reactor fed with reference sludge (detention time 24 d). The test is carried out with bottles containing different ratios of reference and sample sludge to seed sludge (1:10, 1:3 and 1:2 respectively) at 32°C. The gas produced is recorded twice a day for five days. Three amenability factors are then calculated which represent the ratios of the actual gas yield of the sample sludge to that of reference sludge at the same dilution with seed sludge. The presence of toxics, and possibly a typical behaviour can be so evidenced.

A rapid test lasting 22 hours is also proposed by the UK Department of the Environment.
Experiences of sludge digestibility were also carried out at the Italian Water Research Institute. The proposed method is quite similar to the previous one, but the reference sludge is not considered due to the difficulties of providing and/or producing such a sludge in controlled conditions. The test is carried out at 37°C with bottles filled with sample sludge and seed sludge at different ratios (from 1:1 to 1:5). The gas yield is recorded once a day and the test lasts until a marked decrease is observed, in any case no more than 30 days.

In the Federal Republic of Germany a DIN standard named "Amenability to anaerobic digestion" is existing since 1985 (46). It is based on measurements of gas production of mixtures of the sample sludge with an almost fully digested sludge as seed sludge compared with mixtures of a reference sludge (raw sludge of municipal origin) with the seed sludge. The test is carried out at 35°C over a test period of at least 20 days. The results are expressed as sum of particular specific gas production in litres per kg organic mass (determined by loss on ignition) against the associated test duration in days.

A method of toxicity evaluation using micro-algae has recently been demonstrated by Pun and co-workers (27)

The biochemical "stability" of a sludge is an important parameter but so far there is no standard method for quantifying "stability". An unstable sludge can change in its character very rapidly during anaerobic storage and, for example, show a dramatic increase in coagulant demand. It will usually also become more offensive in odour. Microbial activity measurement for anaerobic sludge digestion has been reported by Chung (33).

By contrast, a completely stable sludge, by definition, will show no adverse changes on storage. Bruce (16) described a variety of non-standard tests to assess the "stability" of both raw and digested sludges (odour potential, volatile solids fraction, biodegradable residue, chemical composition, biological activity).

He also described a test for assessing the stability of raw sludges developed at the Water Research Center (now WRc). This test involves anaerobic storage of the sludge in a standard stirred cylinder at a constant temperature (usually 208C). The sludge is stirred slowly and samples are withdrawn periodically for coagulant demand, non-settleable solids (fines) and odour intensity.

Oxygen uptake rate has been proposed as an indicator of sludge stability in aerobic processes (excess activated sludge, aerobic digestion). In this field great progress has been made and several on-line methods for rapid sludge characterisation have been reported (28), (42), (43), (47).

Moreover several tests exist which measure various types of enzymatic activity in activated sludges. A low activity level indicates a well stabilised sludge (30), (24), (25), (23), (31).

The major application of on-line oxygen uptake measurement is process control as it allows the optimisation of the activated sludge process and the control of the influence of toxic effects. The data of two oxygen electrodes are processed continuously using a microprocessor.

## 7. Physical and rheological properties

The physical properties of sewage sludge are especially important for the design of sludge pumps, thickeners and dewatering equipment, for the optimisation of conditioning, dewatering, heat treatment and landfilling operations.

One of the earliest major contributions to the COST 68/681 activity was the work by Colin (12) on methods for characterising the physical state of sludges. A knowledge of just the water content of a sludge is quite inadequate to predict precisely its physical state, which may be liquid, plastic, solid state with shrinkage or solid state without shrinkage. The contribution made by Colin was to introduce a suite of laboratory tests which allow the physical state of a sludge to be defined quantitatively.

Tests are made using viscometers, penetrometers, extrusion equipment and deformation measuring techniques. Physical properties of sludge and relative tests are shown in table 5.

Colin later described some extensive studies using lysimeters and pilot columns to observe the changes which occur in sludges in simulated conditions of a landfill, both the physical and chemical changes in sludge characteristics being observed, and the effect these changes might have on both the landfill structure and any leachate emanating from it. Guezens and Dieltjens (14) also described some methods for determining the mechanical strength of cohesive sludges in relation to their deposition in landfills.

Another fundamental contribution in the area of sludge characterisation was by Frost (18) who showed that there is an inter-relationship between the rheological behaviour of a sludge and its solids-liquid separation behaviour (i.e. settleability, thickenability and dewaterability). The relationship between viscosity and nominal compressive pressure NCP is much stronger than that between viscosity and solids concentration, by a factor of 25.

In essence, Frost propounded that the application of a given NPC to different sludges would produce sludges with similar viscosity but very different solids concentrations. Frost concluded that the explanation for the inter-relationships may lie in the bound water content of sludges and that under a given compressive pressure sludges will thicken to about the same volumetric concentration of particles.
The practical application of this finding is the ability to produce a sludge consistency scale (analogous to a pH scale) which could be used to identify the optimum sludge concentrations for specific treatment and disposal routes.
This latter work is ripe for further study, with the prospect of identifying a key parameter from which all sludges could be fundamentally characterised in relation to physical behaviour.

## 8. Thermal properties

Increasing attention is paid nowadays towards final treatments of sewage sludges like drying, incineration, pyrolysis. The main characteristic connected with these processes is the calorific value of solid and liquid fuels. The experimental technique consists of burning a sample in the bomb filled with pure oxygen and measuring the amount of energy transferred to the calorimeter.

This is possible by determining the temperature increase of a known quantity of water, following combustion of the sample solid. The experiences carried out at the Italian Water Research Institute allowed to set up a standardised methodology consisting in preparing a table of dry sludge sample and then in estimating the temperature difference. Operating procedure is in accordance with ASTM D3286.

Vesilind (45) in a recent paper draws attention between a higher heating value (HHV) obtained by the use of the bomb calorimeter and a lower heat value (LHV) available for sludge incineration, the difference caused by the condensation of water vapor in the calorimeter (79.7 calories per gram of water formed from combustion).

Hiraoka et al (44) reported other two methods named thermogravimetric analysis (TG) and differential thermal analysis (DTA), very useful for classifying municipal refuse and sewage sludge. The TG analysis consists in the continuous determination of the weight of sample vs temperature. The DTA is based on the use of a calorimeter by which it is possible to observe the difference of the temperature produced by a standardised sample and by dried sludge. TG and DTA curves give, in general, various kinds of qualitative information about the characteristics features of thermal reaction process.

**Table 5 : Physical properties - Identification of physical state**

| State | Definition |
|---|---|
| Liquid | Sludge will drain from a vessel through a calibrated orifice in a specific time. This is the |
| Plastic | The state in which material can undergo permanent deformation if the mechanical stress applied to it exceeds a certain threshold. |
| Solid state "with" shrinkage | This is a solid state in which the volume decreases as drying progresses, i.e. the medium is still saturated with water |
| Solid state "without" shrinking | No reduction in volume upon drying |

**Table 6 : Characterisation tests**

| State | Property | Test |
|---|---|---|
| Liquid | Viscosity | Flow rate through a specified viscometer |
| Plastic | Degree of plasticity | Pressure needed to extrude sludge through a specified orifice |
| | Mechanical stability | Casagrande shock apparatus |
| | Shear strength | Penetrometry test |
| | Adhesion | Force needed to extract specified cones from adhesion to plastic sludge |
| Solid state | Degree of shrinking | Volume reduction upon drying |

## 9. Characterisation of sludges for handling and landfilling

Dewatered sludge water content is not sufficient to forecast mechanical properties of the material which determine the choice of the handling devices and the behaviour of the waste in a landfill. Since the general review made by Colin (30) on mechanical properties of sludges, recent progresses were made by using several geotechnical methods on raw dewatered sludge and sludge sampled in landfills (31, 32). The measured parameters are the bearing capacity ($kN/m^2$), the shear strength by using an apparatus developed for rapid determination of shear strength in cohesive soils (Vane), and the cone shear strength. There is no linear relationship between the shear strength and the dry matter content of the sludge.

The influence of the age of the sludge in the landfill on the geotechnical properties is not clear: in two cases there was no significant changes along the time. whereas in the third case a significant increase of shear strength is thought to be due to the low thickness after natural dewatering.

Several geotechnical methods were compared and discussed in a COST 681 workshop (33) held in 1987.

Konodromos (34) compared on sludges and domestic wastes several German standardised methods (DIN standards n°18 136, 52010, 51067, 4094, 4096).

## 10. Disposal of sludge to agriculture

Agricultural use of sewage sludge is still widely practiced in Europe and other parts of the world, particularly USA. Agricultural reuse is regulated by the 86/278 EEC directive or stricter national regulation. In USA the rule 503 applies.

These rules limit the maximum permissible concentration of heavy metals in sludge and soil and therefore standard methods are required for their determination.
Apart from heavy metals other parameters are required such as nutrients (nitrogen, phosphorus, potassium) and organic micropollutants (PCBs). In the framework of COST 681 several inter-laboratory comparisons led to following recommends regarding the analysis of total metal content in sludge.

The use of standard reference material for internal quality control would be a permanent requirement.
The use of a limited number of simple and reliable digestion methods are preferable instead of a large number of individual methods.
Among the methods recommended for use, digestion with aqua-regia proved the most suitable and reliable method and should be preferred to dry ashing.

As regards PCBs there has been a rapid development of analytical techniques using gas-chromatography with sensitive detection systems. The results of inter-laboratory comparison were published in a special report by EEC in 1985.

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